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Ph.D. Thesis

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**Cohesion-based evaluation of viscoelasticity
in Bitumen Stabilised Materials**

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Dedicated to my Family and Mentors, who believed in me every step of the way.

Abstract

The subject of this doctoral dissertation concerns Bitumen Stabilised Materials with bitumen emulsion (BSM-E) used for the construction of road base courses in cold recycling technology. The aim of the dissertation was to assess the cohesion development in these materials, focusing on the influence of the material-related factors and external conditions on changes in the material's performance over time. The findings enabled the identification and analysis of the viscoelastic behaviour of BSM-E mixtures.

The dissertation consists of seven chapters: three comprising the introduction to the research topic and the theoretical background, and four constituting the research part.

The introductory part of the dissertation includes the formulation of the thesis, central and detailed objectives, research scope and limitations. The theoretical part consists of a literature review on cold recycling technology, covering its historical development, current techniques, and global applications. It discusses the classification principles of cold recycling mixtures (CRM) and provides a general characterisation of Bitumen Stabilised Materials (BSM), highlighting their advantages, challenges, and the role of material components in developing their properties. Moreover, an extensive overview of the BSMs' mechanical performance is presented, discussing failure mechanisms, evolutive behaviour, and their implications for pavement design. Particular emphasis is placed on the assessment of viscoelasticity and cohesion, with a summary of current research findings, testing methodologies, and modelling approaches for permanent deformation and mechanical behaviour.

The research part of the dissertation presents the principles of the experimental program and is divided into three sections: research program, cohesion and permanent deformation response assessment, and viscoelasticity assessment. The dissertation concludes with a chapter presenting the final findings of the research and indicating the directions of the future research.

In the *research program* section, the BSM-E component materials are characterised, and the procedures of mix design, sample preparation, and curing are described. Moreover, the author's original method for long-term conditioning of BSM-E specimens, which simulates the confinement conditions within an asphalt pavement base layer, is presented in detail.

The results of the comprehensive assessment of BSM-E mixtures' cohesion, both in terms of initial cohesion and its long-term evolution, are presented in the *cohesion and permanent deformation response assessment* section. As a novel approach, the Flow Number method (AASHTO T378-22) was implemented for the performance evaluation of ten cold recycling mixtures differentiated by RAP content (0%–90%) and BE content (4%–7.4%),

with conditioning periods ranging from 0 to 120 days. The Flow Number method proved effective in quantifying the cohesion development of BSM-E mixtures over time, using the FN, $\epsilon(\text{FN})$, and FN index parameters, which served as measures of permanent deformation response. It was concluded that the material factors – specifically, the contents of RAP and BE – have a significant impact on the cohesive performance of BSM-E mixtures, with mixtures of higher RAP levels and intermediate BE addition showing the most favourable long-term performance. The decision tree method developed in this dissertation, which considers RAP content, presence of the cohesion development over time, and BE content, enabled both the estimation of the viscoelastic response of the material and the selection of BSM-E mixtures subjected to Dynamic Modulus (AASHTO T378-22) testing, the results of which are presented in the *viscoelasticity assessment* section.

The changes in the dynamic modulus and phase angle values of BSM-E mixtures were analysed across a wide range of test temperatures and loading frequencies. The applicability of modelling the viscoelastic performance of BSM-E mixtures using the approach traditionally employed for hot mix asphalt, taken as the reference, was positively verified. The qualitative and quantitative statistical analysis of the constructed Black curves, Cole–Cole diagrams and master curves allowed for the characterisation of the viscoelastic response of BSM-Es and supported the evaluation of the relationship between cohesive properties and viscoelastic behaviour. Based on the analysis of the test results of the 1%-cement BSM-E mixtures designed in this study, a RAP content threshold of $\geq 70\%$ and a BE content range of 4%–5% are proposed as supporting the viscoelastic behaviour.

On the basis of the findings and analyses carried out within the presented research program, it can be concluded that the main objective of the dissertation – namely, the assessment of cohesion and its development over time together with the evaluation of the viscoelasticity of BSM-E mixtures – has been achieved. This was made possible by fulfilling the partial objectives: identifying and verifying the applicability of specific testing methods and conditioning procedures for the quantitative evaluation of changes in mixture properties; assessing the time-dependent cohesion development in BSM-E mixtures considering material-related variables; and application of the time–temperature superposition for evaluation of the viscoelastic response of BSM-E mixtures. The final findings of the research demonstrate that the implemented experimental program enabled the substantiation of the dissertation thesis.

Keywords: *Bitumen Stabilised Materials, cohesion, viscoelasticity, bitumen emulsion, RAP, cold recycling.*

Streszczenie

Przedmiotem niniejszej rozprawy doktorskiej są mieszanki stabilizowane emulsją asfaltową (ang. BSM-E) do podbudów drogowych wykonywanych w technologii recyklingu na zimno. Głównym celem pracy była kompleksowa ocena kohezji i jej rozwoju w czasie ze szczególnym uwzględnieniem wpływu czynników materiałowych wraz z oceną lepkośćprężystości mieszanek BSM-E.

Rozprawa składa się z siedmiu rozdziałów: trzech stanowiących część studialną oraz czterech poświęconych części badawczej.

Część studialna rozprawy obejmuje wprowadzenie do tematyki badawczej oraz przegląd literatury naukowej. Sformułowano tezę, główne i szczegółowe cele badawcze oraz zakres rozprawy. W części literaturowej dokonano kompleksowego przeglądu stanu wiedzy dotyczącego technologii recyklingu na zimno, przedstawiając rys historyczny oraz stosowane metody i techniki recyklingu. Omówiono zasady klasyfikacji mieszanek wykonywanych w technologii recyklingu na zimno oraz scharakteryzowano mieszanki BSM-E, skupiając się na roli poszczególnych składników w kształtowaniu właściwości mechanicznych oraz wskazując na praktyczny aspekt zastosowania tych materiałów w nawierzchni drogowej. Przedstawiono także dotychczasowy stan wiedzy na temat właściwości mechanicznych mieszanek BSM-E, w tym ewolucji tych właściwości w czasie oraz hipotez dotyczących mechanizmów zniszczenia. Szczególną uwagę poświęcono zagadnieniom oceny lepkośćprężystości i kohezji mieszanek BSM-E, podsumowując zróżnicowane podejścia i metody badawcze stosowane do charakteryzowania właściwości tych materiałów.

Część badawcza rozprawy została podzielona na trzy obszary tematyczne: opis programu badań, analizę kohezji mieszanek BSM-E oraz ocenę ich właściwości lepkośćprężystych. W ostatnim rozdziale rozprawy przedstawiono końcowe wnioski z przeprowadzonych badań i analiz oraz wskazano kierunki dalszych badań dotyczących kohezji i lepkośćprężystości w mieszankach BSM-E.

W ramach opisu programu badań scharakteryzowano zastosowane materiały składowe mieszanek BSM-E, przedstawiono procedurę projektowania składów, a także określono warunki przygotowania i dojrzewania próbek. Przedstawiono autorską metodę długoterminowego kondycjonowania próbek, symulującą warunki skrępowania bocznego i nacisku występujące w warstwie podbudowy nawierzchni asfaltowej.

Do przeprowadzenia kompleksowej oceny kohezji mieszanek stabilizowanych emulsją asfaltową zaimplementowano metodę Flow Number (AASHTO T378-22).

Dziesięć zaprojektowanych mieszanek, różniących się zawartością destruktu asfaltowego (RAP) w zakresie 0%–90% oraz emulsji asfaltowej w zakresie 4%–7.4%, poddano badaniom w okresach kondycjonowania od 0 do 120 dni. Pozytywnie zweryfikowano możliwość zastosowania metody badawczej Flow Number do ilościowej oceny rozwoju kohezji w czasie z wykorzystaniem parametrów FN, $\epsilon(\text{FN})$ i FN index, służących jako miary odporności na deformacje trwałe. Wykazano, że czynniki materiałowe – zawartość RAP i emulsji asfaltowej – istotnie wpływają na kohezję mieszanek BSM-E, przy czym mieszanki o wysokiej zawartości RAP oraz umiarkowanej zawartości emulsji asfaltowej wykazują najbardziej korzystne właściwości w zakresie rozwoju kohezji. Opracowana w rozprawie metoda drzewa decyzyjnego, oparta na hierarchicznej ocenie zawartości RAP, rozwoju kohezji w czasie oraz zawartości emulsji asfaltowej, umożliwiła prognozowanie odpowiedzi lepkosprężystej materiału oraz selekcję mieszanek BSM-E do ostatniego etapu programu badawczego.

Zmiany modułu dynamicznego i kąta przesunięcia fazowego mieszanek BSM-E przeanalizowano w szerokim zakresie temperatur badania i częstotliwości obciążenia, modelując właściwości lepkosprężyste tych materiałów z wykorzystaniem podejścia tradycyjnie stosowanego do mieszanek mineralno-asfaltowych. Przeprowadzona analiza jakościowa i ilościowa krzywych Blacka, Cole–Cole oraz krzywych wiodących z użyciem metod statystycznych pozwoliła na scharakteryzowanie odpowiedzi lepkosprężystej mieszanek BSM-E. Na podstawie analizy wyników badań zaprojektowanych mieszanek BSM-E zaproponowano próg zawartości RAP ($\geq 70\%$) oraz zakres zawartości emulsji asfaltowej (4%–5%) jako warunki materiałowe determinujące kohezję mieszanek BSM-E i umożliwiające scharakteryzowanie ich właściwości jako ciała lepkosprężystego.

Na podstawie wyników zrealizowanego programu badawczego można stwierdzić, że główny cel rozprawy – ocena kohezji i jej rozwoju w czasie wraz z oceną lepkosprężystości mieszanek BSM-E – został osiągnięty. Stało się to możliwe poprzez osiągnięcie celów cząstkowych w postaci: identyfikacji i weryfikacji możliwości zastosowania określonych metod badawczych oraz procedur kondycjonowania do ilościowej oceny zmian właściwości mieszanek; oceny zmian kohezji zachodzących w mieszankach BSM-E w czasie z uwzględnieniem zmiennych materiałowych; oraz zastosowania zasady superpozycji czasowo-temperaturowej do opisu odpowiedzi lepkosprężystej mieszanek BSM-E. Wnioski końcowe z przeprowadzonych badań i analiz potwierdzają, że zrealizowany program eksperymentalny umożliwił udowodnienie prawdziwości postawionej tezy rozprawy.

Słowa kluczowe: *mieszanki stabilizowane emulsją asfaltową, kohezja, lepkosprężystość, emulsja asfaltowa, RAP, recykling na zimno.*

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List of abbreviations

AMPT – Asphalt Mixture Performance Tester
BE – bitumen emulsion
BSM – Bitumen Stabilised Material
BSM-E – Bitumen Stabilised Material with bitumen emulsion
BSM-FB – Bitumen Stabilised Material with foamed bitumen
CBTM – Cement-Bitumen-Treated Material
CCPR – Cold Central Plant Recycling
CIR – Cold In-Place Recycling
CMA – Cold Mix Asphalt
CRM – Cold Recycling Mixture
CTM – Cement-Treated Material
DM – Dynamic Modulus
DSR – Deviator Stress Ratio
FB – Foamed Bitumen
FDR – Full Depth Reclamation
FN – Flow Number
HMA – Hot Mix Asphalt
ITS – Indirect Tensile Strength
MCE – Mineral-Cement-Emulsion mixture
OFC – Optimum Fluid Content
OMMC – Optimum Mixing Moisture Content
OCMC – Optimum Compaction Moisture Content
PD – Permanent Deformation
PDR – Partial Depth Reclamation
RA, RAP – Reclaimed Asphalt Pavement
SMA – Stone Mastic Asphalt
TBC – Total Binder Content
TSR – Tensile Strength Retained
TTSP – Time-Temperature Superposition Principle
VA – Virgin Aggregate
V_m – air void content
WMA – Warm Mix Asphalt

1. Introduction

1.1. Background

In the coming years, the expanding road network in Poland will require systematic reconstruction and maintenance as part of the road pavements life cycle. To address this challenge, effective strategies for managing and reusing materials recovered during roadworks will be essential. One of the key materials in this process is Reclaimed Asphalt Pavement (RAP), which can be successfully incorporated into new pavement structures using hot, warm, or cold recycling technologies, applied both in-place and in-plant. Among these methods, cold recycling is gaining increasing recognition due to its environmental and economic benefits [1] [2] [3]. Over the past two decades, Poland's road infrastructure has undergone a remarkable transformation. According to the official statements of the General Directorate for National Roads and Highways, as of December 2024, the national road network has expanded by 1475 km of highways and 3210 km of expressways [4] [5]. Alongside the development of the national road network, local government road infrastructure has also been growing at a dynamic pace. In 2025 alone, financial support from the Government Road Construction Fund for the projects on the district and municipal roads amounts to PLN 2.7 billion, enabling the construction, reconstruction, or renovation of 2100 km of roads [6]. The Government Program for the Construction of National Roads, launched in 2023, aims to deliver over 2,500 km of new or upgraded highways and expressways, as well as finalize nearly 4000 km of ongoing road investments by 2030 [7].

Considering the ecological and economic aspects of road pavement recycling, cold recycling technology is currently gaining popularity as a sustainable way of pavement rehabilitation. It helps to reduce the use of raw materials by replacing them with recycled materials such as Reclaimed Asphalt Pavement (RAP) or Construction Demolition Waste (CDW) [8] [9]. The use of Cold Recycling Mixtures (CRMs) in road construction results in financial and environmental savings, as it helps to cut down on costs and energy consumption compared to hot mix asphalt (HMA) production [10] [11] [12] [13]. Moreover, another advantage of cold recycling technology is the reduction of occupational hazards since no heating processes during manufacturing and paving are required [14]. As stated in the works of Xiao et al. [13] and Valentin et al. [15], the energy consumption of cold in-place recycling technology is approximately 6 times lower than for the traditional HMA technology, with 90–95% of CO₂ emissions generated during the bituminous binders' production process. No need to use high technological temperatures when performing pavement recycling in the cold

technology makes it possible to use reclaimed materials containing hazardous materials, such as tar [16].

The scope of this dissertation covers the technology of cold recycling of asphalt pavements, in which the recycled material – RAP – is used as a component for the production of CRMs called Bitumen Stabilised Materials. The subject of the research is Bitumen Stabilised Materials with bitumen emulsion (BSM-E) used for the construction of road base courses.

Bitumen Stabilised Materials are considered uniquely different from conventional asphalt mixtures in terms of material composition and internal structure. While HMA generally consist of virgin aggregates, bitumen and suitable performance additives, the main components of BSM-E mixtures are RAP, bitumen emulsion, active filler (e.g., cement), and water. Virgin aggregates can be optionally added to correct the grading curve of the mixture and fit it into suitable guidelines. Unlike the traditional fully-bound HMA mixtures, in which the bitumen mastic binds all the aggregate particles together in a continuous manner, the bitumen in BSMs is selectively dispersed amongst the fine mineral particles coming from RAP and virgin aggregates, creating localized bitumen mastic bonds. During the bitumen emulsion breaking process, selected coarse aggregates also obtain a certain level of residual bitumen coating. As a result of this phenomenon, Bitumen Stabilised Materials are characterized as non-continuously bound mixtures which may exhibit properties of both granular and viscoelastic, HMA-like materials [10] [17].

While the mechanical and durability performance of traditional HMA mixtures used in base courses of flexible asphalt pavements is a well-documented and widely studied topic, the evolution of BSM properties during service life remains insufficiently understood [18]. The current research experience does not allow for an unequivocal statement on the BSM layers' short- and long-term mechanical performance in pavement structure. Based on the current state of research, a general statement can be made that Bitumen Stabilised Materials display evolutive mechanical properties, which depend on various material and technological process-related factors, such as the applied binding agents proportions, content and quality of reclaimed asphalt pavement material, the created level of the residual binder-aggregate adhesion, and finally successive evaporation of water due to curing in construction [9] [19] [20] [21].

All of the abovementioned factors are related to the phenomenon of the BSMs' cohesion development understood as an internal integrity of the composite. Determination of the BSMs' cohesion in monotonic triaxial testing dedicated for granular materials has been widely documented and included in the mix design guidelines [10] [17] [22] [23]. Apart from analysis

of the shear performance of these materials, the use of dynamic triaxial tests (EN 13286-7 [S1]) has been reported as a method of assessment of the BSMs' permanent deformation response while treated as unbound mixtures [24] [25] [26]. However, the phenomenon of the BSMs' cohesion in terms of viscoelasticity remains briefly addressed in the literature [27]. Since the investigation of this topic requires a different experimental approach targeted at the determination of the HMA-like materials' permanent deformation resistance, a synthesis and adaptation of testing methodologies primarily applied to assess the behaviour of asphalt mixtures are needed.

One of the critical limitations in designing flexible pavements with base courses made of BSMs, particularly for high-traffic roads, is the insufficient understanding of their initial mechanical properties and durability characteristics. This includes aspects such as fatigue failure mechanisms, which are influenced by the cohesive and viscoelastic nature of BSM mixtures. To address this knowledge gap, the first essential research step is to determine whether BSMs can exhibit a fully linear viscoelastic response. Additionally, it is crucial to identify the material conditions that influence the viscoelastic, time- and temperature-dependent behaviour of BSMs.

The studied issue is crucial for the broader application of BSM mixtures in road pavement construction. It focuses on defining the conditions necessary to achieve a material with consistent and predictable viscoelastic behaviour, which is essential for controlling reflective cracking in flexible asphalt pavements.

1.2. Dissertation thesis

The thesis of the dissertation is that it is possible to determine the threshold contents of Reclaimed Asphalt Pavement (RAP) and bitumen emulsion (BE) for which the BSM-E mixtures exhibit linear viscoelastic properties.

This dissertation attempts to answer the following questions:

- How does the BSM-E component selection influence the cohesive properties of the material?
- Can the cohesion of the Bitumen Stabilised Materials change over time as a result of the influence of external conditions?
- Is it possible to determine the threshold amounts of bitumen emulsion and Reclaimed Asphalt Pavement that will ensure obtaining a viscoelastic material?
- Is the cohesion of the BSM-E mixture related and to what extent to the viscoelasticity of the material?

1.3. Objectives of dissertation

The main objective of the thesis was to assess the cohesion development in Bitumen Stabilised Materials with bitumen emulsion over time. Cohesive properties and permanent deformation response of the Bitumen Stabilised Materials treated as unbound materials have been widely evaluated in the triaxial testing manner, originally applied for cohesive soils. Nevertheless, scarce research on cohesion, understood as composite integrity and resistance to permanent deformation of BSM-Es considering their viscoelastic nature, has been presented to date. Based on the recent state of knowledge, it has yet to be established whether, to what extent, and under what factors the change in cohesion in BSM-E mixtures occurs. In this thesis, it was assumed that the ongoing development of cohesion in the material, which may result from the material consolidation and interactions between the residual bitumen and the virgin aggregate and RAP skeleton, affects the viscoelastic properties of BSM-E mixtures used as base courses in flexible pavement structures.

The detailed objectives of the dissertation were as follows:

1. Identification and verification of the applicability of specific test methods and conditioning procedures to observe and quantify the changes in BSMs' performance over time.
2. Assessment of the cohesion changes occurring in BSM-E mixtures over time, considering the material composition (bitumen emulsion and Reclaimed Asphalt Pavement content).
3. Assessment of the Bitumen Stabilised Materials' viscoelasticity through the application of performance modelling approaches used for asphalt mixtures.

1.4. Research scope and layout

This dissertation analyses the cohesion development phenomenon in Bitumen Stabilised Materials with bitumen emulsion, considering the influence of the material's composition and an effect of the conditioning time and settings. For this purpose, dynamic creep testing was performed on BSM-E mixtures containing various contents of bitumen emulsion and Reclaimed Asphalt Pavement within the conditioning timespan of 0–120 days. Moreover, the research scope includes an assessment of the viscoelastic properties – dynamic modulus and the phase angle – of the selected BSM-E mixture compositions. The validity of the modelling approaches, traditionally applied to describe viscoelastic response of the material, was verified by fitting the gathered experimental data and therefore allowing for a conclusion on the viscoelasticity of the tested BSM-E mixtures.

The dissertation consists of seven chapters.

In Chapter 1, an introduction to the subject of the dissertation is provided. It presents the dissertation thesis, central and detailed objectives, scope, and limitations of the research.

Chapter 2 presents a literature review on cold recycling technology, covering its historical development, current techniques, and global applications. It discusses the classification principles of cold recycling mixtures and provides a general characterisation of Bitumen Stabilised Materials, highlighting their advantages, challenges, and the role of material components in shaping their properties.

Chapter 3 provides an extensive overview of the mechanical performance of BSMs, discussing failure mechanisms, evolutive behaviour, and their implications for pavement design. Particular emphasis is placed on the assessment of viscoelasticity and cohesion, with a summary of current research findings, testing methodologies, and modelling approaches for permanent deformation and mechanical behaviour.

Chapter 4 presents the experimental program developed to address the research hypotheses. It comprehensively describes the research plan, the procedures for mix design, sample preparation, curing, and long-term conditioning, and provides a thorough account of the mechanical testing methodologies utilized.

Chapter 5 focuses on the assessment of cohesion and permanent deformation response of BSM-Es. The chapter discusses the initial cohesion characteristics, including the influence of RAP content and bitumen emulsion dosage on the internal structure integrity and shear failure resistance. Furthermore, it presents the analysis of cohesion evolution over short- and long-term conditioning periods, along with a discussion of the observed trends and mechanisms influencing the BSM-Es' mechanical performance.

Chapter 6 presents the application of the time–temperature superposition principle (TTSP) to BSM-E mixtures, based on dynamic modulus and phase angle data analysed across various temperatures and frequencies. Master curves, Black curves, and Cole–Cole diagrams were used to evaluate their viscoelastic behaviour relative to conventional asphalt mixtures. The chapter concludes with a discussion on the cohesion–viscoelasticity interrelation, summarising key insights derived from the cohesion and viscoelasticity analyses and providing technical and engineering recommendations.

Chapter 7 presents the conclusions drawn from the conducted research. It summarizes the main findings regarding the mechanical performance of BSMs, particularly their cohesion development and viscoelastic response, and outlines recommendations for future studies and potential applications in pavement engineering.

1.5. Limitation of the research

In this study, Bitumen Stabilised Materials with bitumen emulsion and 1% cement content, incorporating locally available, selected component materials, were tested. Although the BSM-E mixtures' composition remains compliant with national technical requirements in terms of cement content, it deviates from the standard engineering practice in Poland, where 2–4% cement is typically used. Cohesion of BSM-Es was evaluated by means of Flow Number testing performed at one temperature, selected on the base of preliminary test results. The modelling of the material's viscoelastic response in this study was limited to the use of existing mathematical models, originally developed for HMA. The experimental investigations in this study were limited to laboratory testing, with no field testing or performance monitoring conducted due to the extensive laboratory programme and time constraints.

I. LITERATURE REVIEW

2. Review on Cold Recycling Technology

2.1. Characterisation of Cold Recycling Technology

2.1.1. Historical background

The beginnings of pavement recycling technology date back to 1915 when the American company The Warren Brothers Co. employed the first hot recycled asphalt mixtures [28]. However, it was only in the 1970s that the dynamic development of pavement recycling technology began, which was contributed to by the oil crisis in 1973 [29]. A sharp increase in the price of crude oil on the world markets, and the corresponding increase in bitumen prices, provided the impetus for the road industry and agencies to find ways to save costs and stretch the existing bitumen supply. The earliest references to cold recycling technology refer to the American experience at the turn of 1970s and 1980s [30] [31]. The first mentions concern predominantly cold in-place recycling (CIPR) operations [13] [32] [33]. In 1980, the first National Cooperative Highway Research Program (NCHRP) guidelines for recycling pavement materials were published as well [34]. According to this report, cold mix processes performed at a central plant were introduced as one of the options for bituminous pavement recycling that could be implemented in case of minor and major structural improvement with or without a new binder.

One of the first experimental cold recycling projects was described in the *Tamworth and Jackson Cold Recycling Project Report* published as a part of the documentation of the *Demonstration Project 39 – Asphalt Recycling* commissioned by Federal Highway Administration (FHWA) [35]. This report presented conclusions regarding the possible methods of removal of existing asphalt pavements, the principles of the RAP base course stabilization technology, as well as cost analyses were also carried out. Moreover, the recommendation of adding a binder in the form of bitumen emulsion was given to strengthen the reclaimed stabilised base course. A considerable number of in-situ cold recycling projects with the use of bitumen emulsion stabilization were successfully completed at that time (e.g., Mercer County Project, US 64 Project) [30].

The first practical experience with cold recycling technology in Europe was gained in the Netherlands in 1975-1981 when several test sections were constructed on several highways to test base and subbase courses performance [36]. The analysed structural layers were paved

as unbound materials without binders, using cement stabilization technologies (*crushed asphalt cement* material) and bitumen emulsion stabilization methods. Both in-place and in-plant recycling strategies were considered. The Dutch experience in the field of cold recycling is followed by the 1980's German tests in which the reclaimed asphalt pavement was reused together with an addition of sand, solvents, bitumen emulsion or cement [37].

The development of cold recycling technology is strongly related to the technological achievements. As stated by Jenkins and Yu [38], the rapid development of recycling equipment (cold milling machines, in-plant cold remixers, cold stabilizers, and recyclers) in 1990s has significantly contributed to the development of mix and pavement design procedures for cold recycled materials produced in foamed bitumen and emulsion technologies. The launch of the first in-place recycler capable of rehabilitating a road in a single pass in 1986 by the Wirtgen Company was the first, important step towards spreading cold recycling technology worldwide [37].

Polish experience in cold recycling technology dates back to the second half of the 1990s [39]. During that time, many roads, both on national and regional level, were suffering from various signs of degradation, such as fatigue cracking, transverse cracking, rutting, and local material loss. Due to a significant deterioration of the existing pavements, which were often inhomogeneous and contained tar binders, an ecologically efficient and economical pavement rehabilitation technology needed to be applied to rebuild safe traffic infrastructure. One of the first introduced cold recycling techniques for the base courses was full-depth reclamation. In 1997, the first Polish requirements for cold recycling mixtures called Mineral-Cement-Emulsion (MCE) mixtures were introduced [40], followed by the revised version in 1999 [41], which became a basic instruction for the rehabilitation of distressed pavements, both in-place and in-plant. MCE mixtures consisted of RAP mixed with virgin aggregates, water and stabilising agents in the form of cement and slow-setting cationic bitumen emulsion. The research programs and road inspection projects carried out over the years 2005-2014 on the pavement sections with MCE base courses constructed according to the recommendations developed in 1999, resulted in the publication of the revised requirements in 2014 [42], which were eventually modified and published in 2019 to be used as current guidelines [39] [43] [44]. Flexible pavement structural systems incorporating cold recycling mixtures in base courses were included in the current *Polish Catalogue of Typical Flexible and Semi-Rigid Pavement Structures* [45]. Yet, their primary use has been limited to light and medium traffic roads.

In the last ten years, comprehensive international research programs were conducted to characterize and unify the design standards, compaction methods, curing procedures, and

performance tests for cold recycling mixtures following the existing scientific and engineering experience and approaches [23] [46]. Several research projects related to cold recycling technology have been implemented in Poland as well. Research work concerned, among others, the implementation and development of recommendations for using Bitumen Stabilised Materials technology for base courses [47] [48], as well as optimizing the binding agent's composition for deep cold recycling mixtures with foamed bitumen [49].

The perspective of the cold recycling technology development in Poland concerns mainly the rehabilitation of the local (municipal and district) roads, whose share in the total Polish road network is 88% [50]. Among the main problems that local roads face, the following can be listed: poor technical condition and severe deterioration signs due to the exploitation time of more than 20 years, lack of adequate bearing capacity and insufficient frost resistance. Moreover, the problems of tar materials' recycling that are present in the existing pavements as well as the stockpiled RAP material's disposal need to be addressed. Hence, with the growing importance of ecological aspects in road construction and the need to use material reclaimed from the demolition of the recently expanded road network, further development of cold recycling technology in Poland should be forecast.

2.1.2. Overview of Cold Recycling techniques

Cold recycling involves recovering and reusing material derived from existing road asphalt pavements without the addition of heat to produce new mixtures that can be applied to road construction [51]. Since the rehabilitation and reconstruction techniques used in cold recycling technology have been developed and extensively studied by many researchers and highway agencies throughout the last decades [52] [53], various classifications of cold recycling technology have been proposed considering factors such as the processing place, reclamation depth and pavement construction technology. The primary classification of cold recycling divides the methodologies depending on the processing place. Thus, Cold In-place Recycling (CIR), performed *in situ*, and Cold Central-Plant Recycling (CCPR) in a production plant are distinguished. Apart from CIR, the FDR technique is mentioned among the techniques performed in-place. Even though some researchers consider CIR and FDR as one category [8] [54], in this thesis, FDR is introduced as a separate technique due to different characteristics of the recycled material compared to CIR which is also referred to as PDR (partial-depth reclamation).

The application of each of the above-mentioned specific cold recycling techniques is dependent on a number of material, technological and logistic factors, such as the existing

pavement distress conditions (type and severity of the distress), service life expectations, the quality of the recycled material and availability of raw materials, type and localization of the road.

2.1.2.1. Cold In-place Recycling (CIR)

According to the adopted method of classification, CIR technique can be also recognized as a *PDR (partial-depth reclamation)*, *surface recycling* or *100% RAP recycling* [10] [55] [56]. In this method, the existing pavement is milled to a depth ranging from 50 mm to 100 mm and the reclaimed material obtained exclusively from asphalt pavement layers is processed on site and reused immediately [51]. It is possible due to the application of milling machines – cold recyclers equipped with a mixing chamber containing a cutting drum that pulverizes the pavement and microprocessor-controlled pumping systems for adding fluids to the recovered material (water, bitumen emulsion or foamed bitumen). The mixing of recycled material (and additional virgin aggregate, if needed) takes place in the milling machine. In some cases, trailer mounted pugmill mixer is used for this purpose. The recycling process is carried out coupling bulk supply road tankers containing the required additives to the recycler. The configuration of the tank trucks and road tankers depends on the particular recycling application (type of the cold recycling mixture produced and the binding agents applied). Moreover, when the use of cementitious binders (e.g., cement or hydrated lime) is required, the separate spreaders spread the powdered stabilizing agents on the surface of the existing road. As the cold recycler advances, the binder is mixed together with the milled material and fluids in a single operation. Bitumen emulsion or foamed bitumen, characterized by a reduced viscosity in ambient temperatures compared to traditional bitumen, are used as bituminous binders in the recycling process.

The compaction of the layer produced in cold recycling technology is carried out by vibratory steel wheel rollers and pneumatic-tired rollers. The CIR rehabilitation process can be performed either by single-unit recyclers (tyre- or track-mounted) with the support of additional machines and equipment or using multi-functional recycling trains [57] [58]. Since CIR does not involve hauling RAP to the central depot, it is more suitable than cold central plant recycling (CCPR) particularly for roads located at a considerable distance from a plant. Considering a limited reclamation depth including only asphalt layers, CIR is considered the best solution for rehabilitation of lower volume road pavements that require a simple surface treatment, but simultaneously consist of structurally sound, stable and well-drained base and subbase courses as well as subgrades [55].

2.1.2.2. Full-depth Reclamation (FDR)

Full-depth reclamation is a cold in-place recycling technique in which the recycling depth ranges from 100 to 300 mm and asphalt layers are milled together with a part of bound (e.g., cement-treated) or unbound base layers (e.g., crushed stone, mechanically stabilised gravel) [10] [59]. The basic FDR approach is to combine the recovered material with water without the use of stabilizing agents by means of reprocessing or mechanical modification treatment, assuming the recycled material does not require an improvement in engineering properties. However, the most common approach is to mix the obtained blend of recycled asphalt and granular base materials with water and binding agents directly on the construction site. Subsequently, cold recycling mix produced in the recycler is paved and compacted as a new cold recycling course with the use of analogous machines and equipment as in partial-depth recycling (PDR, CIR). The FDR recycling process is illustrated in Figure 1. As reported by Tebaldi et al. [56], modern recyclers can perform the FDR process in a single or more passes, providing the desired structural integrity and homogeneity of a new recycled pavement layer. The FDR technique is mainly used to rehabilitate pavements with frequent and severe structural distresses in the form of surface cracking, potholes or deformed granular layers.

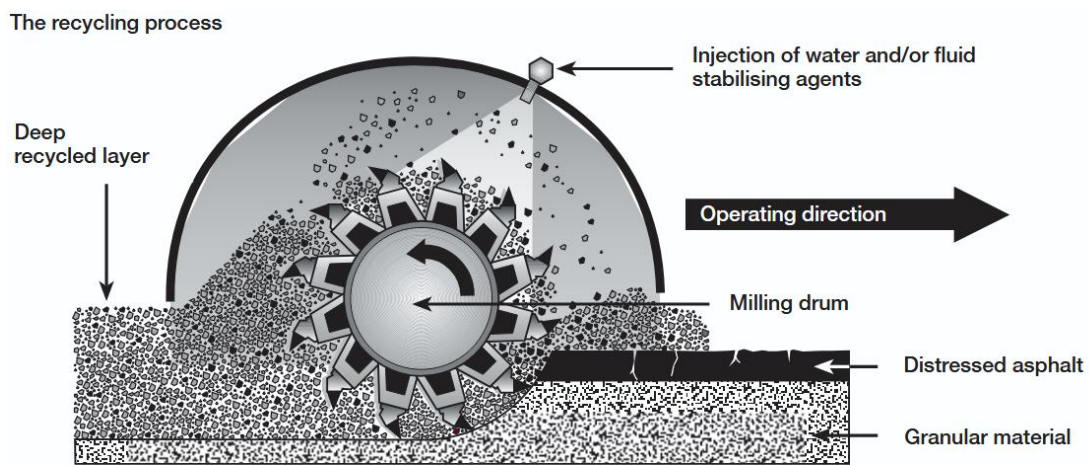


Figure 1 Process of cold in-place recycling: FDR technique with bitumen emulsion [10]

2.1.2.3. Cold Central Plant Recycling (CCPR)

Cold Central Plant Recycling technique (also called as Cold Plant Recycling – CPR or Cold In-Plant Recycling - CIPR) is conducted at a central or mobile plant. Similarly to the cold in-place recycling process, the first step of the CCPR technique is milling the existing, deteriorated asphalt pavement. However, then the recovered material is hauled and stockpiled in plant for later use in CRMs production. Collecting material at a central depot allows for a proper selection, crushing, screening and testing of RAP before its application in the mixing process. The RAP pre-treatment is particularly useful when dealing with asphalt material characterized

with a high qualitative variability or considerable hardness that does not allow for its proper milling and pulverization in-situ [10]. Thus, a better variability control of the input RAP material for the CCPR technique is expected, compared to CIR. Moreover, the CPPR technique allows for a greater control over the component (aggregates, fluids, binding agents) dosing process, enabling the implementation of any required adjustments to provide a suitable mixture quality during mixing, ensuring satisfactory performance of the CRM layer. Since the whole process of the mix production takes place only in plant, the ready-made CRM is transported to the construction site, paved and compacted using pavers, steel wheel rollers and rubber-tired rollers [60].

2.1.3. Classification of Cold Recycling Mixtures

One of the critical challenges in the widespread adoption of CRMs in road construction is the need for a harmonised classification system. Based on the conducted review of scientific literature, research reports, standards and recommendations given by international road agencies, it can be stated that the nomenclature and material qualitative and quantitative criteria used in the classification of cold recycling mixtures (CRMs) have not been fully unified worldwide.

The problem of the existing diversity in characterization of commonly used cold recycling mixtures was addressed, inter alia, in the work of international RILEM Technical Committee TC 237-SIB as well as in the CoRePaSoL research project, which aimed to review and synthesise the available test and mix design procedures applied for CRMs in European countries, United States of America and South Africa [23] [46] [61]. Based on the findings presented in the reports and recommendations resulting from the abovementioned research, it can be concluded that the applied mix design methods, production and curing regimes as well as performance evaluation criteria for cold recycling mixtures significantly differ depending on the country or region in which cold recycling is performed [8] [23] [62]. The expected performance and service life of pavements with CRM layers, local climatic conditions of pavement operation, ground and water conditions as well as characteristics of the stabilised material and binding agents in a given location play an important role in the development of design procedures and achieving desired final properties of CRM mixtures in construction. Moreover, laboratory assessment methodologies of CRMs' properties vary significantly throughout the world as they are strictly connected to the applied technologies of CRM production, starting from different mix design approaches, preparation and pre-treatment of

mixture components, mixing process parameters, pre-compaction and compaction procedures, as well as post-compaction curing conditions [61].

The properties of CRMs depend on the qualitative and quantitative selection of their components, which are Reclaimed Asphalt Pavement (RAP), virgin aggregates, bitumen emulsion or foamed bitumen, water, and active filler [63]. The lack of unambiguity in the nomenclature and classification criteria of CRMs found in the literature is also associated with the diversity of mechanical properties of this group of materials. Depending on the applied combination of both bituminous and cementitious binding agents, cold recycling mixtures may exhibit different behaviour in road construction – from stress dependency characteristic for granular materials (unbound stabilised aggregate) to time and temperature dependence as in the case of viscoelastic materials, such as Asphalt Concrete (AC) [59]. The changes in CRMs' performance occurring as a result of the variation of bitumen and cement content are depicted in Figure 2.

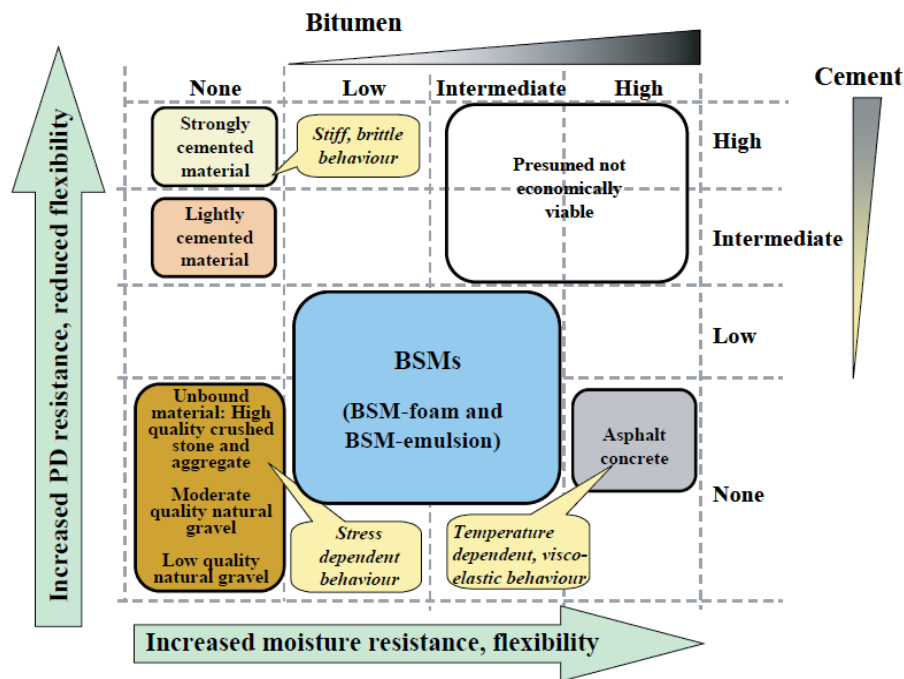


Figure 2 Conceptual composition of pavement mixtures [17]

As can be seen in the Figure 2, the group of cold recycling mixtures consists of materials presenting notably different mechanical performance. The application of high amount of cementitious binder while maintaining low amounts of bitumen in the mix results in an increased stiffness, permanent deformation resistance and bearing capacity of the material. However, the cold recycling mixtures predominantly treated with cement are also characterized with high susceptibility to shrinkage cracking due to their rigidity and brittleness [64]. The increase in the bituminous binder content while keeping low amount of hydraulic binder leads

to an improved flexibility, fatigue performance and moisture resistance of the produced cold recycling mixtures. Depending on the type of the designed pavement structure (rigid or flexible) and the expected resulting properties of the material in the road pavement, the proportion of binders is individually selected to produce a layer that will meet the requirements of the project.

Taking into account the general conceptualization of the development of cold recycling mixtures properties, researchers and engineering practitioners proposed classifications and terminology based on the applied percentage ranges of bituminous and cementitious binders or their ratios in CRMs.

One of the most general classifications was proposed in the work of South African researchers, Liebenberg and Visser [65], where lightly and strongly cemented materials containing exclusively cement in the range of 1.5% – 5.0% were defined as Cement-Treated Materials (CTMs), while Emulsion-Treated Materials were described as mixtures in which bituminous binder was used in the range of 0.5% – 4.5% with cement content not exceeding 2% of the mixture mass. As presented in Asphalt Academy guideline requirements [17], Cement-Treated Materials (CTMs) are defined as cold recycling mixtures with a bitumen/cement (B/C) ratio lower than 1, while the residual bitumen content in Bitumen Stabilised Materials (BSMs) does not exceed 3% by mass of dry aggregate with cement content less than 1% ($B/C > 1$). According to Wirtgen Cold Recycling Manual [10], the anticipated application rate of cement in Cement-Treated Materials ranges from 2.0% to 3.0% of dry aggregate mass without the addition of bituminous binders. Bitumen Stabilised Materials (BSMs) are defined as mixtures in which the residual bitumen content ranges from 1.8% to 4.0% with cement or hydrated lime amount reduced to 1.0% and 1.5%, respectively.

One of the most widespread classifications of cold recycling mixtures was presented in the works of Grilli et. al [66] and Godenzoni et al. [67]. The researchers distinguished four main groups of cold recycling mixtures. A graphical representation of the classification rules with the main criteria used for the distinction of the CRMs - type and amount of binding agents – is shown in Figure 3. Additional information regarding the origin of the mineral material – virgin and recycled aggregate (RAP) – generally used to produce mixtures was provided by the author.

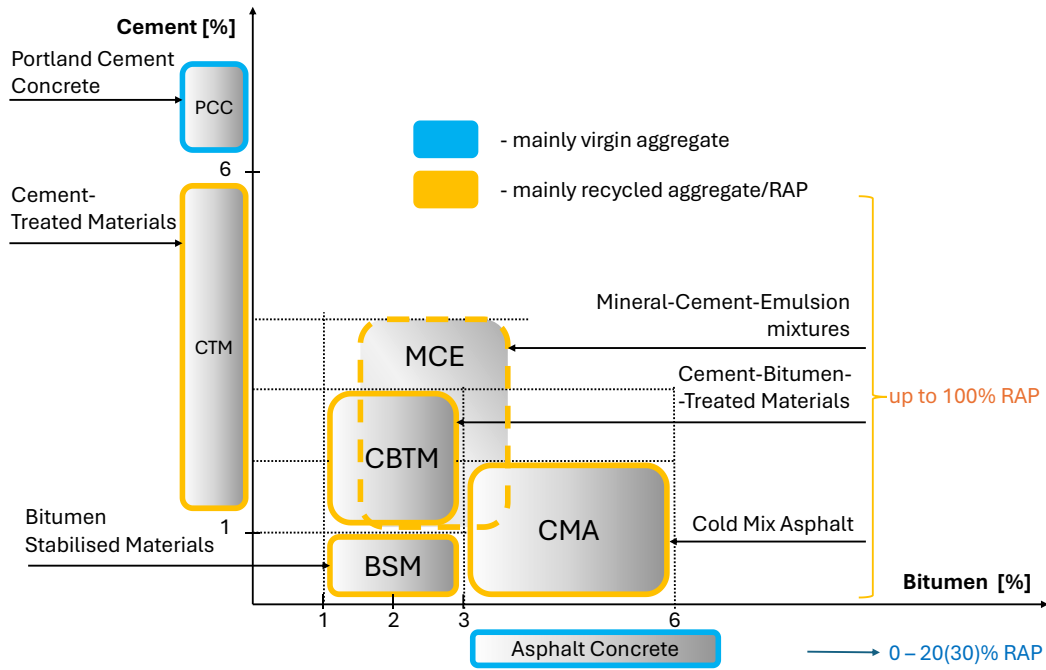


Figure 3 Classification of pavement mixtures (adapted from [66])

Cement-Treated Materials (CTMs) with cement as the only binding agent (from 1.0% to 6.0% mixture mass) are considered to be bound materials with high stiffness, rigidity, and brittleness [68] [69], while on the opposite – Cold Mix Asphalt (CMA) produced with high amounts of bitumen (>3%) and intermediate amount of cement (up to 2.0%) are characterized by an increased moisture resistance and flexibility. Due to this specific composition of binding agents, it is reported that the mechanical behaviour of CAMs can be successfully assessed following the similar approach as for HMA [70]. Cold recycling mixtures containing both cementitious and bituminous binders are called Cement–Bitumen-Treated Materials (CBTMs) and Bitumen Stabilised Materials (BSMs). In BSMs, produced with bitumen emulsion or foamed bitumen, the residual bitumen content is usually less or equal to approximately 3% by aggregate mass with the amount of cement limited to 1%. Finally, CBTMs are designed with a similar amount of bitumen but higher cement contents (up to 2.5%) compared to BSMs, which results in a higher stiffness of the material. In the case of this type of mixtures, the balance between the bitumen emulsion or foamed bitumen and cementitious binder dosages needs to be established in order to obtain a material with optimal stiffness, cracking resistance and thermal sensitivity [66] [71].

Classification presented in the work of Mollenhauer [72], in addition to previously mentioned BSM and CBTM mixtures (in this work referred to as BCSMs – Bitumen-Cement-Stabilised Materials), includes the following types of cold recycling mixtures: Cold Asphalt (CA) – mixture prepared with bitumen emulsion as an only binder, characterised by very high

residual bitumen contents (approximately 6%), Grave Emulsion produced with bitumen emulsion in the amount exceeding 5% aggregate mass and Sealing Cold Recycled Materials (SCRM) incorporating high amounts of bitumen emulsion or foamed bitumen (3% - 6% residual bitumen content) and considerable amount of cement (1% - 6% of aggregate mass) for recycling of road pavements containing hazardous materials (e.g., tar).

In this thesis, the following terminology was adopted:

- Cold Recycling Mixtures (CRMs) for the general group of mixtures produced in cold recycling technology;
- Bitumen Stabilised Materials (BSMs) for cold recycling mixtures with a reduced amount of cement ($\leq 1\%$) and bitumen emulsion (BE) or foamed bitumen (FB) used as a primary binding agent. Consequently, the name of BSM-Es was used for BSMs with bitumen emulsion and BSM-FBs for BSMs with foamed bitumen;
- Cement-Bitumen-Treated Mixtures (CBTMs) for cold recycling mixtures in which bitumen emulsion (BE) or foamed bitumen (FB) and cement ($>1\%$) are both characterized as binding agents;
- Mineral-Cement-Emulsion Mixtures (MCE) for cold recycling mixtures widely-known and used in Poland, containing from 1% to 4% of cement and 3% to 6% of bitumen emulsion;
- Cold-Mix Asphalt (CMA) for cold recycling mixtures containing bitumen emulsion (BE) or foamed bitumen (FB) with a residual binder content less or equal to 6% and cement content lower than 2%.

2.1.4. Field performance

Due to the number of economic and ecological benefits that characterize the cold recycling technology, it can be successfully used in the construction of flexible pavements. However, the application of CRMs in pavement courses requires an individual approach to meet the design requirements as well as technological and material conditions of a specific project. Depending on the severity of the pavement deterioration (rutting, transverse, reflective, and longitudinal cracking or potholes) and the desired pavement structure performance, a suitable rehabilitation technique can be chosen to create a new, fully functional pavement layer. Nowadays, it can be estimated that nearly 90% of cold recycling rehabilitation projects worldwide is performed using the in-place techniques (both CIR - PDR and FDR) [10] [73] [61]. The application of CRMs' layers is also possible in newly constructed pavements when the CCPR technique is used [74].

Despite the fact that cold recycling technology method has many significant benefits, the limitations which impede the popularization of this technology are still present – mainly regarding the lack of comprehensive and unified mix design and pavement design approaches, as well as a still limited practical experience of many contractors and road agencies in cold recycling technology. As reported in *Asphalt in Figures 2023 EAPA report* [73], in general, the minority of the available RAP material is currently used in cold recycling processes in European countries and USA, with the most popular way of recycling RAP being still hot and warm processes. Among the European countries, the biggest shares of the available RAP used in cold recycling techniques (on-site and in-plant) were reported subsequently in Czech Republic (30%), Croatia (21%), and Spain (17%).

The practical experience described so far in the literature shows that the use of cold recycling mixtures is mostly limited to the construction of base courses (to a lesser extent of binder and subbase layers) in low- and medium-volume traffic roads [13] [75] [76] [77] [78]. The evidence on application of cold recycling technology to heavy-trafficked highways is scarce, with single cases of trial sections performed [79] [80]. However, as stated in the *Cold In-Place Recycling (CIR) Study* published by FHWA [81], American federal agencies with an extensive CIR experience do not have traffic restrictions on the cold recycling application, with all traffic loads and pavement courses taken into consideration. Service life of cold recycled pavements in which cold recycling mixture layer is used in conjunction with an HMA overlay has been reported to range from 20 to 34 years [74] [81].

According to Polish requirements for flexible and semi-rigid pavements design [45], the typical solutions enable the use of cold recycling mixtures in base courses constructed in a one-layer design variant for low and medium traffic categories (KR1-KR3) and a two-layer option for a higher-medium traffic category KR4 with the upper layer made from asphalt concrete. In general, cold recycling mixtures should be paved in layers of an overall thickness of 15 cm to 20 cm depending on the road traffic load category. Base courses of high-traffic category roads (KR5-KR7) can also be performed in cold recycling technology. However, the mixture production is then restricted to stationary plants and its application is based on individual pavement construction design approach.

Promotion of the use of cold recycling technology on a wider scale requires a better understanding of the CRMs' mechanical performance in construction. Currently, a considerable amount of research is being done to evaluate the development of CRMs' properties in the long term and verify the appropriateness of the existing mix design methodologies. Main conclusions

from selected works that consider the topic of the cold recycling mixtures' field performance assessment are presented below.

The evaluation of the long-term (3 months to 12 months) field performance of CCPR trial sections performed in Malaysia showed satisfactory mechanical and durability properties of binder courses constructed with CRMs in comparison to traditional rehabilitation methods incorporating HMA [75]. 100% RAP mixtures stabilised with 3.5% of bitumen emulsion and 1.5% cement were used. Nearly 10% reduction in central deflection in falling weight deflectometer (FWD) testing of pavements containing CRM-E layers indicates that cold recycling mixtures have a potential of improving the overall structural pavement capacity. Moreover, road sections containing CRMs displayed an excellent rutting resistance with no noticeable increase in the average rut depth in the analysed timeframe, with an average rut depth less than 2.5 mm after 12 months. No cracking was observed in any of the sections after 1 year of exploitation.

Godenzoni et al. [67] evaluated the evolution of mechanical performance of CBTM subbase layers over a period of 8 years by means of field FWD surveys and laboratory stiffness modulus testing. The analysed cold recycling mixtures contained 100% RAP and were stabilised with bitumen emulsion (3%) or foamed bitumen (3%) and cement (1.75% - 2%). Based on the test results, the researchers concluded that in the timespan of 3 to 5 years from construction, the stiffness of CBTM courses increased as a result of curing phenomena. During the next 3 years of pavement exploitation, the average 55% decrease in CBTMs' stiffness was observed due to traffic-related damage.

Recent works on the application of CRMs in road construction indicate that due to the variability of possible types and contents of binding agents that can be used to produce cold recycling mixtures, their mechanical response may significantly vary even within a single CRM classification group. In the research performed by Dołżycki and Jaskuła [39], 56 road sections with base courses constructed in cold recycling technology were examined after 10 years of exploitation, after the occurrence of transverse reflective cracking. It was observed that insufficiently detailed mix composition recommendations, non-suitable quality acceptance criteria and therefore, inappropriately chosen contents of cement (up to even 7%) and bitumen emulsion (below 3%) in CRMs resulted in creation of overly stiff base layers prone to shrinkage cracking which induced transverse cracking of the pavement. Therefore, current trends in the design and construction of road surfaces with CRM layers are to design materials with a predominant role of bituminous and not cementitious bonds.

2.2. Bitumen Stabilised Materials

2.2.1. General characterisation

Bitumen Stabilised Materials are cold recycling mixtures that consist of RAP, virgin aggregates, bituminous and cementitious binding agents and water. As stated in Chapter 2.1.3, the quantities of residual bitumen derived from bitumen emulsion or foamed bitumen do not typically exceed 3% by mass of dry aggregate in the mixture, however, the experimental use of higher dosages of bitumen in BSMs' – bringing them closer to CMA – was also reported [70] [82]. A limited amount of active filler such as cement or hydrated lime (up to 1% by mass) can be added to achieve a certain level of tensile and compressive strength as well as permanent deformation resistance of BSMs without compromising on the flexibility of the layer. Thanks to such combination of binding agents, the bituminous binders – bitumen emulsion or foamed bitumen - play a predominant role in the development of the shear properties of the material. BSMs are characterized by a lower stiffness and rigidity, higher temperature sensitivity as well as lower susceptibility to cracking and improved shear properties, compared to other cold recycling mixtures with higher active filler share, such as CTMs and CBTMs [47] [59] [67].

BSMs can be produced using in-place (PDR or FDR) and in-plant (CCPR) recycling techniques and are most commonly used to construct base layers of flexible pavements. Wide variety of treated RAP materials can be used to produce BSMs: bituminous pavement courses, as well as granular layers or cement-treated base materials. Regardless of the used cold recycling technique, multiple fractions of virgin aggregates can be also added in BSMs to obtain a suitable grading curve with a sufficient amount of fine particles, which is vital for satisfactory bitumen distribution in the mix. The main feature that distinguishes BSMs' internal structure from HMA is that the residual bitumen does not create a continuous mastic phase in the mix [83]. Instead, due to the processes of bitumen emulsion breaking or foamed bitumen dispersion, localized mastic bonds in the mixture are formed with the bitumen distributing preferentially amongst the fine particles, but not exclusively. In the case of BSMs with bitumen emulsion, also coarse aggregate particles obtain some residual bitumen coating. The examples of the cross-sections of BSM and HMA mixtures, with an indication of the local bituminous bonds in cold recycling mixture, are shown in the Figure 4.

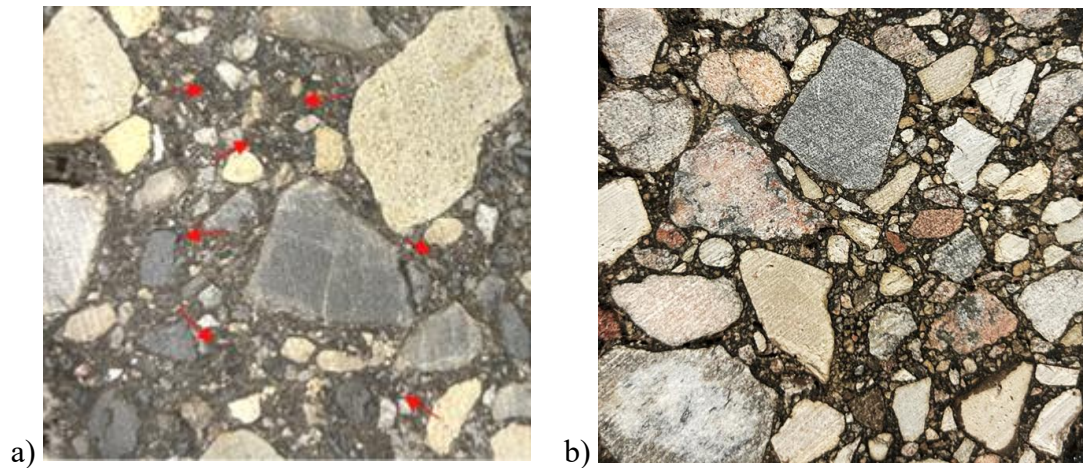


Figure 4 Sample cross-section: a) BSM mixture with locally dispersed bitumen, b) HMA mixture with a continuous bitumen film (photos of the Author)

Due to the specific, non-continuously bound structure of BSMs, they are considered to exhibit a combined mechanical properties of both granular and viscoelastic materials. However, no complete agreement regarding the mechanical performance of BSM mixtures has been achieved so far, which leads to a diverse approach to testing and describing the mechanical behaviour and failure mechanisms of BSM mixtures, as well as the design principles applied in construction of pavement structures with BSM layers.

Many literature sources emphasize the similarity of the mechanical properties of BSM mixtures to the properties of unbound materials, with BSM mixtures characterized by an improved stiffness and shear performance – significantly increased cohesion (up to approximately 6 times) and practically retained internal friction angle compared to the stabilised parent aggregate [84]. Moreover, BSMs exhibit reduced moisture sensitivity compared to parent granular material [10]. Following this approach, a stress-dependent mechanical behaviour of BSMs is assumed, with the mixture's properties being characterized utilizing test methodologies primarily applied to granular materials and cohesive soils, e.g., monotonic and dynamic triaxial testing [85]. As stated in the design guidelines [10] [17], the failure mechanism of permanent deformation is assumed for Bitumen Stabilised Materials in the pavement design process. Schematic comparison of the internal structure of the unbound materials and BSM mixtures is shown in the Figure 5.

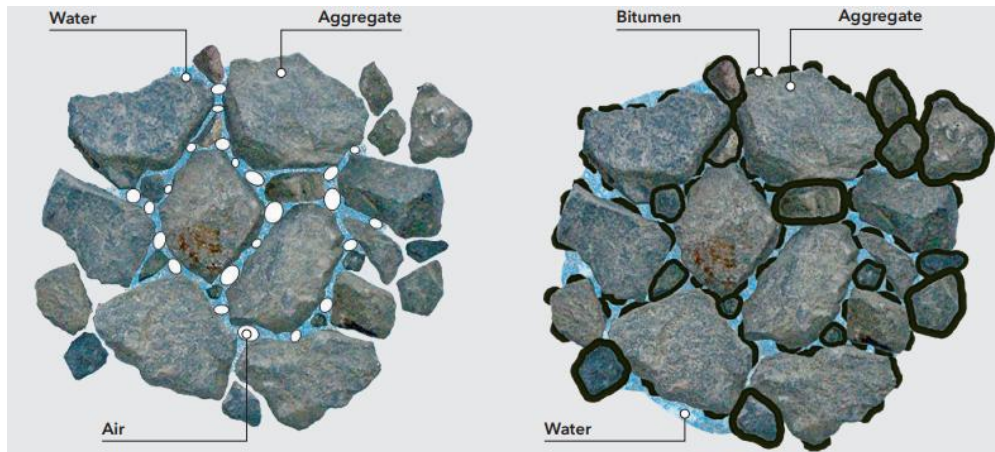


Figure 5 Unbound granular mixtures (on the left) vs. non-continuously bound Bitumen Stabilised Materials (on the right) [10]

On the other hand, as indicated by international research conducted to this date, BSMs can also display performance similar to bound, viscoelastic mixtures, given their inclusion of bitumen and a limited cement content [83] [86]. The verification of the time- and temperature-dependency of BSMs is carried out using test methods originally developed for HMA mixtures, with appropriately adapted test conditions and parameters to perform testing in the linear viscoelastic domain. Based on the conducted research, Bitumen Stabilised Materials exhibit time- and temperature-dependency, indicating the viscoelastic characteristics of these mixes. However, the influence of testing frequency and temperature on the dynamic modulus of BSMs is lower than that in HMA [87] [88].

Due to their specific mix composition (i.e. presence of water and cementitious binders), BSM mixtures, unlike HMA, are reported to present an evolutive behaviour during service time in road construction [89] [90] [91]. These changes in the BSM layer's mechanical and volumetric properties (e.g., resilient and dynamic modulus values, air void content) are predominantly caused by the curing phenomenon, related to the natural reduction of the moisture content, while being influenced by the cementitious binders' hydration process [92]. Currently, several leading, and sometimes contradictory, hypotheses in the literature regarding the evolution of BSM mixtures' properties can be found, indicating a wide variety of mechanical behaviour that can be observed over time – from an elasto-plastic response of stress-dependent materials to a viscoplastic or viscoelastic one, characteristic for bound asphalt mixtures.

2.2.2. Role of components

2.2.2.1. *Reclaimed Asphalt Pavement and virgin aggregates*

SELECTED PHYSICAL CHARACTERISTICS

Reclaimed Asphalt Pavement is defined as “*removed and/or processed pavement materials containing asphalt binder and aggregate*” according to the AASHTO MP31-22 [S2] standard. The reclaimed material is obtained in the cold planning and milling processes using milling machines or removed in larger slab sections with the use of bulldozers or excavators and then processed accordingly to the project needs. The quantitative and qualitative characteristics of the material recycled from an existing pavement vary considerably depending on the structure (material quality, mixture type and thickness of pavement layers), as well as age, degree of deterioration and the applied rehabilitation techniques to the existing pavement [10] [17] [93]. Since cold recycling technology is performed using different reclamation depths and, therefore, can be applied to a wide range of pavement layers, from subgrade, subbases or granular base layers to binder and surface courses, the Reclaimed Asphalt Pavement can include materials that are derived exclusively from the bituminous HMA layers, or additionally include material coming from unbound granular layers or bound cement-stabilised materials.

It should be also noted that RAP, as a parent material used in BSMs, displays different surface characteristics and mechanical properties compared to virgin aggregates. Since the RAP particles consist of conglomerates of aggregates and mastic containing aged and stiff binder, they are considered to create structurally different coarse particles’ skeleton in the cold recycling mixture than in the case of natural or crushed aggregates, which consequently affects the mechanical response of CRMs [94]. Therefore, two main issues should be considered in the cold recycling mixtures design process – the influence of RAP content in the mineral mixture on the mechanical performance of the mixture as well as the possible interaction between the aged binder from RAP and the fresh residual binder and its effect on the properties of CRMs.

The issue of homogeneity of the reclaimed mineral material plays an important role in the development of final properties of CRMs. As reported by Tebaldi et al. [8], homogeneity of RAP considered in terms of aggregate size, mineral rock type, binder type and content is a vital criterion that needs to be considered in the cold recycling mixtures design process as one of the factors ensuring predictability as well as reproducibility of both mechanical and volumetric properties of the designed mixes.

Similarly to warm and hot recycling mixtures, the phenomenon of blending of the “old” binder from RAP with a “fresh” binder added to the mix still remains insufficiently explored.

Since cold recycling mixtures are produced at ambient temperatures, it is generally acknowledged that RAP is introduced into the mix as an aggregate and therefore, its “black curve” is taken into account in the mix design. However, there is still no consensus regarding the “activity” of RAP which is connected to the capacity of an aged binder present in aggregate-mastic clusters to glue the particles together after compaction as well as its ability to participate in the creation of local bituminous bonds together with the residual bitumen in the short- and long-term exploitation perspective of the CRM layer.

Researchers proposed testing methods to determine the activity of RAP particles which are based on both properties of RAP and bitumen extracted from recycled pavement. Tebaldi et al. presented a cohesion assessment method carried out on 100% RAP samples subjected to Indirect Tensile Strength tests [8] [95] [96]. In Wirtgen Cold Recycling Manual [10], the assessment of the penetration value of the recovered binder is performed. RAP with binders of the penetration lower than $18 \cdot 0.1$ mm is treated as inactive, while the activity of the reclaimed material is potentially considered for penetration values above $25 \cdot 0.1$ mm. The additional, organoleptic assessment of RAP activity can be performed considering the visual appearance, brittleness and adhesion of the RAP particles [10]. However, since the processing temperatures applied in cold recycling technology are significantly lower than those required to soften the severely aged bitumen, the amount of RAP binder is generally not considered in calculating the total bitumen content in CRMs [10] [97].

In one of the earliest reported research, Mamlouk and Ayoub [98] evaluated the long-term behaviour of artificially aged cold recycling mixtures containing bitumen emulsion and concluded that the emulsion did not have a long-term softening effect on the aged binder in RAP. The phenomenon of the RAP bitumen activity and the influence of different cold recycling aggregates on the mechanical properties (ITS) of BSMs with bitumen emulsion and foamed bitumen were investigated as a part of the international CoRePaSol project [99]. The research showed that the increase in the content of RAP resulted in increased ITS values of the mixture, which can be interpreted as "activity" of the aged RAP binder and may be considered in mix design studies. On the other hand, the RAP content negatively affected the compactability and California Bearing Ratio (CBR) values of BSM layers. Research conducted by Kim [100] [101] showed that stiffness of BSM-FBs (referred to as CIR-foam) was not only influenced by the foamed bitumen content but also by the content and state of aged RAP binder. It was stated that the stiffer the aged RAP binder, the higher is the optimum residual bitumen content in the cold recycling mixture.

Virgin aggregates – coarse and fine crushed aggregates or fillers - are added to CRMs to adjust the mixtures' grading curve to the recommended grading by supplementing missing fractions in RAP. Requirements for the aggregates' properties depend on the recommendations and guidelines applied in each country [62]. Nevertheless, the most important characteristics of the virgin aggregates considered in CRMs' mix design are the type of the parent rock material and particle size distribution. In BSM-Es with cationic bitumen emulsions a satisfactory level of the aggregate-residual bitumen adhesion can be obtained both in the case of acidic and basic rocks. However, due to the chemical nature of the emulsion breaking process, acidic rocks with silica contents above 65% and alkali contents below 35% such as quartzite, granite, rhyolite or sandstone are not suitable for treatment with anionic stable grade bitumen emulsions, which are mostly used in cold recycling in warm and dry climates (e.g., in South Africa and North America) [102].

GRADING

Suitable grading of the mineral mixture is required to secure the desired mechanical and volumetric properties of BSMs. Grading envelopes, which are specified in guidelines and mix design recommendations, vary depending on the country or region where cold recycling is performed [103] [61]. The factor that most influences the creation of an "aggregate interlock" in BSMs is the fine particle content (< 0.063 mm or < 0.075 mm), which plays an important role in the case of BSMs cohesion development. Due to the differences in the way the residual bitumen is distributed in Bitumen Stabilised Materials produced with foamed bitumen and bitumen emulsion, the required threshold values for the amount of fines slightly differ for each bituminous binder. According to literature sources and different national recommendations [13] [61] [63] [103], the recommended range of fines content in BSMs generally varies from 2% to 10%, with exceptions in the form of Spanish and Portuguese specifications for BSMs [104] [105], where the recommended content of fines in recycled material is within the range of 0%-3%, and South African recommendations [17], which allow the use of mineral material with a maximum content of fine fractions of 20%. Moreover, in the TG2 Asphalt Academy specification [17] the use of the Fuller-Thompson equation originally proposed for concrete [106] was recommended to provide the maximum density gradation for a given maximum aggregate size and therefore achieve the minimum value of voids in the mineral aggregate (VMA) in the BSM mix. The recycled material's gradation (especially information on the amount of fractions < 4.75 mm) is also used as an indicator while determining the optimum content of bituminous binders.

The obtained grading of the reclaimed pavement material depends on the used recycling technique, depth of reclamation, milling equipment and operation conditions as well as further treatment and handling of the obtained recycled material. Since RAP consists of aggregate-mastic clusters that can be broken during transportation, paving and compaction processes and are sensitive to temperature variations during storage and mixing, its aggregate size distribution may evolve during technological processes. This phenomenon should be particularly considered in the case of asphalt layers showing structural heterogeneities and a high degree of deterioration, in which the recycled material is likely to exhibit low resistance to fragmentation [8]. To ensure the quality of RAP coming from a particular source, one of the simple testing methods proposed by RILEM Technical Committee TC-237 SIB is the fragmentation test [107] [108]. A way to reduce the impact of RAP material heterogeneity on the performance of CRM mixes is using the CCPR recycling technique, in which the RAP hauled from the construction site to the central plant is processed using a series of operations, including crushing and screening to obtain a suitable gradation and eliminate foreign matters and contamination.

The introduction of virgin aggregates into the mixture may be necessary to obtain the mixture grading within the required grading envelope. Selected fractions of coarse and fine crushed aggregates, mineral fillers or aggregates with a continuous gradation can be added to the mixture when the mineral mixture containing 100% RAP is characterized by an inappropriate gradation (usually low amount of fine particles [10] [109]) or the designed BSM mixtures contain only a limited amount of Reclaimed Asphalt Pavement.

PLASTICITY

The Plasticity Index (PI) is one of the basic geotechnical parameters of cohesive materials (soils) that refers to the amount of clay present in the material and is defined as a range of moisture contents over which the material deforms plastically. Based on this parameter, one can conclude about the degree of material cohesion [110] [111]. In the BSM mix design process, PI is used as a guideline for using cement or hydrated lime [10] [17]. High clay content in fine fractions of the recycled material is unfavourable in the context of the development of bituminous bonds in BSMs, which highly rely on fines when the residual bitumen dispersion is considered. Due to the ability of clay to retain relatively high levels of moisture, it exhibits the tendency of agglomeration into lumps, which consequently prevents the effective breaking of bitumen emulsion on filler particles in the mix. Moreover, the agglomerates of clay which are highly moisture susceptible are considered as discontinuities that weaken the structure of the mixture. Since clay particles are included in the <0.063 mm or <0.075 mm particle fraction,

not only grading and fines content, but also the PI value of the mineral material should be carefully verified in the design process.

2.2.2.2. Bituminous binders

Bitumen can be introduced into the BSM mixture in the form of bitumen emulsion (BE) or foamed bitumen (FB). Since the subject of the dissertation are Bitumen Stabilised Materials with bitumen emulsion, the emphasis will be placed on describing the mechanism of creating bituminous bonds in the material for this specific type of binder.

The manufacturing process of a typical bitumen emulsion, which is an oil-in-water (O/W) system with water (or soap) as the continuous phase and bitumen as the dispersed phase is illustrated in Figure 6.

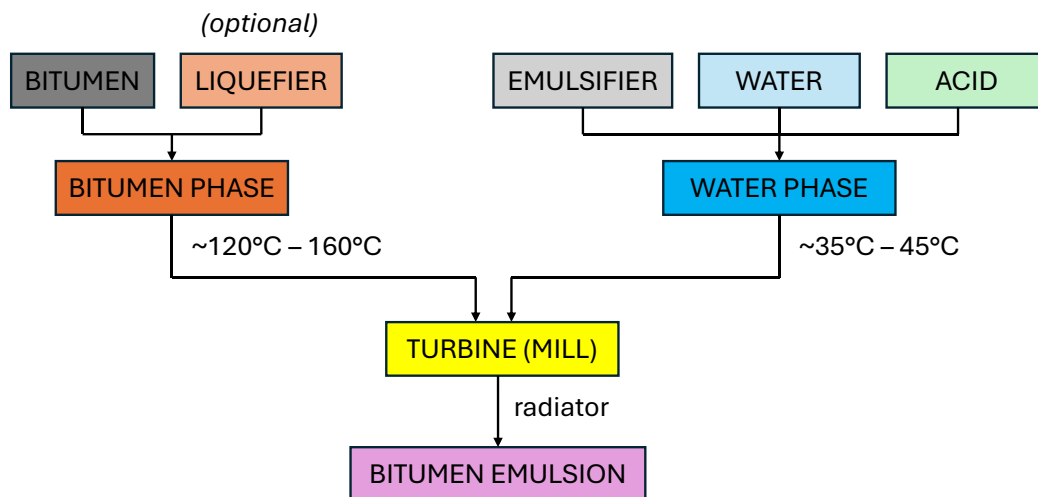


Figure 6 Bitumen emulsion production process (adapted from [112])

The bitumen content in bitumen emulsions commonly used for BSM production ranges from 60% to 70%. The emulsifier's (surfactant's) basic function is to reduce the surface tension between the bitumen and water, thereby preventing the bitumen droplets' coalescence and stabilising the system. Depending on the type of the surfactant used in the production process, the bitumen emulsion can be classified as cationic, when fatty amines (e.g., imidazolines, amidoamines) are incorporated, or anionic, when the fatty acids (e.g., lignin, tall oil) are used [113], [114]. In cationic emulsions, bitumen particles are surrounded by a positive charge, which enhances their adhesion to negatively charged mineral surfaces, while in the anionic ones, bitumen particles acquire a negative charge, making them more compatible with positively charged mineral surfaces. Due to the differences in the charges of the bitumen droplets present in BEs, each aggregate-bitumen emulsion combination results in a distinct adhesivity performance, as previously mentioned in section 2.2.2.1. Nevertheless, cationic bitumen emulsions are generally more versatile for use in cold recycling mixtures as they can

work effectively with a wider range of aggregate types, including both negatively and positively charged materials [113]. The other reason for the cationic bitumen emulsions being preferred in BSMs, is that the positively charged bitumen droplets are much more likely to be adsorbed to cement grains in comparison with those in anionic bitumen emulsions [115]. It should be also noted that the bitumen type and source are paramount parameters in the chemical interaction of the surfactants with the bitumen in bitumen emulsion production, mainly in terms of their stability [113]. Bituminous binders with lower penetration values present greater challenges in achieving stable emulsions due to their higher viscosity. They require higher processing temperatures and more effective emulsification techniques to ensure uniform dispersion within the aqueous phase. Conversely, softer bitumen with higher penetration values are easier to emulsify, as they can be more easily broken down into fine particles, exhibiting a potential for an improved stability of the emulsion.

BE plays an essential role in the development of Bitumen Stabilised Materials' volumetric and mechanical properties on both levels of the "fresh mix" and the final mixture. During the BSM production stage, bitumen emulsion contributes to fluids for compaction, influencing the workability and compactability of the mix, which results in specific initial density and pore structure of the material [116]. The process of creation of the localized non-continuous bituminous bonds in the material takes place due to the bitumen emulsion breaking process, where the bitumen separates from the water phase as a result of physical (water evaporation) and chemical phenomena. In BSM-E mixes, the bitumen emulsion disperses preferentially amongst the finer particles, but not exclusively – coarser virgin aggregate and RAP particles can obtain additional surface bitumen coating, unlike for the BSM-FBs, where foamed bitumen distributes exclusively to the finer particles, producing "spot welds" of a mastic of bitumen droplets and fines [17]. Based on the above statement documented in the literature sources, it can be hypothesized that due to the specific nature of bonds in BSM-Es (localized mastic bonds and partial, semi-continuous film on the coarser particles), undertaking research on the assessment and development of their viscoelastic properties is a more justified direction than in the case of BSM-FBs. Following the unbound material approach, it is the bitumen emulsion addition that increases in the cohesion of the parent granular material, while the specific nature of bituminous bonds contributes to keeping the angle of internal friction on a similar level [10].

For cold recycling mixtures, which are characterized by a bitumen/cement ratio higher than 1, such as Bitumen Stabilised Materials, it has been well-acknowledged that the bituminous phase becomes dominant in the structure of the mixture with cement hydrates

playing the role of dispersed hard inclusions [117]. Bitumen emulsion is responsible for creation of bituminous bonds, and – as a result of increase in cohesion of the material – its resistance to the moisture-induced damage. It also provides sufficient mixture's flexibility, which should lead to minimization of the risk of shrinkage cracking of BSM layers in construction, common for cement-treated materials [59]. According to the Polish requirements for cold recycling mixtures design, the minimum bitumen emulsion content recommended is 3% [44], while the Wirtgen Cold Recycling Manual [10] does not present precise requirements regarding the minimum bitumen emulsion content. As hypothesized by Liebenberg and Visser [65], an increase in the residual binder content in excess of 3% has a positive influence on the ITS value regardless of the cement content, with the possibility of BSMs behaving in a viscoelastic manner with similar properties to that of asphalt mixtures. Moreover, following the BSM mix design procedures presented in the leading guidelines [10] [17], the knowledge of the influence of the bitumen emulsion content on the Indirect Tensile Strength is a base for determining the optimum BE content in the mixture. Moreover, the influence of BE content on the mixture's durability characteristics – i.e. moisture susceptibility - is expressed by the Tensile Strength Retained (TSR) parameter. As reported by Jenkins et. al [26], Bitumen Stabilised Materials with higher residual bitumen contents are characterized by greater flexibility but lower resistance to permanent deformation. In the work of Miljković [118], the influence of not only the amount of bitumen emulsion but also its characteristics (i.e. emulsifier content) on the mechanical performance of cold recycling mixtures with BE was emphasized. It was stated that at lower emulsifier contents, premature BE breaking led to a fast water loss, and possibly to insufficient level of cement hydration in the bitumen emulsion mortars and mixtures at the end of the curing period. Furthermore, this resulted in poor adhesion of the residual bitumen at the interface to the aggregate, and finally in inferior mechanical performance of the tested composites.

2.2.2.3. *Active filler*

Within the term “*active filler*” one can distinguish a group of fillers that alter the BSMs' properties chemically, namely: slow and normal hardening types of Ordinary Portland Cement (OPC), slag cement and supplementary cementing materials (SCMs) such as hydrated lime, and fly ash. The amount of the active filler should not exceed 1% of the mixture (maximum of 1.5% for hydrated lime) [10]. Main advantages for incorporating a limited active filler's dosage in BSMs are the improvement of bitumen-aggregate adhesion, providing a satisfactory residual bitumen dispersion level in the mix, increase of the initial stiffness and strength gain rate of the

mixture due to acceleration of the curing process, as well as improvement of the mixture's water resistance without affecting the flexibility of the material [119] [120].

The observed role of active filler in the bitumen dispersion in BSMs is slightly different in the presence of bitumen emulsion and foamed bitumen. Since the subject of this work are BSMs with bitumen emulsion and the most commonly used active filler is cement, the analysis presented in this chapter will be limited to the phenomenon of cement – bitumen emulsion interaction during formation of bituminous bonds, which has not been fully explained to date. Understanding of this mechanism is an integral factor in development of design procedures for high-performance BSM-Es. It is generally acknowledged that cement assists the extraction of the water phase from a bitumen emulsion, promoting breaking and therefore supporting the creation of the intergranular bituminous bonding in BSMs [121] [122]. The results of the meso-scale testing mixtures of bitumen emulsion and cement presented in the work of James et al. [123], showed that OPC performs a dual role in increasing the BE breaking rate and stiffening the residual binder in the cold recycling composites. The higher the OPC addition level, the faster the BE breaking occurs. The penetration of the residual bitumen containing cement composite was approximately 10% lower than that of the original binder recovered from BE when 1% OPC is used. The interaction between cement and bitumen emulsion was studied in further detail by Pouliot [124]. The research demonstrated that the cement hydration process is nominally influenced by adding of a small quantity of bitumen emulsion. The introduction of bitumen droplets inside a cement mortar matrix led to a significant reduction in compressive strength and elastic modulus as well as a slight decrease in flexural strength compared to reference cement composites. As stated by Du [121], in theory, the cement content controls the elastic modulus whereas the bitumen emulsion content provides the strain capacity of cold recycling mixtures. Yang et al. [125] investigated the performance and microstructure of CRMs with bitumen emulsion and 0%–5% cement. The findings presented for mixtures containing 1% cement allow to conclude that a limited addition of cementitious binder positively influences the low-temperature cracking resistance compared to CRMs with higher cement dosage (3%–5%). Moreover, the high-temperature stability and moisture damage resistance beneficially increases when even small contents of cement are added. The SEM microstructure analysis and the energy spectrum analysis revealed that for low cement contents, the structure of the CRM mortar is mainly constituted by the bituminous phase, with cement hydration products being enwrapped by residual bitumen from bitumen emulsion and the proportion of hydration products increasing gradually with an increase in the cement content.

2.2.2.4. *Moisture*

Water is a component that significantly distinguishes Bitumen Stabilised Materials from HMA. The influence of moisture content on the mixture properties in fresh and cured state is significant for both BSM-Es and BSM-FBs. However, due to a different physical form of bitumen emulsion and foamed bitumen, a different role of fluid components in development of the mixture characteristics is observed. Since the subject of this thesis are BSM-Es, the effect of moisture coming from the emulsion and additional water on the short- and long-term BSM cohesion behaviour will be further analysed in this section.

The main roles of water in BSM-Es can be identified as follows [10] [17] [23]:

- A reducer of the absorption of bitumen emulsion water into aggregate,
- A prevention aid for premature bitumen emulsion breaking,
- A lubricant for the friction angle reduction, providing the mix workability and satisfactory compactability at ambient temperatures,
- BSM curing time extender.

The Optimum Fluid Content (OFC) determined at the laboratory design stage is considered the required content of moisture to achieve the Maximum Dry Density (MDD) of the mixture. Most commonly used methodologies of the OFC determination are the modified AASHTO T-180 [S3] and modified Proctor EN 13286-2 [S4] test procedures. Depending on the design recommendations, different formulas are used to represent the OFC components and therefore determine the amount of water to be added during laboratory mixing [23]. According to the leading BSM design manuals [10] [17], the OFC is defined as the sum of the total amount of bitumen emulsion applied (not only the water fraction - approximately 40% of BE mass), the moisture content of the material itself and any other water applied independently of the water fraction of BE. On the other hand, the Polish cold recycling mixture design recommendations [44], apart from the water externally applied to the dry components, include the water fraction of BE but only half the amount of the bitumen from emulsion as fluids contributing to OFC. Considering the BSM-Es' optimum mixing moisture content (OMMC) and the optimum compaction moisture content (OCMC), the total fluid content of the material can be equal and should not exceed the OFC.

In general, two distinct phases of changes in the BSMs' moisture content are identified: bitumen emulsion breaking and curing [10] [17]. The first phenomenon has been described in the 2.2.2.2 section. The curing phenomenon, directly linked to the elimination of BSMs' moisture due to evaporation particle charge repulsion and pore-pressure induced flow paths [10]

[126], is the main influential process for the development of bonding between the residual bitumen and mineral aggregate interfaces and consequently building of mechanical, cohesive and durability properties [93]. It is well-acknowledged that BSMs' mechanical properties are closely related to the moisture content, which changes over time and should be monitored on an ongoing basis during the production and paving process. Firstly, the reduction in moisture content leads to an increase in the tensile and compressive strength, and the stiffness of BSMs [10]. With the limited short-term stiffness being a vital issue in BSM field applications in terms of permanent deformation failure, it should be noted that for cold recycling mixtures, at an early stage, it is the inner particles frictional resistance and cement hydration that provide the material strength, while the bitumen coating formed in the interface after water evaporation provides a higher strength for CRME after curing [127]. It is due to the fact that the residual bitumen particles start to establish "bridges" among the virgin aggregate and RAP particles, acting as a bituminous binder that holds the granular particles in place, desirably creating a strong adhesive bond. In the PhD thesis, Twagira [90] indicates the influence of temperature as a powerful accelerating factor for water evaporation, as well as the impact of air void content on the moisture transport in the mixture and highlights the connection between the moisture evaporation and curing phenomena and the evolution of adhesion and cohesion in BSMs. Moreover, due to the fact that water works as a lubricant in BSMs facilitating the homogenization and compaction of the fresh mix, its content influences the level of mix densification in the fresh state, and consequently has an impact on the air void content in the cured mixture. It is generally stated that BSMs will only present their final characteristics when curing process is concluded, indicating the evolution of the mixtures' properties over time and posing a challenge in describing and modelling their behaviour for long-term application purposes.

The field moisture-induced changes in the BSMs' properties are simulated in laboratory by application of the specific curing procedures to the specimens in both mix design and testing stages. Due to a limited amount of cement in the BSMs' composition, the majority of sources recommend applying the accelerated curing procedures with various set of conditions, including the dry-mass oven drying at temperatures ranging from 40°C to 60°C, optionally combined with temporary sealing of the samples [10] [17] [23] [128].

2.2.3. Application in road pavements – advantages, challenges, perspectives

The main benefits of the application of cold recycling mixtures in road construction, such as the reduction of material transportation costs and virgin materials' consumption, minimization of the hazardous substances' emissions during production and paving or technical possibility of reusing tar asphalt layers, can be also attributed to the case of Bitumen Stabilised Materials. When compared to cold recycling mixtures treated with higher amounts of cementitious binders (CBTMs or CTMs), the application of more flexible BSMs in construction offers an advantage of minimization of the transverse shrinkage cracking occurring in flexible pavements [59] [10]. Multiple cases of the use of BSM mixtures in flexible pavement construction as base and wearing courses have been reported in recent years. In 2018 in South Africa, the Camps Bay Drive was successfully rehabilitated using BSM produced with the CCPR in-plant technique. 8,150 tonnes of RAP were used to construct a 100% RAP BSM-FB base layer with 2.1% foamed bitumen and 1% cement. The 200-mm layer was opened to traffic up to 24 hours after construction and its acceptance control was carried out by means of Indirect Tensile Strength (ITS) and Minimum Dry Density (MDD) tests [129]. Positively verified CIR rehabilitation of a heavy-trafficked semirigid highway pavement with the use of cold recycling mixtures containing foamed bitumen and 1% of cement was described by Loizos and Papavasiliou [80]. A successful 4,350-km link road widening in France, Yvelines, using the cold recycling in-place technique with 100-mm BSM base layers with foamed bitumen (2.4%) and lime (1%) was performed by Wirtgen and Colas companies in 2021. As reported by the contractors, completion of the recycling process in one single pass allowed for a significant simplification of production logistics, with savings of approximately 20 trucks an hour taking away the recycled material and 11,000 tonnes of the recycled material itself that would have had to be moved using traditional rehabilitation techniques [130]. In 2022, an emulsion-based BSM was applied to rehabilitate residential roads in the Coventry County (United Kingdom), where 9,500 m² of the pavement was recycled using the Recycol® in-situ recycling solution developed by the COLAS company. According to the greenhouse gas emission calculations performed, the application of 70-mm BSM layers resulted in 70% carbon (CO₂ eq/m²) savings over the application of the traditional HMA solutions, considering the aspect of the materials' extraction, transport and mixture manufacturing [131]. In the work of Filho et al. [132], the early findings from a CCPR section built with a 100% RAP BSM base course with bitumen emulsion and no cement were presented. After 1.5 years of service, good structural capacity of the layer in terms

of stiffness, as well as no signs of rutting distresses influencing the driving quality were observed.

Considering the above-mentioned examples of road rehabilitation using BSM mixtures, it can be concluded that currently a significant increase in interest in the use of this technology in rehabilitation and road surface construction is observed. However, what should not be omitted, is the fact that the application of BSMs in pavement construction is connected to facing specific challenges, related to the material and technological aspects. One can distinguish several factors influencing the performance of BSM layers in construction in the short-term and long-term perspective, e.g., quality and homogeneity level of the recycled underlying layers, the appropriateness of the selected type and content of bituminous and cementitious binders, as well as the density, thickness and uniformity of the constructed BSM layer [10] [93]. The main concerns are the possible variations in CRMs' volumetric and mechanical properties due to high heterogeneity of the recycled material, the need for understanding and monitoring changes of the mechanical response of the material over time, as well as – from a technological side – an extension of the pavement construction process compared to the traditional HMA technology as an additional time is needed for the layer to gain initial strength before being covered with subsequent pavement layers [25] [133] [134] [135]. Due to a fundamentally different mix composition to HMA, BSM layers are characterized by a slow (BSM-Es) to medium (BSM-FBs) rate of initial strength gain which is closely connected to the moisture loss. Depending on local pavement design regulations, a specific time and conditions have to be applied to effectively carry out the paving process and minimize any traffic delays. Premature opening for traffic before reaching sufficient strength and stiffness of the BSM later may lead to reaching failure conditions in the form of permanent deformation, creating an irreversibly negative effect on the structural and functional state of the pavement [59]. Therefore, conditions influencing the amount of moisture present in BSM layers, such as effectiveness of pavement's drainage as well as climatic conditions should be taken into account when applying BSM materials into pavement structure. BSM mixtures are extremely sensitive to environmental weather conditions during the production and paving processes. Firstly, it is since the construction of BSMs' base layers is carried out at ambient temperatures without additional heating, and secondly, due to the presence of water in the paved BSM layer. The temperature of the reclaimed material influences the obtained degree of aggregate particle coating with bitumen, especially in the case of BSM-FBs [17]. Summarizing the recommendations presented in the international guidelines [10] [17] [44], BSM-E and BSM-FB mixes should not be produced and compacted at temperatures below 5°C

(with an aggregate temperature above 10°C). Following these conditions helps to ensure the proper bitumen distribution, aggregate particle coating and therefore satisfactory mixture performance. Nevertheless, it imposes restrictions related to the season in which works can be performed.

Moreover, the evolutive behaviour of BSMs considering changes in stiffness and volumetric properties over time, connected to a specific binder agents composition and moisture content variations, is still insufficiently explored. The lack of comprehensive information on the long-term behaviour of these materials and uncertainty regarding their failure mechanisms contribute to slowing down the development of this technology, which consequently complicates the process of pavement design with BSM mixtures.

In summary, while cold recycling techniques incorporating BSMs are well established and their advantages and limitations generally understood, there remains a need for further data on mechanical performance, rutting resistance, failure mechanisms, and long-term durability. A thorough understanding of the material's nature and the evolution of its mechanical behaviour is essential for effective engineering and reliable prediction of BSM properties

3. Performance of Bitumen Stabilised Materials

The mechanical performance of Bitumen Stabilised Materials is still insufficiently researched and explained. Despite the fact that there is a consensus about the material being non-continuously bound due to the presence of bituminous and optionally cementitious binders applied in a cold manner, the views on the material's mechanical behaviour presented in scientific research, national standards, and requirements as well as road agencies' reports significantly differ. Due to a heterogeneous and complex structure, BSMs exhibit a mixed mechanical response typical for granular unbound materials, visco-elasto-plastic and viscoelastic mixtures [89] [136] [137] [138]. Thus, the BSMs' possible failure mechanisms in pavement structures are still being analysed, with phenomena of permanent deformation, moisture damage and fatigue cracking being taken into consideration. Moreover, particular research attention is paid to the curing-linked and material-dependent evolution of the properties of these materials in time, under repeated loading and changing climate conditions [65] [89] [139]. Due to the similarity of the material properties, in the literature review presented below, both BSM-E and BSM-FB mixtures were considered.

3.1. Evolutive behaviour – hypotheses and previous research

Over the years of research on Bitumen Stabilised Materials, several hypotheses have been developed regarding their evolutive behaviour in pavement structures. It has been generally stated that the changes in the BSMs' mechanical and volumetric properties in construction occur due to their specific mix composition – mainly the progressive reduction of the moisture content (curing) of the layer and long-term chemical and physical interaction between bituminous and cementitious binders [116] [140]. Despite collection of a considerable amount of data on changes in the mechanical response of BSMs both in laboratory testing and pavement testing performed in situ, the phenomenon of the evolutive behaviour remains insufficiently understood [141]. It should be noted that the overall laboratory assessment of the BSM properties evolution is problematic due to the lack of unified specimen curing requirements, which could reliably reflect the material's curing conditions in the field [132]. Moreover, the variety of the cold recycling layers' composition, curing conditions and monitoring timespans in field testing do not allow for a comprehensive comparison of the BSMs' mechanical response over time. Based on the conducted literature research, two main scenarios considering the changes in the mechanical performance of BSMs in road pavements can be found, each made up of two clearly defined phases, but of a contradictory nature.

The first theory, presented by Liebenberg and Visser [65] [139] in the early 2000s, assumed that during the service life, Bitumen Stabilised Materials (referred to as *emulsion-treated materials*) present response similar to that of lightly cemented materials and experience an Effective Fatigue Phase, followed by a post-cracked, Equivalent Granular Phase. In the first stage, the fatigue life of the material and its flexibility based on the strain-at-break parameter are analysed. The stiffness of the material gained after construction and dependent on the cement content, is assumed to gradually diminish until reaching the terminal elastic modulus of approximately 25% of the initial value. After the fatigue failure, the material is supposed to approach the second stage, in which the mechanical response is similar to that of unbound granular materials, with the shear strength parameters determining the materials' failure due to permanent deformation.

However, subsequent research have questioned such development of the BSMs' mechanical properties. In the vast majority of case studies, in which the falling weight deflectometer (FWD) was used to evaluate the structural capacity of pavements rehabilitated utilizing BSMs, the gradual increase and subsequent stabilization of the BSM layers' stiffness rather than a decrease after construction were observed [67] [80] [133] [141] [142]. As stated in the work of Ebels [91], the BSMs' fatigue only occurs after substantial time, while most of the permanent deformation takes place in the pavement's early life. Therefore, a more reliable, second hypothesis based on the in situ gathered data was developed. In the first, curing phase, BSMs exhibit stress-dependent behaviour, characteristic for unbound granular materials, with a systematic increase in stiffness due to moisture reduction. At this stage, the permanent deformation resistance plays a vital role in characterization of the material. In the second phase, however, BSMs display a quasi-viscoelastic behaviour with temperature and time dependency, resembling bound materials – HMA. At this stage, after the phase of stabilization, a reduction of stiffness over time is observed. Due to the non-continuously bound nature of the material, the mode of failure is still being investigated, with fatigue cracking considered as a possible, but not only, failure criterion [26].

The South African experience with BSMs in-service behaviour presented in the work of Jenkins [83], indicate that the evolution of the BSMs' stiffness is influenced by various factors, including the curing behaviour and traffic loading. The initial values of resilient modulus (M_r) of BSMs were observed to increase in the first year after construction, primarily due to curing behaviour manifesting in moisture reduction. It was observed that this phenomenon dominates the gain in resilient modulus during the early stages of a BSM's life. However, the long-term resilient modulus of BSMs shows signs of reduction under traffic loading, indicating the rupture

of the adhesion of the bitumen to the aggregate. This reduction in stiffness is likely a result of the spot welds formed by the bitumen being negatively influenced by traffic, leading to a decrease in the effective stiffness of the BSM layer, but with residual benefit of the bitumen stabilisation remaining evident.

Prediction of the evolutive behaviour of Bitumen Stabilised Materials presented in the work of Twagira [90], assumes the presence of the curing and stable phase of the material's stiffness development, as well as a stiffness reduction phase, with the nature of changes in the BSM properties depending on the long-term durability of the material (Figure 7). Moreover, the role of moisture damage in weakening of the BSM cohesion leading to material failure by rutting or fatigue was emphasized in the work.

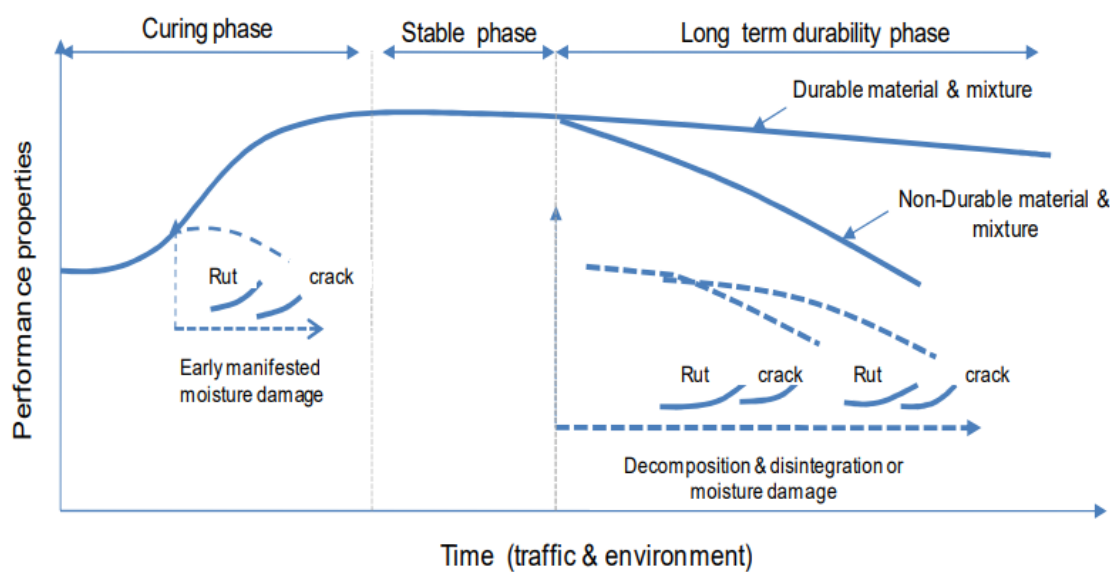


Figure 7 Prediction of the initial and long-term performance of BSMs [90]

In the work of Nivedya et al. [20], a phenomenon of densification of the BSM-FB layers due to traffic loading is also taken into consideration while predicting nature of changes occurring in the material's volumetric and mechanical properties. Particular attention was paid to changes occurring in the internal structure of the mixture – packing of the coarse aggregate skeleton consisting of both RAP particles and virgin aggregates. The approach presented in the research assumes a continuous evolution of the material's response from an elastoplastic at an early stage to a viscoelastic or viscoplastic one over the long term. The predominantly granular material response is expected to be observed immediately after laying and compaction, with the layer characterized by a high air void content and the shear resistance of the mixture mobilized mainly by the intergranular pressure and less by the bitumen mastic. As the layer densifies due to repeated loading, the air void content decreases reaching the “refusal air voids” level and the resistance to applied loading is mobilized by pressure in the aggregate skeleton with the

possibility of the binder-coated aggregate particles influencing the time- and temperature-dependent response. Moreover, a partial rebonding of RAP particles and bitumen mastic influencing the cohesive properties of the material is considered because of the healing nature of the binder and due to considerable amount of pressure to trigger such reaction. It was stated that at ambient temperatures, the material response can be dominantly pressure-dependent (stress-dependent), whereas at high temperatures, the material's behaviour can resemble the one of viscoelastic bound materials. The rheological model was originally developed to describe the two-staged response of the material during service life.

3.2. Failure mechanisms

Based on the current research conducted in the field of Bitumen Stabilised Materials, their possible failure mechanisms have been identified in direct connection to the assumed mechanical behaviour. However, no general agreement has yet been reached on this issue. While some sources point to the granular nature of the material, the basic failure mechanism of which is constituted by permanent deformation, the others indicate that the failure mechanism can be similar to that of cohesive, bonded materials, which is fatigue cracking [10] [17] [25] [26] [139] [143]. The existing inconclusiveness of scientific literature on the most likely failure mode of BSMs is connected to the inconsistency in the approaches applied to describe the mechanical response of these materials. Researchers point out that due to a wide variety of quality and quantity of the recycled pavement material and binding agents used to produce Bitumen Stabilised Materials, ideally, their failure performance should be examined individually for each project.

According to South African technical guidelines [17], the typical mode of failure of BSM layers is permanent deformation. In this case, the density level achieved during construction is critical to ensure the required structural capacity and permanent deformation resistance of the layer. Considering the evolutive behaviour of BSMs (as analysed in section 3.1), these mixtures are specifically prone to a premature permanent deformation failure at early stages of construction as a result of a traffic overload due to a relatively low initial strength and high moisture content. The Wirtgen Cold Recycling Manual [10] also identifies the moisture-induced damage as a BSM distress mechanism connected to permanent deformation, due to a relatively high air void content compared to HMA and the partially coated nature of the aggregate. Moisture susceptibility is related to the loss of adhesion between the bitumen and aggregate particles and is understood as the damage caused by exposure of a BSM to high moisture contents and the pore-pressures induced by wheel loads.

Jenkins [83] explored the mode of failure of BSMs employing the principles of fracture mechanics. It was concluded that BSMs comprise visco-elasto-plastic behaviour and that their fatigue testing is achievable. However, considering the applicability of classical fatigue behaviour under repeated loading, it was presumed that BSMs do not fail in fatigue like HMA due to the discrete distribution of bituminous bonds in the material and therefore, no conditions for the potential crack to develop. Instead, broken bituminous bonds are supposed to result in particles re-orientating (micro-shearing), causing permanent deformation as in granular materials.

As stated in the CEDR CoRePaSoL reports [144] [145], the permanent deformation mode of failure characteristic for granular materials is more likely to occur in case of cold recycled mixes with lower binder content ($< 2.5\%$ of the mixture mass), mainly during the initial curing phase. In contrary, BSMs containing high residual bitumen content might display fatigue behaviour similar to HMA, mainly in an advanced curing stage. Within the internal structure of BSM, instead of complete aggregate coating, local bridges are formed, which can be destroyed due to repeated loading even if typical fatigue cracks do not appear on the surface. However, when BSM-Es are considered, better coating of aggregates with residual bitumen is achieved than in the case of BSM-FBs. Moreover, it was assumed that the aged binder included in RAP might play an active role in the development of bituminous bonding, and consequently mechanical properties of the mixture [99]. Higher binder content (either in form of added binder or in RAP) usually improves the coating and fatigue resistance of those materials, but often decreases the mix stiffness. As stated in [144], the current literature sources indicate that fatigue behaviour similar to HMA is observed for BSMs in some cases, but frequently the resistance against rutting is decisive for the proper long-term behaviour of pavements with cold recycled layers.

The fatigue failure of BSM-E mixtures containing 90% RAP, 4% bitumen emulsion and 1% cement was investigated in uniaxial cyclic tension fatigue tests in the latest research by Graziani [146]. It was observed that at the beginning of the fatigue test, the specimens showed a cement-dominated damage behaviour, followed by the breaking of the cementitious bonds, which led to the transition towards a bitumen-dominated damage behaviour. Eventually, further damage of the bituminous bonds led to the specimen collapse. Longer fatigue life was noted for BSMs with respect to CBTMs with a 2% cement addition. However, as stated by researchers, as long as the field behaviour is concerned, typically BSMs are not prone to fatigue as the permanent deformation accumulation is more severe.

3.3. Viscoelastic performance

3.3.1. Viscoelasticity assessment

The topic of viscoelasticity of BSMs is extensively studied. Many works emphasize the similarity of the properties of BSM mixtures to traditional asphalt mixtures, due to the leading role of bituminous bonds in the development of the rheological, mechanical and durability properties of these mixtures. It is presumed that the viscoelastic response of HMA and BSMs could be considered analogous as both materials are dependent on the rheology of bitumen. From the internal structure point of view, the filler and partially fine aggregate fraction and residual bitumen constitute the mastic which is expected to exhibit time- and temperature-dependent response during loading and unloading. However, it should be noted that since the bitumen in HMA creates a continuous film on the virgin aggregates, the viscoelastic response of HMA almost exclusively depends on the viscoelastic characteristics of the bitumen used. In the case of Bitumen Stabilised Materials, which are characterized by a specific internal structure with local bituminous bonds, the influence on the viscoelasticity should be anticipated in the presence of “fresh” residual binder from bitumen emulsion or foamed bitumen, the aged binder enclosed in mastic-aggregate RAP clusters and the possible interaction of these two components. A particular attention should be given to the fact that the content of “fresh” residual bitumen in BSMs can be approximately more than 2 times lower than the typical content of bitumen in HMA [17]. Moreover, RAP, which makes up the major part of the mineral mixture in BSMs and consists of both virgin aggregates previously utilized in the asphalt layer and aged bitumen mastic, is characterized by a significant material heterogeneity in terms of gradation, shape regularity and internal structure also due to a presence of additional air voids. These factors are reported to highly influence mechanical response and thermal sensitivity of BSMs [8] [99] [107]. Additionally, due to a limited influence of cement on the mechanical response of BSMs and their moisture-related stiffness changes, their viscoelasticity assessment is challenging.

Based on the extensive review of the BSMs’ mechanical properties testing, it can be stated that Bitumen Stabilised Materials, apart from the stress dependent behaviour, also exhibit a time- and temperature-dependent behaviour typical for viscoelastic materials. As stated by Valentin et al. [86], despite the complicated structure of BSMs, their behaviour can be described using testing methodologies and results’ assessment techniques applicable to asphalt mixtures. Following this assumption, in the European CoRePaSoL project a comprehensive study of 90%-RAP BSM-Es with no cement addition was carried out. The principle of time–

temperature superposition has been applied to describe the viscoelastic behaviour of BSM mixtures. The analysis of the master curves for loss and storage parts of the complex modulus E^* and phase angle δ created based on the four-point bending beam test results confirmed the applicability of linear viscoelastic theory to BSMs. It was observed that BSM mixtures are thermo-mechanically sensitive materials which exhibit almost purely elastic behaviour in the lowest test temperatures and the highest frequencies. Compared to analogous CBTM mixtures produced with 3% cement addition, BSMs displayed an increased thermal susceptibility. The temperature dependence, characteristic for viscoelastic materials, was also displayed for BSMS with bitumen emulsion in the indirect tensile strength (ITS) and IT-CY stiffness modulus testing carried out in the work of Valentin et al [145]. Higher temperature-sensitivity of BSM-Es in comparison to CBTM mixtures containing 3.0% of cement was also confirmed based on the ITSM test results in the work of Konieczna et al. [47]. As reported by Graziani [146], the results of complex modulus testing performed at 20°C and 10 Hz frequency showed that when compared to asphalt concrete, BSM-E mixtures were characterised by two times lower phase angle values.

Time- and temperature-dependent response of BSMs was confirmed in the work of Meneses [128], in which BSM-Es containing 85% RAP, 4% bitumen emulsion and cement or hydrated lime (0%, 1%) were subjected to dynamic modulus testing according to the AASHTO T342 [S5] standard. Compared to asphalt concrete (AC) mixtures, BSMs presented lower dynamic modulus for the entire frequency and temperature range tested. Phase angle results showed that at intermediate and high frequencies BSMs present more viscous behaviour with lower capacity to recover from deformations in regard to AC. At low-frequency/high-temperature conditions, BSMs showed more elastic behaviour, with lower phase angle values, which can be attributed to the mixtures' mineral skeleton differences between HMA and BSMs. However, the granular material-like response of BSMs was also confirmed in the triaxial resilient modulus tests, with the stiffness depending on the stress rate in the material. It was observed that BSMs containing hydrated lime showed less dependence on confining pressure level than BSMs containing cement.

The research carried out by Kuna and Gottumukkala [87] confirmed that BSMs exhibit less frequency- and temperature-dependency compared to HMA. In this work, the AASHTO TP62-07 [S6] dynamic modulus tests were conducted at different compressive stresses (50 kPa–300 kPa) with confinement pressure on 50%-RAP BSM-FB mixtures with 0% and 1% cement addition. The results showed that besides the increase of the dynamic modulus values with the increase in loading frequency and the decrease in temperature, indicating the

viscoelasticity of the material, the stress dependency of BSM-FBs was also observed, with the dynamic modulus increasing with an increase in the deviatoric stress value, especially at high test temperatures.

The results of the stress-controlled cycling tension and tension-compression tests performed by Nivedya et al. [20] on 80%-RAP BSM-FBs indicated that the mechanical response of the material regardless of the test temperature, air void content and cement content was viscoelastic in nature. In the lower testing temperature (15°C), BSMs exhibited behaviour of a viscoelastic solid material, while at 45°C, of a viscoelastic fluid one. The influence of the hydraulic binder's presence (0% or 1%) in the mixture was observed only at a higher test temperature, while the air void content influenced the properties of the material for all test variables. Moreover, the researchers observed that the application of the existing HMA dynamic modulus test protocols for BSM testing needs to be performed with caution due to the anomalies noticed in phase lag and strain evolution [138].

Valuable insights on the validity of the time-temperature superposition principle (TTSP) for Bitumen Stabilised Materials with bitumen emulsion are presented in the latest work of Kuchiishi et al. [147]. It was stated that BSMs exhibit complex mechanical behaviour but considering the results of triaxial resilient modulus tests and dynamic modulus tests, it was found that only the latter allowed to notice the existing differences in the mechanical response of BSMs with bitumen emulsion and foamed bitumen. Based on the assessment of master curves, as well as Black and Cole-Cole diagrams, it was observed that BSM-E mixtures were characterized by a wider modulus variation (steeper master curve slope) in the tested frequency and temperature range compared to BSM-FBs. The reason for this phenomenon is presumed the form of the residual bitumen distribution within the mixture (additional thin layer of bitumen covering the surface of the aggregate particles after emulsion breaking. Moreover, it was observed that BSM mixtures which contained only virgin aggregates in the mineral mixture and low residual bitumen content, also cannot be considered as purely elastic.

3.3.2. Review of testing methodologies

Complex modulus (E^*) is a property that defines the relationship between stress and strain for a linear viscoelastic material. The dynamic modulus ($|E^*|$), which is the normal value of the complex modulus, is a performance-related property that can be used for asphalt mixture evaluation and for characterizing its stiffness for mechanistic-empirical pavement design. This parameter is determined in dynamic testing, in which a specimen at a given test temperature is subjected to a controlled sinusoidal (haversine) compressive stress at various

loading frequencies. The applied stresses and resulting axial strains are measured as a function of time and used to calculate the dynamic modulus and phase angle. The complex modulus is defined as the ratio between the applied stress and the measured strain, and is represented by an imaginary number comprised by a real part (E' , storage modulus) and an imaginary part (E'' , loss modulus). The phase angle φ is the angle in degrees between a sinusoidally applied peak stress and the resulting peak strain.

The complex modulus (1) and dynamic modulus (2) are calculated using the following equations:

$$E^* = \frac{\sigma_0 e^{i\omega t}}{\varepsilon_0 e^{i(\omega t - \varphi)}} = \frac{\sigma_0}{\varepsilon_0} \cos\varphi + i \frac{\sigma_0}{\varepsilon_0} \sin\varphi = E' + iE'' \quad (1)$$

$$|E^*| = \frac{\sigma_0}{\varepsilon_0} = \sqrt{(E')^2 + (E'')^2} \quad (2)$$

where:

σ_0 - stress amplitude,

ε_0 - strain amplitude,

ω - angular frequency,

t – time,

φ - phase angle.

Based on the literature review it can be stated, that test methodologies based on compressive loading originally used for asphalt mixtures testing (AASHTO T342 [S5], AASHTO TP62-07 [S6], and AASHTO T378-22 [S7]) are also successfully applied to characterise the viscoelasticity of cold recycling mixtures [128] [148] [149]. Due to the fact that the material is susceptible to damage resulting from cutting and the slab samples pose difficulties in handling [86], the majority of complex modulus tests are performed on cylindrical specimens in stress-controlled mode. In order to synthesize and adapt the HMA testing methods, the test conditions (loading stress/target strain levels, temperatures and frequencies) are suitably modified to operate within the range of linear viscoelasticity (LVE) of tested cold recycling materials. As reported by Jaczewski et al. [148], the linear viscoelasticity limits for cold recycling mixtures depend on mixture composition (especially quantities of binding agents) and the test temperature. Following the observations presented by Meneses et al. [128], application of the highest standard temperatures used for asphalt mixtures testing (i.e., 54.4°C) might unpredictably influence the mechanical behaviour of cold recycling mixtures due to possibility of the RAP residual binder reactivation. Table 1 presents the summary of the selected applications of the HMA dynamic modulus testing methodologies for cold recycling mixtures'

viscoelasticity evaluation. Gathered data indicates a wide variety of testing conditions as well as considerable differentiation in specimen preparation and curing techniques applied.

Table 1 Applications of dynamic modulus testing procedures for cold recycling mixtures' viscoelasticity evaluation

Standard	Source	Test temperature	Test frequencies	Target strain amplitude	Specimen preparation	Specimen curing
AASHTO T342 [S5]	Meneses et al. [128]	4.4–37.8°C	25–0.1 Hz	50–150 $\mu\epsilon$	Cylindrical (d=100 mm, h=150 mm) gyratory compaction	7 days, 20°C (in sealed bags) + 7 days, 40°C (oven)
	Kuchiishi et al. [147]	4.4–54°C			Cylindrical (d=100 mm, h=150 mm) vibratory hammer compaction	28 days, 40°C (oven, for BSMs with cement) 7 days, 40°C (oven, for BSMs with hydrated lime)
AASHTO T378-22 [S7]	Jaczewski et al. [148]*	10°C, 20°C	25–0.1 Hz	65–110 $\mu\epsilon$	Cylindrical (d = 100 mm, h = 150 mm), gyratory compaction	> 1 year storage
AASHTO TP62-07 [S6]	Chomicz-Kowalska et al. [149]*	-10–37.4°C	25–0.1 Hz	50-100 $\mu\epsilon$	Cylindrical (d = 100 mm, h = 150 mm), gyratory compaction	24h, 20°C (in moulds) + 72h, 40°C (oven)
	Kuna et al. [87]	5–40°C	10–0.01 Hz	15-321 $\mu\epsilon$		24h, 40°C (oven, in moulds) + 72h, 40°C (oven, after extraction)
EN 12697-26 [S8]	Valentin et al. [86]	0–30°C	50–0.5 Hz	- (strain-controlled mode)	Beams (dimensions acc. to standard), slab compaction	2 days, 20°C + 4 days, 50°C + 14 days, 20°C.

* - CBTM testing

3.4. Cohesion and permanent deformation response

Cohesion is defined as an *intermolecular attractive force acting between two adjacent portions of a substance, particularly of a solid or liquid* [150]. When the geotechnical engineering aspect is taken into account, the term “cohesion” refers to the internal strength or bonding between soil particles that enables the soil to resist shear forces [151] [152]. As stated by Chaturabong and Bahia [153], at the macro level of asphalt mixtures, cohesive forces constitute the integrity of the material. In HMA, characterized by a continuous bitumen film surrounding the aggregate, cohesion at a micro level can be defined as deformation under load that occurs at a distance from the aggregate substrate and beyond the influence of mechanical interlock and molecular orientation [153]. Due to the complex mechanical response of BSMs, which are non-continuously bound composites stabilised with bitumen, the characterization of their cohesive properties and permanent deformation response might be realized using approaches suitable for granular as well as for bound materials.

Recoverable (elastic) or non-recoverable (plastic or permanent) deformations occur when pavement is subjected to cyclic vehicle loading during its service life. Non-recoverable deformations are responsible for the rutting phenomenon, causing road surface deterioration and affecting the driving safety and comfort. In general, the permanent deformation resistance of BSMs depends on various factors connected to the mix composition: RAP content and quality, aggregate gradation and fine particle content, residual bitumen content, presence of cementitious binders, moisture content, as well as specimen curing time and conditions [154] [155].

3.4.1. Unbound material approach

According to the granular material approach, Bitumen Stabilised Materials are conglomerates of coarse particles, fine particles, bitumen mastic and air voids. The material’s shear strength, tensile strength, and stiffness are determined by cohesive and frictional strength components. When the material works in tension, the BSMs’ behaviour is determined mainly by the cohesive element realised by the flexible mastic (residual bitumen and fines), but when it is subjected to compressive forces, the response is determined by both the cohesive and the frictional strength provided by inter-particle friction. The relative role that the frictional or cohesive component plays in determining the BSMs’ strength and stiffness depends almost entirely on the state of the mastic [17]. Due to the fact that this PhD dissertation covers the topic of the development and assessment of the cohesion of BSM mixtures in terms of their viscoelastic behaviour, this

section will discuss only basic and general information regarding permanent deformation response within the unbound material approach.

Cohesion (c) and permanent deformation (ε_p) response of BSMs evaluated as unbound materials are examined by performing respectively: monotonic and long-term cyclic tests in a triaxial setup [24] [137]. As stated by Cizkova et al. [22], triaxial testing is considered one of the most suitable approaches to classify BSMs with low amount of residual bitumen and hydraulic binder. As reported by Jenkins et al. [26], the non-linear Mohr-Coulomb plasticity model – widely used in soil mechanics – can be successfully applied for characterization of the BSMs' shear performance. The materials' shear strength (τ) and, as a result, resistance to permanent deformation, is therefore calculated based on the normal (principal) stress (σ), cohesion (c) and friction angle (φ_c) values as follows (3):

$$\tau = c + \sigma \tan \varphi_c \quad (3)$$

Following the granular material approach, BSMs present negligible capacity to transfer tensile stress, and cohesion (c) is therefore viewed as an “adhesiveness” of the material [22] [59].

Dynamic triaxial testing, on the other hand, allows for capturing the material's shear failure and perform modelling of the BSMs' permanent deformation response [24] [26]. Taking into account the stress-dependent nature of BSMs, the stress ratio-based parameters (such as Deviator Stress Ratio firstly proposed in 2000 by Jenkins [85]) are commonly introduced to determine the strain accumulation rate during Repeated Load Permanent Deformation (RLPD) tests carried out in a triaxial set-up [59] [137]. Nevertheless, based on the current state of research, it can be stated that a comprehensive BSM classification based on the RLPD triaxial test results is challenging due to a strong dependence of the chosen Deviator Stress Ratios on the individual mixtures' shear properties [26].

3.4.2. Bound material approach

BSMs' permanent deformation performance in terms of viscoelasticity remains briefly addressed. Single cases of the application of rutting, wheel tracking (WTT) and Flow Number (FN) dynamic creep tests for determination of the CRMs' and BSMs' permanent deformation response have been reported to date.

Yang et al. [125] presented the results of rutting tests performed on 70%-RAP BSM-E specimens to evaluate their stability at high temperatures (60°C). Based on the calculated performance indexes – rut depth (RD) and dynamic stability (DS) – it was concluded that the permanent deformation resistance is positively influenced by the formation of cement hydration

products with the most significant differences in BSMs' permanent deformation performance occurring at low hydraulic binder contents. The decrease in BSMs' rutting susceptibility with increase in the cement amount was confirmed in the work of Dong and Charmot [155], in which specimens of 100%-RAP BSM-E mixtures containing up to 0.75% cement were subjected to wheel tracking tests at 60°C.

Kim and Lee [27] investigated creep properties of 100% RAP Bitumen Stabilised Materials produced with low bitumen emulsion amounts (0.5%–1.5%) without cement addition. The effects of different RAP sources with the same residual bitumen content of 5.8%, different bitumen emulsion types and contents (0.5%–1.5% of mixture mass) on the obtained flow number values were studied. As reported by the researchers, the dynamic creep test parameters were determined by trial and error for BSMs to reach the tertiary point within the termination limit time of 10 000 seconds (or 5% level of cumulative permanent strain). The test temperature of 40°C was applied. Based on the test results, it was concluded that the RAP source influences the permanent deformation characteristics of tested BSMs, with lower flow number values obtained for RAP containing a more stiff residual binder. Moreover, the flow number of BSMs decreased with the increase in the bitumen emulsion content in the mixture. The recommendations were given to introduce the dynamic creep tests in the mix design process as a complementary assessment method to validate the optimum emulsion content in the case of the CRMs' field performance prediction.

The results of the research performed by Kim et al. [88] indicate the influence of the RAP source and residual bitumen content in BSM-FB mixtures on the Flow Number values. It was observed that BSMs containing fine RAP material with a stiff residual asphalt binder exhibited higher FN values compared to BSMs with RAP of a coarser grading with a soft residual binder.

In the latest work of Li et al. [77], the standard AASHTO T378-22 [S7] procedure was adopted to conduct dynamic creep tests on pavement core samples to determine the high-temperature stability of cold recycling mixtures at 55°C and 60°C and compare it with the performance of an SMA overlay mixture. It was observed that the volumetric properties of CRM core samples had a significant influence on the permanent deformation response of cold recycling mixtures.

Silva et. al [156] examined the permanent deformation response of 100% virgin aggregate and 89% RAP BSM-FB mixtures containing 1% hydrated lime by means of three Flow Number test methods: ABNT NBR 16505 standard procedure, Kim and Lee's method [27], and FN testing with individually determined loading stress levels using 3D-Move software

simulation of flexible pavement response. Based on the results of FN tests adapted for cold recycling mixtures, the researchers observed a major role of the loading stress conditions applied to the material in its permanent deformation response, as well as indicated the need for a further investigation of the FN test's critical temperature. The importance of the specimen's curing duration in the case of developing the BSMs' permanent deformation (PD) response was also highlighted.

3.4.3. Review of testing methodologies

The Flow Number (FN) is a property related to the permanent deformation resistance of the material determined in the dynamic creep testing. The Flow Number testing methodology was primarily developed for Hot and Warm Mix Asphalt under the NCHRP 9-19 and NCHRP 9-29 projects. Dynamic creep and recovery tests are performed using the Asphalt Mixture Performance Tester (AMPT) device in stress-controlled mode under unconfined conditions. Cylindrical specimens at a specific, constant temperature are subjected to a haversine cyclic uniaxial compression load pulses of 0.1 s every 1.0 s (Figure 8). The resulting permanent axial strains in the material are measured and presented as a function of the load cycles in the form of a cumulative permanent strain curve, which comprises of the primary, secondary, and tertiary stage (Figure 9). The primary stage is characterized by an initially rapid accumulation of the permanent deformation, connected to the densification of the material. Subsequently, the rate of the PD growth decreases and stabilizes, with the material reaching the second phase of a constant increase of permanent deformation. In this phase, the material's resistance to withstand loading diminishes which is connected to a gradual cohesion loss. The cycle number at which the tertiary phase commences is the Flow Number value. Thus, the FN is defined as the load repetition value at which the plastic flow of the material occurs, i.e. the shear deformation under constant volume starts. The higher the FN value, the greater is the material's PD resistance. Following this definition, the Flow Number can be also adopted as a dynamic creep test indicator for evaluating the ultimate cohesion of BSMs.

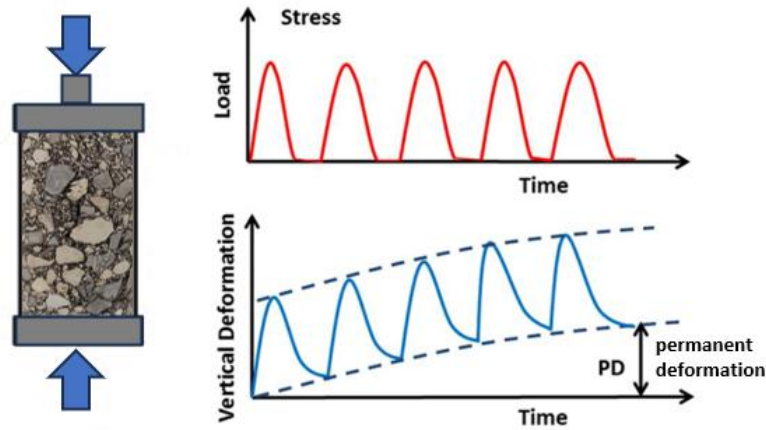


Figure 8 Dynamic creep (Flow Number) test setup, loading and deformation scheme, adapted from [157]

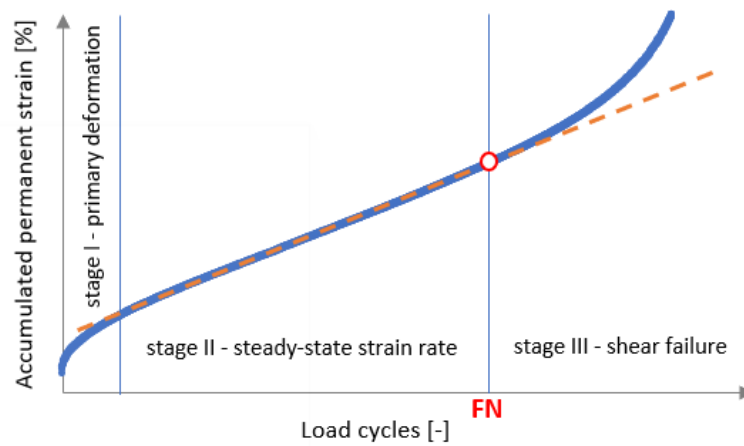


Figure 9 Typical relationship between permanent strain and number of cycles in dynamic creep test

Due to the differences in the mechanical response of Bitumen Stabilised Materials and HMA, the current research on the adaptation of the dynamic creep test methodologies for characterization of the BSMs' tertiary flow behaviour is based on the synthesis and adjustment of the testing conditions originally applied to asphalt mixtures. Table 2 summarises the selected applications of the dynamic creep testing methodologies for cold recycling mixtures' cohesion and permanent deformation response evaluation. Based on the literature review, it can be stated that the parameters specified for BSM testing vary significantly depending on the composition and volumetric properties of the tested mixtures. Therefore, selection of the dynamic creep test conditions in the case of BSMs requires an individual approach.

Table 2 Applications of dynamic creep testing procedures for cold recycling mixtures' cohesion and permanent deformation response evaluation

Standard	Source	Test temperature	Loading stress level	Test termination conditions	Specimen preparation	Specimen curing
AASHTO T378-22 [S7]	Kim and Lee [27]	40°C	70 kPa, 140 kPa no confinement	10 000 cycles or 5% accumulated permanent strain	Cylindrical (d=100 mm, h=150 mm) gyratory compaction	3 days, 40°C (oven)
	Kim et al. [88]	40°C	138 kPa no confinement		Cylindrical (d=100 mm, h=150 mm) Gyratory compaction	3 days, 40°C (oven)
	Li et al. [77]	55°C, 60°C	700 kPa, no confinement	Not specified	Cylindrical (d=100 mm, h=130 - 140 mm), core samples	In situ curing
NBR 16505 [S9]	Silva et al. [156]	60°C	204 kPa no confinement	10 000 cycles or 5% accumulated permanent strain	Cylindrical (d = 100 mm, h = 150 mm), gyratory compaction	0 or 28 days, 40°C (oven)
		50°C	400 – 550 kPa no confinement			
Not specified	NCAT [158]	54.5°C	70 psi (482.6 kPa)	Not specified, plastic strain at 20 000 cycles evaluated	Cylindrical (dimensions acc to AASHTO PP94 [S10]), gyratory compaction	3 days, 40°C (oven)
BSI DD 226:1996 [S11]	Gómez-Meijide et al. [159]	30°C	100 kPa no confinement	5000 cycles	Cylindrical (d = 101.6 mm, h = 50 mm), static compaction	3 days, 50°C (oven), 18 months storage at 20°C

3.5. Modelling of Bitumen Stabilised Materials' mechanical response

3.5.1. Permanent deformation

Among the most common approaches used for the determination of the paving materials' PD characteristics one can distinguish repeated load permanent deformation (RLPD) testing, in which accumulated permanent strain is recorded as a function of number of load cycles over the test period. Permanent deformation response of BSMs can be modelled based on the results of long-term dynamic triaxial tests (granular material aspect) as well as of rutting and dynamic creep tests, traditionally applied to asphalt mixtures (bound material aspect).

Since the first mechanical testing approach assumes the BSMs' stress-dependency and insensitivity to temperature resulting from a non-continuous dispersion of the residual bitumen in the mixture, the models applied mostly rely on the number of loading cycles and stress state present in the material, as well as shear parameters determined using the Mohr-Coulomb failure criterion. The test loading scheme and accumulated permanent strain curve obtained as a result are presented in Figure 10. As stated in the work of Orosa et al. [24], the permanent strain development prediction for unbound materials subjected to a large number of cyclic loads can be accurately performed using complex elastoplastic models considering both permanent and elastic deformations. However, due to their implementation difficulty, a wide application of the mechanical-empirical (ME) models has been reported for predicting the PD development in Bitumen Stabilised Materials [24] [59] [137].

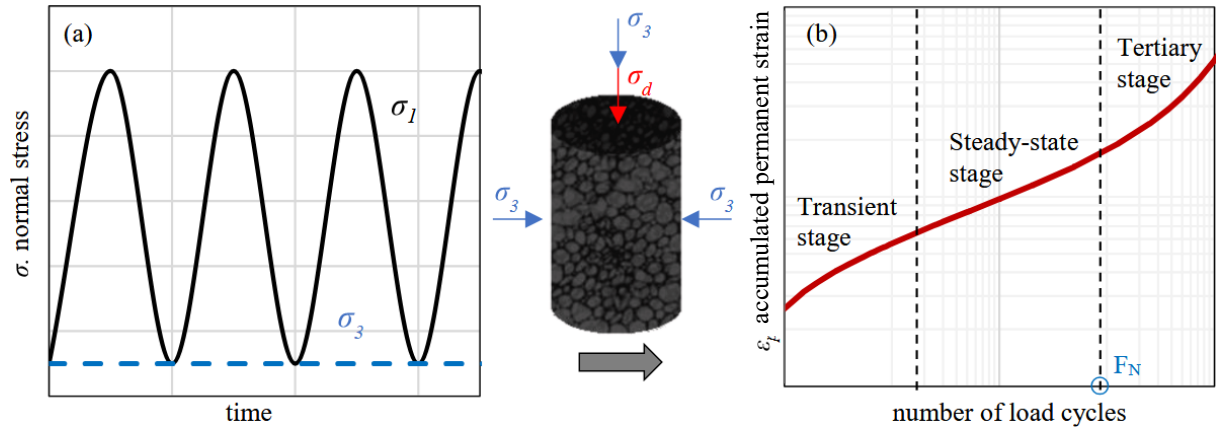


Figure 10 Stress applied during dynamic triaxial testing, and the results obtained: (a) sinusoidal variation of the vertical stress and CCP; (b) shape of the accumulated permanent strain curve, adapted from [24]

Among the most commonly adopted models, one can distinguish:

- Paute's model [160], which depends on the number of load cycles (N), permanent strain after the first 100 cycles ($\epsilon_p(100)$) and the stress state (A) in the material based on deviatoric (q) and mean (p) stress values, described by the following equations (4,5,6):

$$\epsilon_p = \epsilon_p(100) + f(N) \cdot A \quad (4)$$

$$f(N) = 1 - \left(\frac{N}{100}\right)^{-B} \quad (5)$$

$$A = \frac{\frac{q}{p + p^*}}{a - b \cdot \frac{q}{p + p^*}} \quad (6)$$

where:

a, b, B – regression parameters,

$p^* = m/s$; $q = mp + s$ – failure equations of the material.

- Huurman's model [161] (a variation of the Francken model) - an exponential model that depends on the number of load cycles (N), in which the regression parameters are a function of the stress level in terms of σ_1 and SR , described with the following equations (7,8,9,10):

$$\varepsilon_p = A \cdot \left(\frac{N}{1000} \right)^B + C \cdot \left(e^{D \frac{N}{1000}} - 1 \right) \quad (7)$$

$$A = a_1 \cdot (SR)^{a_2}, B = b_1 \cdot (SR)^{b_2}, C = c_1 \cdot (SR)^{c_2}, D = d_1 \cdot (SR)^{d_2} \quad (8)$$

$$SR = \frac{\sigma_1}{\sigma_{1,f}} \quad (9)$$

$$\sigma_{1,f} = \frac{(1 + \sin\varphi) \cdot \sigma_{3,f} + 2c \cdot \cos\varphi}{(1 - \sin\varphi)} \quad (10)$$

where:

ε_p – permanent strain,

N - number of loading cycles,

σ_1 - major principal stress,

$\sigma_{1,f}$ - major principal failure stress,

$\sigma_{1,f}$ - minor principal failure stress,

c - cohesion,

φ – internal friction angle,

$A, B, C, D, a_1, a_2, b_1, b_2, c_1, c_2, d_1, d_2$ – regression parameters and coefficients.

Based on the literature review, it can be stated that the models including the exponential term are considered to provide the best fit of the experimental data obtained for BSMs, as they allow for a reliable characterization of the rapid increase in the predicted permanent strain for high numbers of load cycles in the tertiary stage of PD development [24].

Presuming the viscoelastic nature of the material, modelling of the permanent deformation response of Bitumen Stabilised Materials should be performed using approaches recommended in specific standards for asphalt mixtures, which provide satisfactory experimental data fitting accuracy. As concluded by Biligiri et al. [162], the best-suited model for representing all three stages of permanent strain development in viscoelastic materials is the Francken model, developed on the basis of triaxial RLPD tests [163]. This mathematical

function relies solely on the number of load cycles (N) for the accumulated permanent strain curve representation and is described as follows (11):

$$\varepsilon_p(N) = AN^B + C(e^{DN} - 1) \quad (11)$$

where:

$\varepsilon_p(N)$ - permanent strain,

N - number of loading cycles,

A, B, C, D – regression coefficients.

The power part of the model describes the relationship between the cumulative permanent strain values and load number cycles in the primary and secondary stage, i.e., the phase of initial rapid strain development and a subsequent “linear”, steady-state strain gain rate. The exponential component, on the other hand, allows for capturing the onset of the tertiary stage and a reliable representation of the shear failure (flow) phase of the viscoelastic material.

The regression coefficients are determined by numerical optimization. Once fitted, the first (12) and second (13) derivatives of the Francken model are determined analytically.

$$\dot{\varepsilon}_p(N) = \frac{d\varepsilon_p(N)}{dN} \quad (12)$$

$$\ddot{\varepsilon}_p(N) = \frac{d^2\varepsilon_p(N)}{dN^2} \quad (13)$$

The Flow Number is defined as the cycle number corresponding to the minimum rate of the permanent axial strain change and representing the inflection point at which the permanent deformation of the asphalt mixture enters a rapid-developing phase. The FN value is therefore calculated as an N load cycle number, at which the second derivative of the Francken function changes from negative to positive [162].

Based on the literature review, it can be stated that in the available publications on the topic of dynamic creep testing of BSMs (section 3.4.3), the information on the type of models applied to describe the permanent strain accumulation curves and determine the FN values as well as on the experimental data fitting accuracy is scarce. Considering the wide variety of the BSMs' mechanical response scenarios, the evaluation of the validity of the Francken model variant applied for asphalt mixtures' characterization is recommended.

3.5.2. Viscoelasticity

According to the current research, the BSMs' viscoelastic response prediction is performed by master curve construction based on the functional testing results (i.e., dynamic modulus, phase angle). Due to a complex nature of the material, the literature sources indicate the application

of both linear (stress independent) and non-linear (stress dependent) viscoelastic domains for the BSMs' mechanical characterisation.

Presuming the HMA-like behaviour of BSMs, as long as their mechanical properties are defined under linear viscoelastic (LVE) conditions, the material should be treated analogously as thermorheologically simple, with its rheological behaviour described using the time–temperature superposition principle (TTSP) valid within the theory of linear viscoelasticity [164]. According to TTSP, the influence of temperature on the value of the dynamic modulus or phase angle of the bituminous mixtures, observed mainly in relation to their viscous properties with unchanged elastic properties, is equivalent to the influence of time [165].

Based on the literature review, it can be stated that the BSMs' master curve construction in the linear viscoelastic domain is commonly performed using the sigmoidal fitting function (14) to fit the logarithm of the dynamic modulus ($|E^*|$) test data. The temperature-dependent horizontal shift factor a_T can be defined e.g., by a second order polynomial equation (15) or the Arrhenius equation (15), while the reduced frequency is calculated according to the equation (17) [166], as specified below:

$$\log(|E^*|) = \delta + \frac{E^*_{max} - \delta}{1 + e^{\beta + \gamma \log(f_r)}} \quad (14)$$

$$\log(a_T) = \alpha_1 T^2 + \alpha_2 T^2 + \alpha_3 \quad (15)$$

$$\log(a_T) = \frac{\Delta E_a}{2.303R} \left(\frac{1}{T} - \frac{1}{T_r} \right) \quad (16)$$

$$\log(f_r) = \log(f) + \log(a_T) \quad (17)$$

where:

f_r – reduced frequency,

E^*_{max} – maximum dynamic modulus limiting the master curve – upper asymptote,

δ, β, γ – model fitting coefficients,

$\alpha_1, \alpha_2, \alpha_3$ – shift factor polynomial equation coefficients.

T – temperature,

ΔE_a – activation energy (fitting parameter),

R – gas constant equal to $8.314 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$,

T – the existing temperature (K),

T_r – the reference temperature (K).

The representative examples of the application of such approach are presented in the works of Kuchiishi et al. [147], Meneses et al. [128] and Gomez-Mejide et al. [119], where dynamic modulus tests were performed according to the AASHTO T342-22 [S5]

and AASHTO TP62-07 [S6] standards. The linear viscoelasticity modelling was also applied in the work of Mathaniya et al. [167], where BSM-E specimens were subjected to four-point beam strain-controlled dynamic modulus testing. In this case, the Arrhenius equation has been applied for calculation of the horizontal shift factor a_T to construct master curves.

The modelling approach realised in the non-linear viscoelastic domain was utilized by Kuna and Gottumukkala [87]. In this work, the stress dependent response of BSMs was evaluated by performing dynamic modulus tests not only in various temperature and frequencies, but also at stress increments ranging from 50 kPa to 300 kPa. The dynamic modulus ($|E^*|$) master curves were then constructed by shifting the test data using both horizontal a_T and vertical b_T shift factors, describing the magnitudes of shift of measured frequency and stress, respectively. An originally developed model based on the sigmoidal function was proposed for the determination of the stress-dependent $|E^*|$ master curves, presenting log-log plots of the reduced dynamic modulus – reduced frequency values, with the shift factors calculations carried out using nonlinear regression and least square optimization.

In the discussed research cases, the applied mathematical approaches yielded satisfactory results in terms of master curve fit, indicating that the Bitumen Stabilised Materials' mechanical response under dynamic loading conditions can be predicted over a wide range of temperatures and frequencies using methodologies originally applied to viscoelastic asphalt mixtures.

However, it should be noted that the application of mathematical functions to model master curves does not give information on constitutive relations in the material. Nivedya et al. [20] [138] proposed a non-linear viscoelastic constitutive modelling approach by developing viscoelastic solid and viscoelastic fluid models based on the multiple natural configurations and thermodynamics. A satisfactory data fitting accuracy was obtained for the tension and tension-compression test results. Nevertheless, the researchers emphasize that the validity of the developed models is restricted to a small temperature regime and the assumptions made regarding the isotropy and incompressibility of BSMs might be relaxed based on the availability for more experimental data.

To the knowledge of the author, the use of simple rheological models to fit the dynamic modulus test results and identify the thermo-rheological behaviour of Bitumen Stabilised Materials has not been analysed to date. However, examples of a successful application of the Huet-Sayegh and 2S2P1D models for mechanical response simulation has been reported for Cement-Bitumen Treated Materials (CBTMs) [168] [169].

3.6. Literature review summary

The aim of the literature review was to provide a general view on the cold recycling technology, with a major focus put on the topic of Bitumen Stabilised Materials with bitumen emulsion. The aspects of the general material characterization, the role of subsequent mixture components and BSMs' application challenges in road construction were analysed. Moreover, the state-of-the-art research on the BSMs' performance considering the evolutive behaviour, failure mechanisms, cohesion and permanent deformation response, as well as viscoelasticity assessment was presented.

Based on the literature overview, the following main conclusions can be drawn:

- 1) In many aspects, cold recycling mixtures (CRMs) are an ecologically and economically advantageous alternative to HMA mixtures used for the flexible pavements' base course construction, in line with the trends of sustainable development in the road industry. The interest in their application in construction is indicated by documented cases of successful asphalt pavement rehabilitation with the use of CRMs, including Bitumen Stabilised Materials.
- 2) Bitumen Stabilised Materials with bitumen emulsion are multi-phase composites which mechanical response is highly dependent on the mixture composition. A profound understanding of the role of specific components and their potential interactions is essential for achieving the desired performance characteristics.
- 3) Due to the widely described moisture-induced changes occurring in the material, to obtain a comprehensive view of the BSMs' performance, it is necessary to perform testing in a function of time, with the implementation of specific laboratory curing and sample conditioning procedures.
- 4) Adapting the testing methodologies originally applied to bound asphalt mixtures for BSM characterization in terms of cohesion, permanent deformation response, and viscoelasticity is possible. However, such an application requires an individual approach, considering the differences between the internal structure of HMA and BSMs and the tested material's specific mechanical and volumetric properties.
- 5) One of the main factors limiting the pace of popularization of the BSM technology is the fact that the scientific research conducted so far does not provide a clear answer to questions regarding the mechanical behaviour of these mixtures (varying from enhanced granular to quasi-bound nature) and its changes over time.

A consensus has yet to be reached on their failure mechanisms. Yet, further investigation into these aspects is vital for proper material characterization and selecting the appropriate performance testing methods for further use in road pavement design.

II. EXPERIMENTAL PROGRAM

4. Research program

4.1. Research plan

The experimental part of the dissertation includes the selection and characterisation of the input materials, description of the adopted mix design and testing methodologies, as well as presentation and analysis of the test results with conclusions and recommendations considering further research.

A chart outlining the general components of the research program adopted in the dissertation is presented in Figure 11.

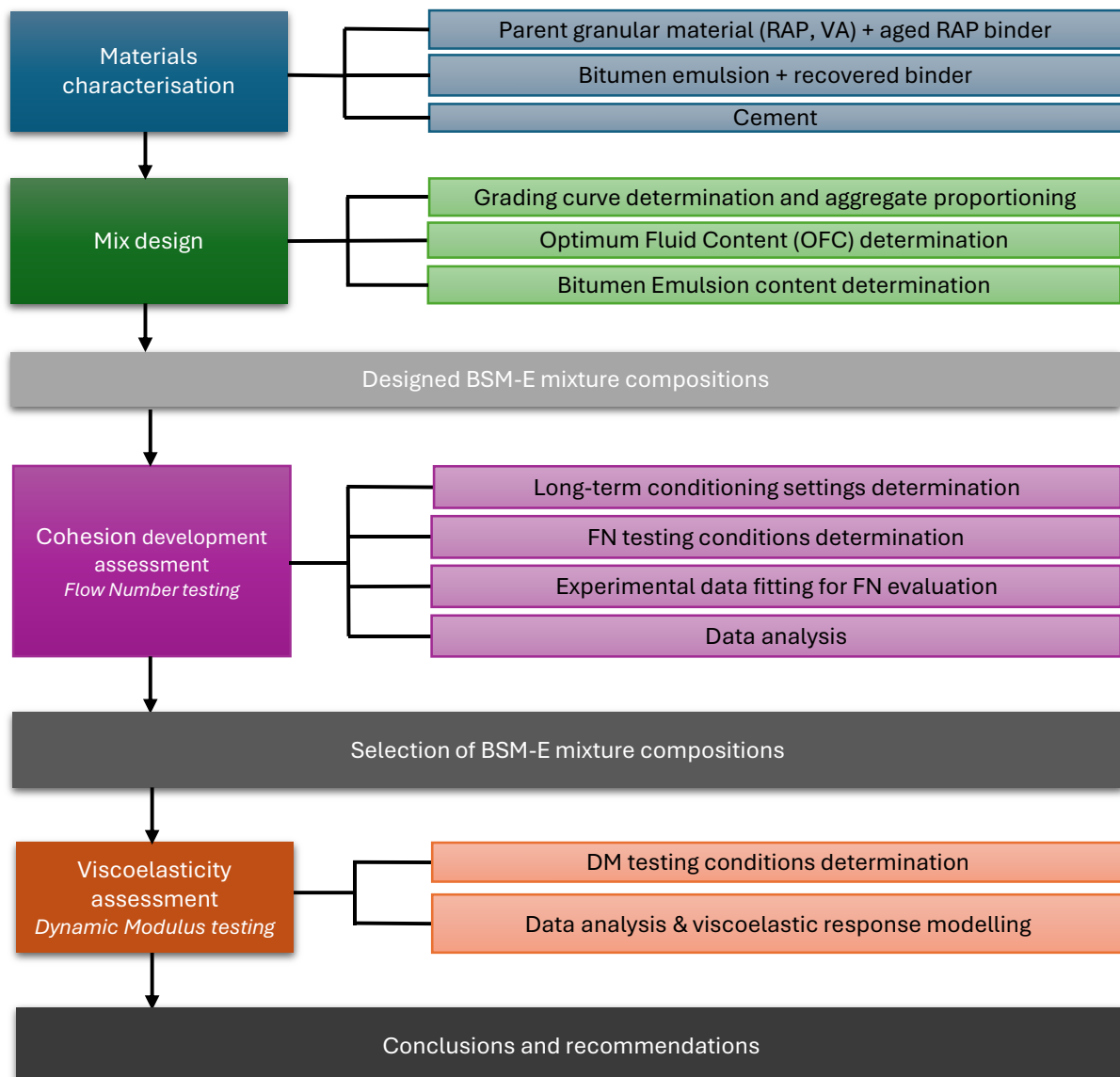


Figure 11 General research program

4.2. Material characterisation and mix design

The designed BSM-E mixtures were prepared using the following materials: RAP, virgin dolomite and limestone aggregates, slow-setting cationic bitumen emulsion, cement, and water. The BSM-E design approach used in this work was based on a modification of the procedure presented in the Wirtgen Cold Recycling Manual [10]. The basic assumption adopted in the BSM-Es' design considering their material composition was the differentiation in terms of the RAP and bitumen emulsion contents while maintaining a constant, high content of fine aggregate fractions (<0.063 mm). The amount of RAP ranged from 0% to 90% of the mineral mixture mass, while the bitumen emulsion content from 3% to 8% was applied in the mix design stage. The constant cement content of 1% was added.

In order to relate the cohesive and viscoelastic properties of BSM-E mixtures to those of HMA, specimens of the asphalt concrete mixture typically used for base courses, designated as AC 22, were prepared to serve as a reference. The produced asphalt mixture was characterized by a 4.0% bitumen content (35/50 penetration grade), maximum aggregate size of 22 mm and an average air void content of 4.5%.

4.2.1. Reclaimed Asphalt Pavement and virgin aggregates

Reclaimed Asphalt Pavement was derived from the milling of wearing and binder courses of the S7 expressway located in Poland. The material was characterized by the density of 2.599 g/cm^3 measured in water, following the PN-EN 1097-6 [S12] standard, as well as the average binder content of 4.8% by mass (according to PN-EN 12697-1 [S13]). The properties of the RAP binder are presented in Table 3.

Table 3 RAP binder properties

Property	Standard	Unit	Value
Penetration at 25°C	PN-EN 1426 [S14]	0.1 mm	16
Softening point	PN-EN 1427 [S15]	°C	68.6
Elastic recovery	PN-EN 13398 [S16]	%	35 (stretching length = 89 mm)

The grading of the RAP material was characterized following the procedures described in PN-EN 12697-2 [S17] and PN-EN 933-1 [S18] standards. The RAP grading curves for the material recovered after binder extraction in accordance with PN-EN 12697-1 [S19] (white curve) and of the unprocessed material (black curve) are presented in Figure 12.

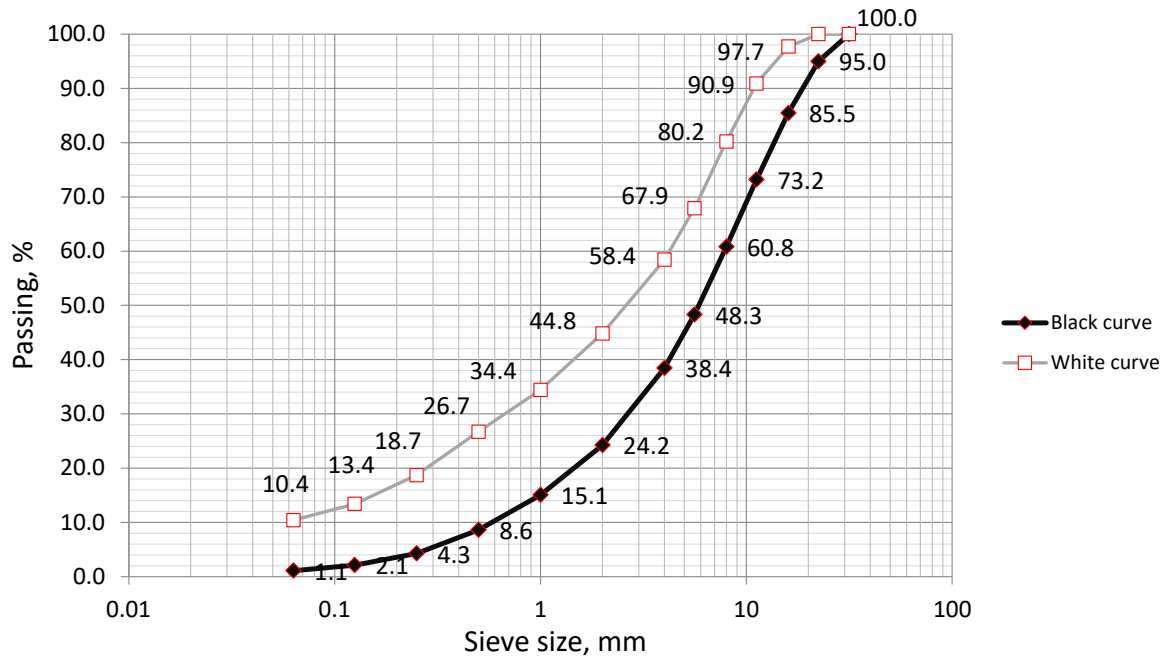


Figure 12 RAP white and black grading curves

The cohesion test on the RAP material was performed to evaluate the presence of potentially active bitumen in the recycled material. Testing was performed following the protocol presented in the recommendations introduced by the RILEM Technical Committee 237-SIB [170]. 100% RAP samples with a maximum aggregate size of 22.4 mm without additional bitumen were compacted using the Marshall compactor according to PN-EN 12697-30 [S20] with 50 blows per side to be then subjected to Indirect Tensile Strength (ITS) tests following the PN-EN 12697-23 [S21] standard. Six replicates were produced. The compaction temperature of 20°C was chosen due to its being applicable to the BSM-E technology performed at ambient temperatures. Based on the results of the cohesion test, it was concluded that the RAP material used in the study could not be considered to have a cohesive contribution from the aged bitumen or mastic, as all the produced specimens were unable to support self-weight, falling apart during the demoulding process and making it impossible to perform the ITS testing (Figure 13). However, it should be noted that, according to the standard [S21], the procedure requires testing of the specimens only 24 hours after compaction.



Figure 13 Part of the 100% RAP Marshall sample compacted at 20°C after demoulding

Considering the cohesive characteristics of the RAP material used and the commonly applied mix design approaches for BSM-E mixtures, a black RAP grading curve was adopted for design purposes, taking into account the fine particle content of 1.1%.

The virgin aggregates (continuously graded dolomite aggregate 0/31.5 mm, continuously graded limestone aggregate 0/4 mm, and limestone filler) were used to design BSM-E grading curves to meet the grading criteria presented in the Wirtgen Cold Recycling Manual [10]. The continuously graded crushed dolomite aggregate (M'80 0/31,5 mm Jaroszowiec CEMEX Polska) was used in the research. The continuously graded limestone aggregate 0/4 mm was produced by the Nordkalk Miedzianka company. The limestone filler used, delivered by WKG company, was produced in the grinding and fractionation processes of limestone.

The selected characteristics of virgin aggregates according to the PN-EN 13242 [S22] standard are presented in Table 4. The grading of virgin aggregates is presented in Table 5.

Table 4 Selected properties of virgin aggregates (PN-EN 13242)

Property	Virgin aggregate		
	Continuously graded dolomite aggregate 0/31.5	Continuously graded limestone aggregate 0/4	Limestone filler
	Value		
Grading	G _{A85}	G _{A90}	-
Tolerance category	GT _{A20}	GT _{C20}	-
Density [g/cm ³]	2.79 ± 0.10	2.71 ± 0.10	2.73

Table 5 Grading of virgin aggregates

Sieve size # [mm]	Dolomite aggregate 0/31.5 mm	Limestone aggregate 0/4 mm	Limestone filler
	[% retained on sieve]		
45	0.0	0.0	0.0
31.5	4.5	0.0	0.0
22.4	17.5	0.0	0.0
16	13.3	0.0	0.0
11.2	12.3	0.0	0.0
8	9.6	0.0	0.0
5.6	9.0	0.6	0.0
4	5.8	2.9	0.0
2	7.4	25.6	0.0
1	4.3	24.9	0.0
0.5	2.9	14.9	0.0
0.25	2.2	9.0	0.0
0.125	1.9	5.8	0.3
0.063	1.0	4.4	9.7
<0.063	8.3	11.9	90.0

The following RAP contents in the mineral mixture were used to design the grading curves of BSM-E mixtures: 0%, 30%, 50%, 70%, 90%. The proportions of virgin aggregates have been selected so that the grading criteria presented in the BSM-E mix design recommendations [10] were met. The mixing proportions of RAP and virgin aggregates were set to obtain the same amount of fines ranging between 9.8% and 10% of the mineral mixture mass, which is the recommended maximum value for BSM-Es [10]. The PN-EN 933-1 [S18] standard sieve set was utilized for the mineral material granulometric analysis, with the fines fraction considered particles passing through the 0.063 mm sieve. In Table 6, the mixing proportions of RAP and virgin aggregates (VA) in the mineral mixture are presented. The following abbreviations were adopted: VA: 0/31.5 - continuously graded dolomite aggregate 0/31.5 mm, VA: 0/4 - continuously graded limestone aggregate 0/4 mm, VA: filler - limestone filler.

Table 6 Mixing proportions of RAP and virgin aggregates (VA) in the BSM-Es mineral mixtures

Mineral mixture designation	RAP content [%]	VA: 0/31.5	VA: 0/4	VA: filler
		% content		
0% RAP	0	90	8.5	1.5
30% RAP	30	60	5.5	4.5
50% RAP	50	40	4.0	6.0
70% RAP	70	20	2.0	8.0
90% RAP	90	0	0	10

Grading curves of the designed BSM-E mixtures with the required grading envelope in accordance with the Wirtgen Cold Recycling Manual [10] are shown in Figure 14.

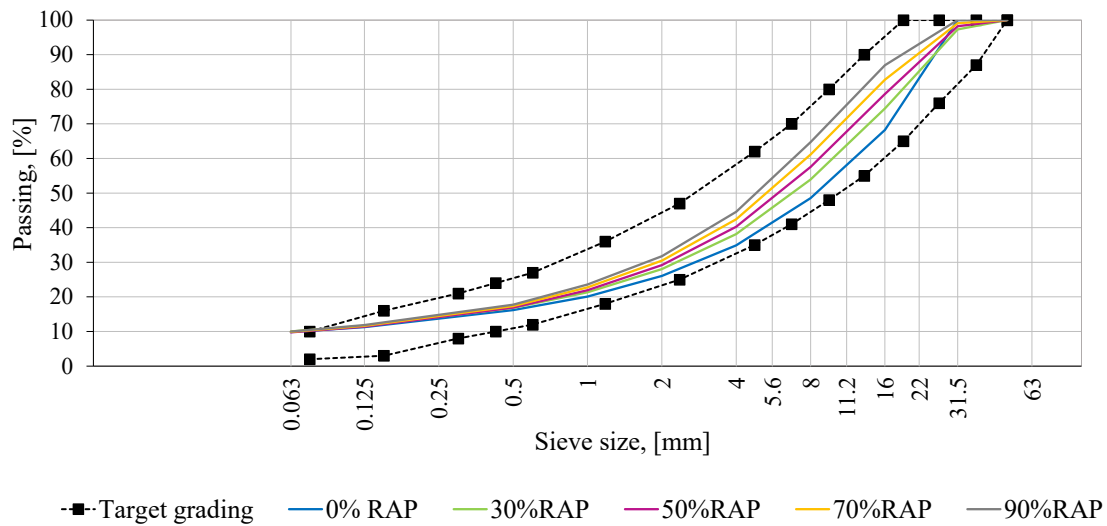


Figure 14 Designed BSM-E grading curves and target grading curves [10].

The fine particle (<0.063 mm) structure in the BSM-E mineral mixtures is presented in Figure 15. Depending on the content of the recycled material, the share of particles derived from RAP in the fine particles ranged from 0% (for a 0% RAP mixture) to 10% (for a 90% RAP mixture).

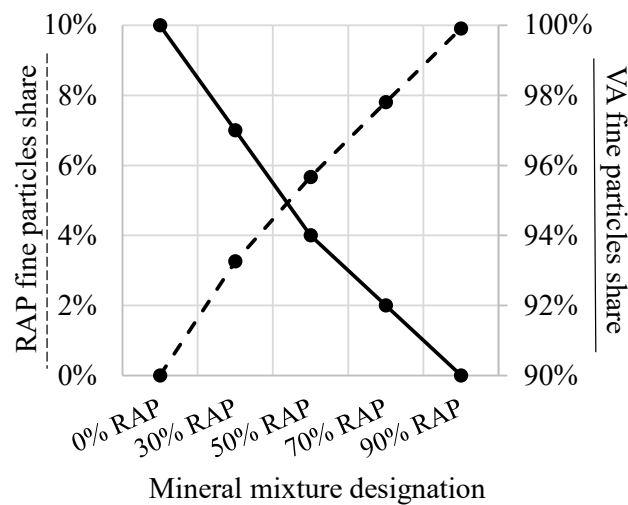


Figure 15 Fine particle structure in BSM-E mineral mixtures depending on the RAP content

4.2.2. Water content

Determination of the Optimum Fluid Content (OFC) and Maximum Dry Density (MDD) relationship for the mineral material was performed following the modified Proctor moisture-density relationship test procedure (PN-EN 13286-2 [S4]). Test samples for each RAP-virgin aggregate mineral mixture composition (as presented in Table 6) with a 1% cement addition were prepared. Four different water contents were applied to the mineral material. Samples were compacted in a B-type mold, using a 4.5 kg Proctor hammer in 5 layers, applying

56 blows per layer. Maximum Dry Density (MDD) and Optimum Fluid Content (OFC) were determined based on the established relationships between the calculated samples' dry densities for the subsequent moisture contents plotted on graphs. MDD corresponds to the moisture content at which the dry density is the highest. Thus, OFC corresponds to the moisture content at which the MDD occurs. The detailed results of the MDD and OFC determination for individual RAP contents in the mineral mixture are presented in Appendix 1. The summary of the determined OFC values is shown in Table 7.

Table 7 OFC values for BSM-E mineral mixtures

Mineral mixture designation	OFC [%]
0% RAP	6.4
30% RAP	6.1
50% RAP	6.0
70% RAP	5.8
90% RAP	5.9

4.2.3. Cement

Slag cement CEM III/A 42.5N – LH/HSR/NA, as per PN-EN 197-1 [S23] standard, was added as an active filler in a constant amount of 1% of the mineral mixture mass. The material characteristics are presented in Table 8. Such type of cement, containing 36–65% (m/m) of ground-granulated blast furnace slag, was used to minimise the impact of the cementitious binder's hydration on the development of the BSM-E mixture tensile strength due to a reduced amount of Portland clinker in the binder composition. Moreover, slag cement is an ecologically beneficial alternative to OPC as it is characterized by an embodied CO₂ value of 398–622 kg CO_{2e}/t, which stands for up to approximately 56% decrease in the CO₂ emissions compared to Ordinary Portland Cement (OPC) [171]. The binder applied was typically characterized for slag cement by a more developed specific surface area and higher water demand than the average values for OPC, which may influence the required moisture content in the BSM-E mixtures. This issue was addressed in the BSM-E design process by introducing the active filler into the RAP-aggregate mixtures during the Optimum Fluid Content determination process.

Table 8 Selected properties of slag cement CEM III/A 42.5N – LH/HSR/NA

Property	Standard	Unit	Value
Early compressive strength (2-day)	PN-EN 196-1 [S24]	MPa	15.9
Compressive strength (28-day)		MPa	55.2
Specific surface area (SSA)	PN-EN 196-6 [S25]	cm ² /g	4703
Setting time	PN-EN 196-3 [S26]	min	231
Water demand	PN-EN 196-3 [S26]	%	33.9
Density	PN-EN 196-6 [S25]	g/cm ³	2.97

4.2.4. Bitumen emulsion

The industrially produced slow-setting cationic bitumen emulsion type C60 B10 ZM/R for tack coats and cold recycling purposes (according to PN-EN 13808 [S26]), containing the 70/100 penetration grade base bitumen was used. The properties of the bitumen emulsion according to the standard requirements [S26] are presented in Table 9, while the basic properties of the bitumen binder recovered from the emulsion are shown in Table 10.

Table 9 Properties of bitumen emulsion type C60 B10 ZM/R

Property	Standard	Unit	Value
Residual binder content	PN-EN 1428 [S27]	%	58.8
Mixing stability with cement	PN-EN 12848 [S28]	g	0.0
Residue on sieving – 0.5 mm sieve	PN-EN 1429 [S29]	%	0.01
Storage stability by sieving (7 days storage) – 0.5 mm sieve	PN-EN 1429 [S29]	%	0.0
Viscosity - Efflux time 2 mm at 40°C	PN-EN 12846-1 [S30]	s	28
Adhesion with reference aggregate (basalt aggregate)	PN-EN 13614 [S31]	% coverage of aggregate surface	90

Table 10 Properties of the binder recovered from bitumen emulsion type C60 B10 ZM/R

Recovered binder property	Standard	Unit	Value
Penetration at 25°C	PN-EN 1426 [S14]	0.1 mm	54
Softening point	PN-EN 1427 [S15]	°C	48.7

Considering the presented characteristics of bitumen emulsion, its applicability in the BSM mixtures' design process was confirmed according to the Wirtgen Cold Recycling Technology guideline [10].

Determination of the bitumen emulsion content in the BSM-E mixtures was carried out following a modified design approach based on the Wirtgen Cold Recycling Manual procedure [10]. The bitumen emulsion contents applied in the mix design ranged from 3% to 8% of the mass of the mineral mixture and cement (with an increment of 1%). The use of high BE contents was intended to verify the development of the ITS strength of BSM-E mixtures with a systematic increase in the bitumen content until the vast majority of the moisture was a result of an emulsion addition. Based on the adopted grading curves and the determined Optimum Fluid Contents, thirty mix compositions of BSM-Es were prepared. A diagram illustrating the principle of selecting material variables in the BSM-E mix design process at the optimum bitumen emulsion content determination stage is presented in Figure 16.

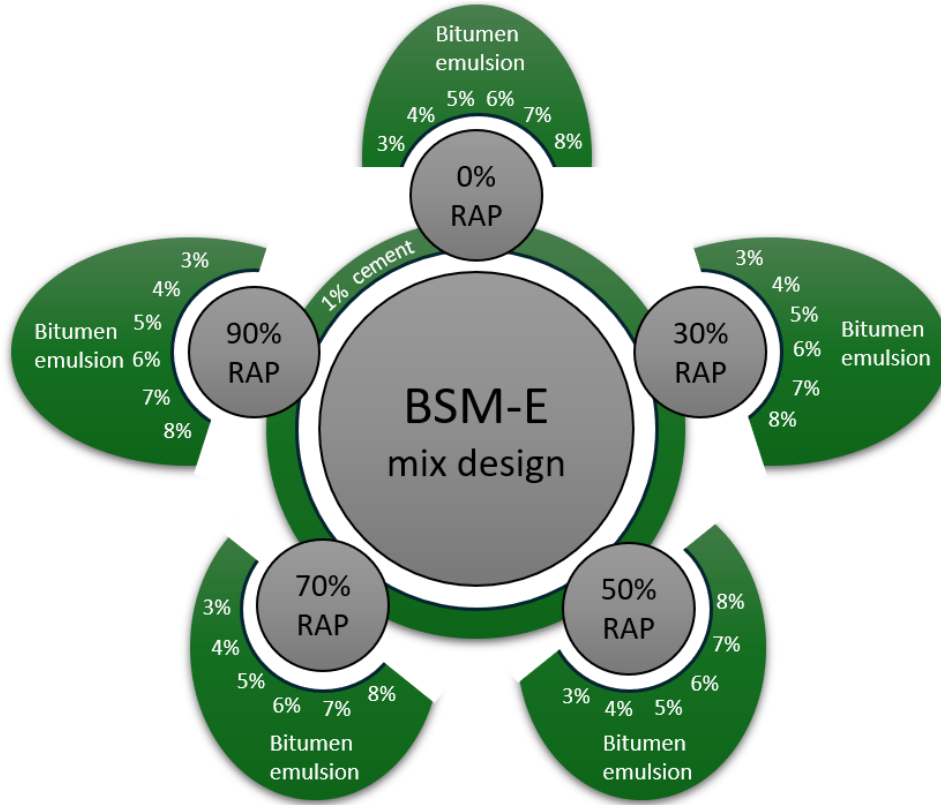


Figure 16 Material variables' selection in the BSM-E mix design process

For design purposes, it was assumed that the OFC of the BSM-E mixtures consists of water from the bitumen emulsion, moisture content of the granular materials, and water added separately to the mixture. Since the bitumen contained in the emulsion is considered to have an influence on the workability of the fresh mix [10] [44], this factor was also included in the determination of the amount of water to be added to reach the OFC for each RAP/virgin aggregate composition.

The amount of additional water was determined using a modified formula from the Wirtgen Cold Recycling Manual [10], as given in equation (18):

$$w_a = OFC - n_{mc} - BE_w - 0.5 \cdot BE_b \quad (18)$$

where:

w_a – amount of water to be added to the mixture [%],

OFC – Optimum Fluid Content [%],

n_{mc} – natural moisture content of RAP and aggregates [%],

BE_w – amount of water present in bitumen emulsion [%],

BE_b – amount of bitumen present in bitumen emulsion [%].

Indirect Tensile Strength testing was performed according to the procedure described in section 4.3.3.1. The ITS_{dry} and ITS_{wet} test results for each BSM-E mixture type were plotted against the relevant bitumen emulsion content. The one-variable regression models were then

fitted to the experimental data to determine the bitumen emulsion content corresponding to the maximum ITS values. The validity of the applied regression models describing the relationship between ITS_{dry} or ITS_{wet} values (dependent variables) and the BE content (independent variable) was evaluated based on the coefficient of determination R^2 and p -values obtained in the lack-of-fit test.

4.3. Testing methodology and data analysis

4.3.1. Sample preparation and curing

BSM-E mixtures were prepared in laboratory conditions at ambient temperature (25 ± 2)°C. For the Indirect Tensile Strength testing according to the PN-EN 12697-23 [S21] standard, cylindrical specimens with a height of (63.5 ± 1.5) mm and diameter of 101.6 mm were compacted using the Marshall hammer by applying 75 blows per side. Perforated moulds with 24 holes 2 mm in diameter were used to facilitate water drainage during the compaction process. After compaction, samples were left in moulds for 24 hours to allow sufficient strength to develop and therefore avoid any damage during the extrusion process. Then, an accelerated curing procedure was applied – demoulded BSM-E samples were put in a forced-draft oven at 40°C and dried to constant mass for 72 hours. After curing, samples were left to cool down for 24 hours at room temperature before testing.

For the Flow Number and Dynamic Modulus testing according to the AASHTO T 378-22 [S7] standard, cylindrical specimens with a height of 170 mm and diameter of 150 mm were produced in the gyratory compactor according to the PN-EN 12697-31 [S32] standard, imposing compaction force of 600 kPa, 1.25° gyration angle and 30 gyrations (Figure 17 a). Prepared samples were demoulded after compaction and dried in an oven at 40°C until reaching constant mass. Subsequently, coring and cutting the samples to testing dimensions were performed to obtain cylindrical specimens with 150 mm in height and 100 mm in diameter (Figure 17 b). Specimens were then dried to reach constant mass at room temperature before applying the long-term conditioning procedure.

The number of samples prepared for each test is specified in section 4.3.3 for each method. The mixture samples' volumetric properties: maximum density ρ_{mv} (PN-EN 12697-5 [S33], pycnometer method) and bulk density $\rho_{b,dim}$ (PN-EN 12697-6 [S34], method D) were determined to calculate the air void content V_m (PN-EN 12697-8 [S35]).

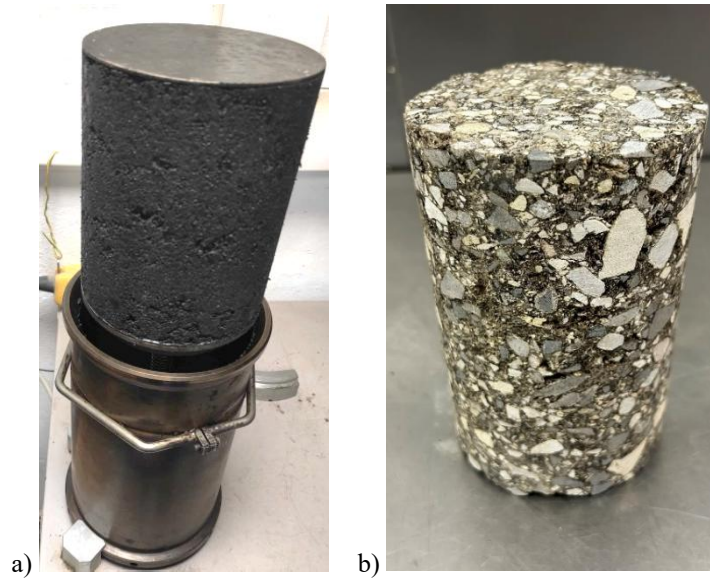


Figure 17 BSM-E specimen for testing in AMPT device: a) demoulded after gyratory compaction, b) air-dried after coring and cutting to testing dimensions.

4.3.2. Long-term sample conditioning

Specimens for the Flow Number and Dynamic Modulus testing were subjected to long-term conditioning to observe potential changes in BSM-E mixtures' cohesion over time. Before performing FN tests, samples were stored at room temperature of $(20 \pm 2)^{\circ}\text{C}$, with side confinement and top pressure applied to partially simulate pavement conditions. The side confinement was realized by wrapping the specimens with PVC pipes of a nominal diameter of 110 mm and a wall thickness of 2.2 mm. They were cut in half to adjust their diameter and enable the fitting of the produced cylindrical specimens inside. Wrapped samples were then subjected to compression by installing three steel worm drive clips placed at an equal distance from each other. The introduced side confinement was intended to simulate the lateral pressure existing in the pavement structure and to prevent the specimen's side surface from deteriorating during the conditioning process. To maximize the similarity of the side compression conditions for each sample, the same width of the pipe cutout was maintained, with the clamps screwed mechanically so that the pipe edges met each other. The top pressure, simulating the pressure created by the pavement structural layers laying above the BSM-E base course, was realized by putting a PVC container with a cardboard bottom, filled with fine basalt aggregate 0/2 mm of 1.55 g/cm^3 average bulk density in the loose state on the side-compressed sample. The BSM-E samples' conditioning confinement setup is presented in Figure 18.

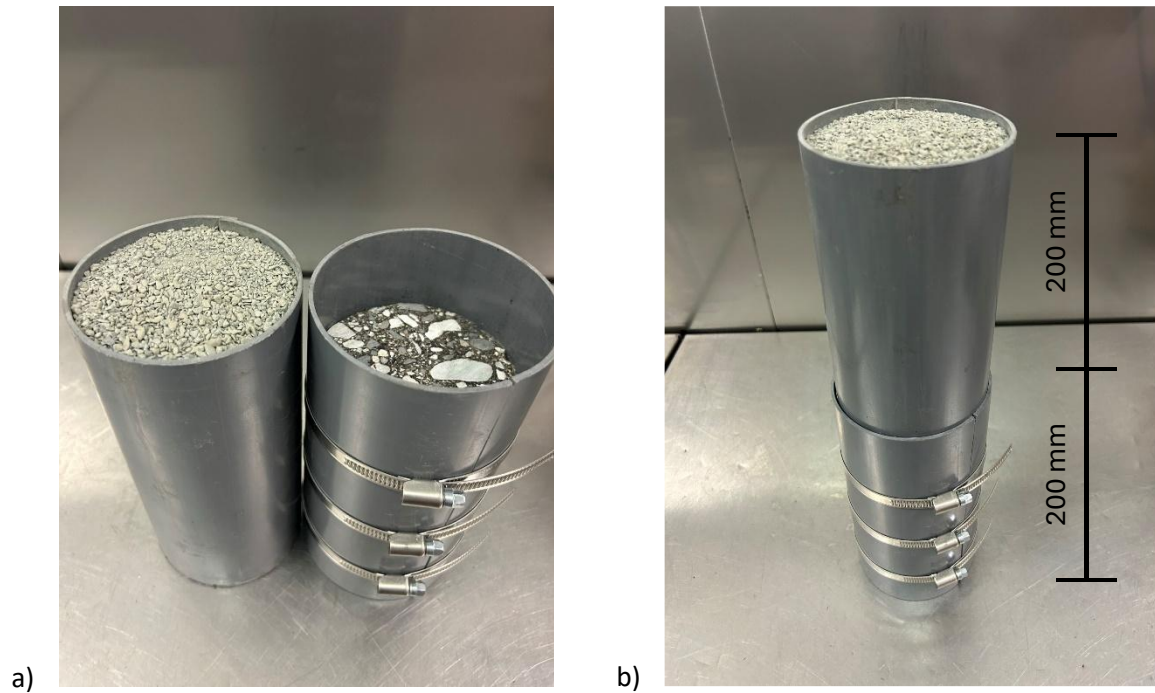


Figure 18 Long-term sample conditioning system: a) top loading container filled with basalt aggregate and BSM-E specimen with side confinement, b) BSM-E sample conditioning setting.

The total mass of the top loading container equal to 2.36 kg was calculated based on the data regarding the thickness of the surface, binder, and base courses required for the flexible pavement construction with cold recycling layers in Poland, according to the Polish Catalogue of Typical Flexible and Semi-rigid Pavement Structures [45].

The long-term conditioning procedure was applied to samples for a duration of 0, 7, 14, 28, 90, and 120 days to monitor any potential changes in the cohesion of BSM-E samples subjected to side confinement and top pressure in time, measured using Flow Number dynamic creep testing.

4.3.3. Mechanical testing

4.3.3.1. Indirect Tensile Strength

The Indirect Tensile Strength testing was performed according to the PN-EN 12697-23 [S21] standard as a part of the BSM-E mix design process presented in the Wirtgen Cold Recycling Technology guideline [10]. For the purpose of the bitumen emulsion content determination, Marshall specimens were subjected to ITS tests at 25°C. Ten Marshall specimens were prepared for each BSM-E mixture according to the preparation protocol described in section 4.3.1. Half of the specimens were conditioned for 4 hours at the testing temperature under air conditions to determine ITS_{dry} values, and the remaining half was tested after an additional

24-hour soaking in water (ITS_{wet}). Tensile Strength Ratio (TSR) values, as the ratio of ITS_{wet} to ITS_{dry} , were calculated to evaluate the BSM-Es' water resistance.

4.3.3.2. Flow Number

The principles of the AASHTO T 378-22 standard Flow Number testing methodology were adopted to assess the development of the BSM-Es' cohesive properties through permanent deformation response evaluation. Tests were performed using the Asphalt Mixture Performance Tester (Figure 19) on a minimum of three specimens for each BSM-E mixture.



Figure 19 Asphalt Mixture Performance Tester (AMPT) device for Flow Number and Dynamic Modulus testing:
a) testing setup with BSM-E specimen, b) BSM-E specimen after testing.

The adaptation of the dynamic creep and recovery testing methodology, primarily developed for hot and warm mix asphalt under the NCHRP 9-19 and NCHRP 9-29 projects, consisted of the selection of suitable Flow Number test parameters – loading stress level and temperature – to obtain reliable data on the primary, secondary and tertiary permanent deformation zones of the designed BSM-E mixtures. Based on the performed literature review on the current FN testing experience in cold recycling mixtures' testing (section 3.4.3), preliminary laboratory research was carried out to verify and adapt the commonly applied conditions to the BSM-E mixtures. The results of the preliminary testing carried out on Bitumen Stabilised Materials with bitumen emulsion were discussed in the work of Konieczna and Król [172]. It was observed that the material composition and volumetric properties of BSM-Es are of primary importance for the selection of testing conditions. While the application of low cyclic loading stress levels (i.e., 140 kPa and 200 kPa) did not allow for the estimation of FN

independently of the mixture characteristics and test parameters, the RAP content and air void content significantly influenced the BSM-Es' permanent deformation response at intermediate to high loading stress conditions (400 kPa, 600 kPa) and test temperatures of 40°C and 60°C. Based on the preliminary research results and taking into account the material and volumetric characteristics of the designed BSM-E mixtures, the following conditions were applied for Flow Number testing:

- Repeated axial stress level: 600 kPa,
- Confining stress: 0 kPa (unconfined tests),
- Test temperature: 50°C,
- Sample conditioning: no mixture conditioning; sample pre-test 4-hour conditioning at the test temperature.

The determination of Flow Number values was performed by fitting the data to the Francken model according to principles presented in sections 3.4.3 and 3.5.1. Data analysis was carried out based on the results provided by the testing software with the additional support of the Python programming language script with the use of statistical analysis and visualization packages (e.g., SciPy, NumPy, Pandas, Matplotlib), which was originally developed by the Author. The data analysis procedure is schematically presented in Figure 20.

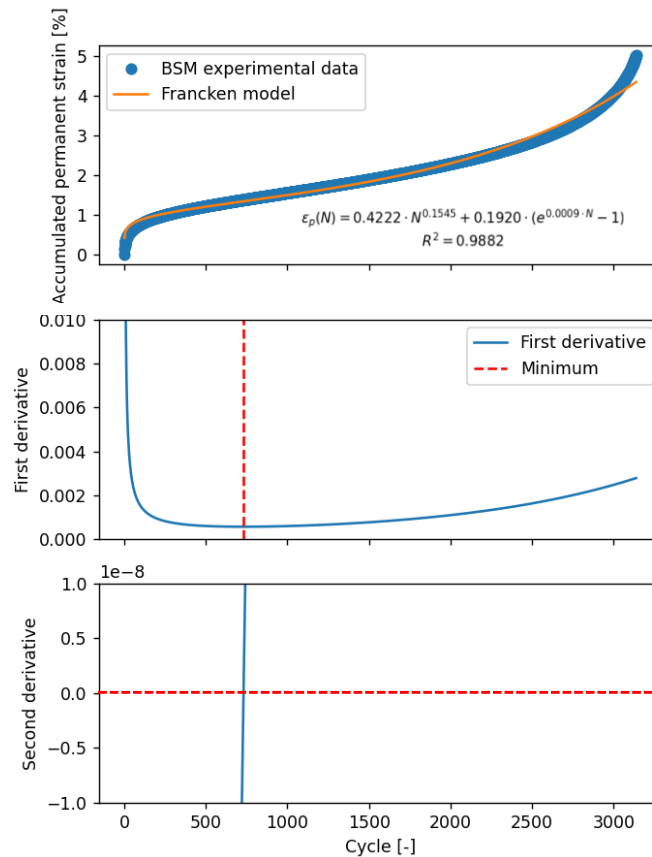


Figure 20 Flow Number testing data analysis procedure

The data analysis approach included a quantitative assessment of the Francken model fit based on the R^2 value and an analysis of the first and second derivatives' values for the FN value determination. A threshold of $R^2 \geq 0.70$ (70 %) was adopted as indicative of sufficient fitting accuracy.

4.3.3.3. *Dynamic Modulus*

Dynamic Modulus testing for the selected BSM-E mixtures was carried out in the AMPT device according to the AASHTO T 378-22 procedure. The testing machine enables the determination of the dynamic modulus for cylindrical specimens subjected to cyclic vertical loading applied at various temperatures and frequencies. The application of loading results in vertical deformation of the sample, which is recorded using three linear variable displacement transducers (LVDTs) evenly distributed along the specimen's perimeter at 120° intervals. The sensors were mounted in holders bonded to the specimen surface with epoxy adhesive prior to testing. The specimen testing setup is presented in Figure 21.

Based on the current testing experience in cold recycling mixtures' testing presented in the scientific literature (section 3.3.2), the following testing conditions were applied:

- Temperatures: 5°C, 10°C, 20°C, 40°C,
- Frequencies: 0.1 Hz, 0.5 Hz, 1 Hz, 2 Hz, 5 Hz, 10 Hz, 20 Hz, 25 Hz,
- Target peak strain amplitude: 50 – 70 $\mu\epsilon$,
- Sample conditioning: no mixture conditioning; sample pre-test 4-hour conditioning at the test temperature.



Figure 21 Dynamic Modulus testing setup

The tests for each specimen were carried out in a temperature sequence from the lowest to the highest. At every temperature level, measurements were performed in a single series, beginning with the highest frequency. For each set point, a minimum of two specimens per selected BSM-E type were tested, in accordance with the applicable standard requirements. For the evaluation of the BSM-Es' viscoelastic behaviour, the Dynamic Modulus $|E^*|$ with the storage (E') and loss (E'') moduli and phase angle ϕ were analysed. Dynamic Modulus master curves were constructed using the standard logistic sigmoidal-shaped (SLS) model and phase angle master curves were developed using the Gauss model with the Arrhenius shifting procedure.

4.3.3.4. Statistical analysis

The relationship between the ITS values and the bitumen emulsion content at the mix design stage was analysed using both simple and second-order polynomial regression models using the *Statgraphics 18* software. The statistical significance of the models was evaluated by means of the ANOVA *p-value*, with a significance level of $\alpha = 0.05$. In addition, the lack-of-fit test was performed to verify whether the deviations between the experimental data and the model predictions could be attributed to random error. A non-significant *p-value* in the lack-of-fit test was interpreted as an indication that the model adequately fitted the experimental data. The goodness-of-fit evaluation was complemented by the assessment of the coefficient of determination (R^2), which quantified the proportion of the variability in the ITS values explained by the regression model. The selected models were subsequently used to determine the BE contents corresponding to the maximum of the regression function, which were adopted for the BSM-E mix design.

The assessment of cohesion evolution was performed using Flow Number testing over 120 days of conditioning after specimen curing, with a full factorial Design of Experiment (DoE) approach adopted due to the nature of the studied phenomenon, which required tracking and describing changes in permanent deformation performance over time at successive individual time points.

The Flow Number values were determined using a *Python*-based script by fitting the Francken model (11) to the experimental data. The goodness-of-fit was evaluated using the coefficient of determination (R^2). The regression parameters were estimated using the Least Squares Method. The first and second derivatives of the fitted function were determined analytically to identify the inflection point (see section 4.3.3.2).

For comparisons of mean FN values between the analysed mixtures, a one-way ANOVA was first conducted to test the null hypothesis of equal means across all groups. The ANOVA assessing the statistical significance of differences between the groups of the FN test results was preceded by the Shapiro–Wilk normality test for each group and Levene’s test for homogeneity of variance. Given the sample size in each group (≤ 5 specimens) and the limited statistical power associated with more conservative post-hoc procedures such as Tukey’s HSD, the Fisher’s Least Significant Difference (LSD) test was applied as a protected post-hoc method. In this approach, pairwise comparisons were performed only if the overall ANOVA result was statistically significant at $\alpha = 0.05$, thereby controlling the inflation of Type I error. The LSD test was chosen to maximise sensitivity in detecting potential differences between small groups, and the results were interpreted with caution in light of the increased risk of false positives inherent to multiple comparisons.

In the case of the comparisons carried out in section 5.1.2, common Fisher’s LSD limits for the differences in the mean group values of FN, $\varepsilon(\text{FN})$, and the FN index (Table 19, Table 20, Table 21) were applied due to the equal group sizes.

The master curve fitting for dynamic modulus and phase angle, performed as part of the viscoelasticity assessment, was carried out in Microsoft Excel using the *Solver* add-in, with an iterative Least Squares Method applied to minimise the sum of squared residuals.

5. Cohesion and permanent deformation response assessment

The main objective of the thesis was to assess the cohesion development in Bitumen Stabilised Materials with bitumen emulsion (BSM-Es) over time.

In the first stage of the research, the initial cohesion of the mixtures was described by analysing the Flow Number test results collected directly after curing (0-day conditioning). Two separate groups of mixtures were distinguished to address two potential issues connected to the development of the mixture’s cohesion – namely: (1) the integrity of the internal structure connected to the mixture composition; (2) influence of the RAP content. Therefore, the first analysed group consisted of BSM-Es with various RAP contents (0% to 90%) and BE contents determined in the mix design process, while in the second group, mixtures with various RAP contents (0% to 90%) and a constant BE content (4%) were included. Additionally, the results obtained for BSM-E mixtures were compared to those of the reference HMA mixture (AC 22), characterised in section 4.2. FN (Flow Number), $\varepsilon(FN)$ - accumulated permanent strain at Flow Number, and $FNindex = \varepsilon(FN)/FN$ were introduced as the material cohesive performance

measures. The suitability of these parameters for effectively describing the material's permanent deformation response was assessed based on the statistical data analysis.

In the second stage of the research, the cohesion evolution phenomenon was evaluated throughout a 120-day conditioning period. Statistical analysis of test results allowed for an identification of the BSM-E mixtures that exhibited increase in the permanent deformation resistance over time. Short- and long-term conditioning sub-periods were distinguished using statistical analysis to quantify the changes in the FN and FN index parameters. Based on the established relationship between the mixtures' compositions and their level of cohesion development, BSM-E mixtures showing the potential for viscoelastic behaviour were identified and selected for the Dynamic Modulus testing.

5.1. Initial cohesion

5.1.1. Assessment of the internal structure integrity

The research aimed to assess the initial cohesion of BSM-Es, with particular emphasis on the influence of the material-related factors on resistance to permanent deformation. To interpret the nature of the material cohesion in terms of the ability to withstand dynamic creep loading, a series of mixtures – designed according to the principles outlined in section 4.2 and characterized by varying contents of RAP and BE – were analysed. The mechanical and durability performance of these mixtures, determined in the mix design stage, together with their physical and volumetric properties, constitute the concept of the internal structural integrity adopted in this study. Thus, the term *internal structure integrity* refers to the characteristics of the binder-mineral skeleton system within BSM-E, encompassing the distribution and interaction of the bituminous binder, aggregates, and air voids. It reflects the material's capacity to maintain a stable and uniform structure under load, influenced by factors such as RAP and BE content, total binder content, air void content and distribution, and the characteristics of the mechanical interlock of RAP and virgin aggregates supported by the localised bituminous bonds.

5.1.1.1. Mix composition

Based on the Indirect Tensile Strength test results carried out according to the procedure described in section 4.3.3.1 as a part of the mix design process, the ITS_{dry} and ITS_{wet} regression models for mixtures with different RAP contents were fitted (Figure 22, Figure 23). The plots include the graphical interpretation of the two-sided confidence limits (green-shaded, specified for current mean values of the dependent variable) and prediction limits (yellow-

shaded, specified for predicted individual values of the dependent variable). The confidence interval of 95% was applied for the statistical analysis. Equations of the fitted regression models and determined goodness-of-fit parameters: R^2 and p -value are presented in Table 11 and Table 12 for the ITS_{dry} and ITS_{wet} , respectively.

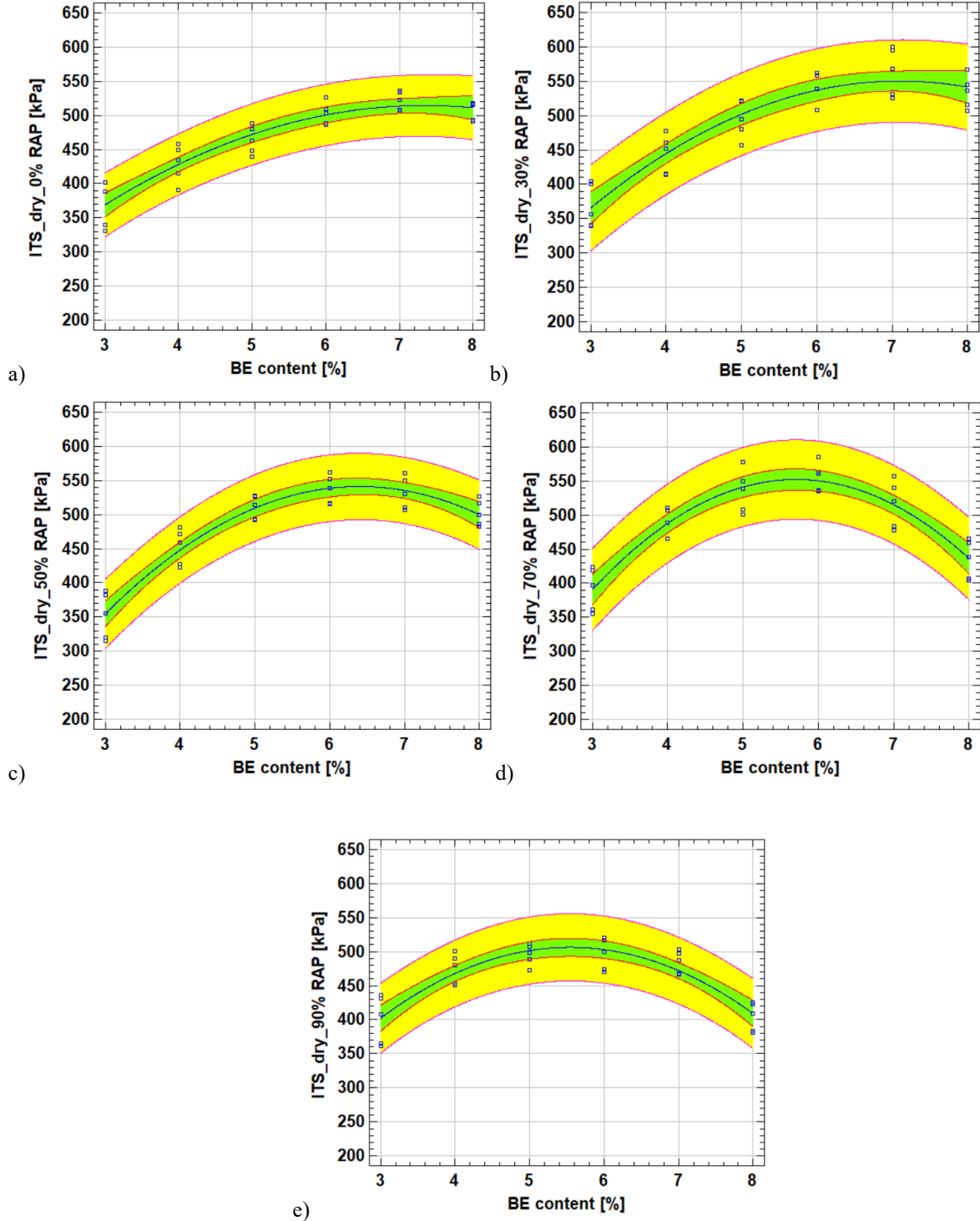


Figure 22 Regression models for ITS_{dry} test results for BSM-E mixtures containing: a) 0% RAP, b) 30% RAP, c) 50% RAP, d) 70% RAP, e) 90% RAP (source: *Statgraphics 18* software)

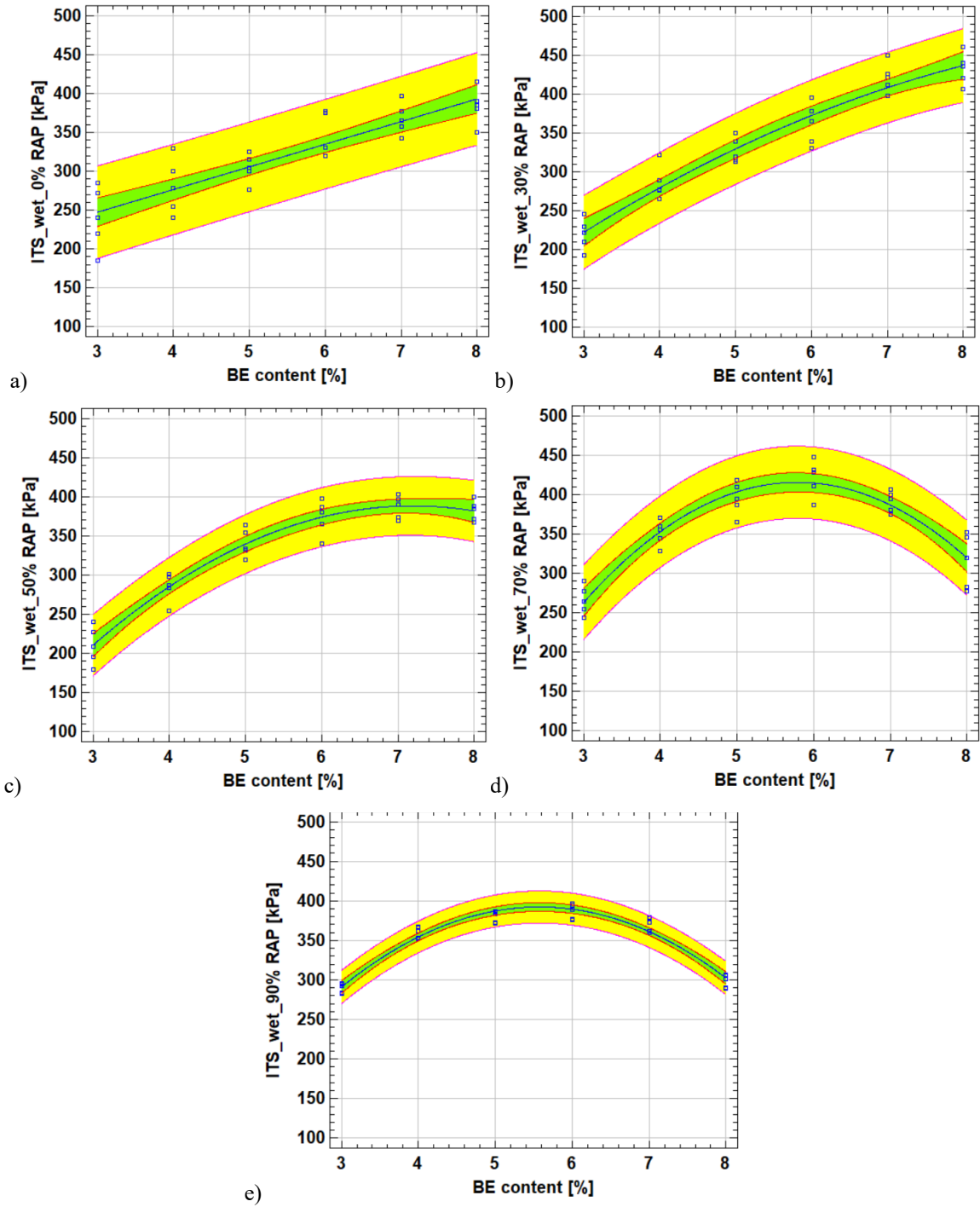


Figure 23 Regression models for ITS_{wet} test results for BSM-E mixtures containing: a) 0% RAP, b) 30% RAP, c) 50% RAP, d) 70% RAP, e) 90% RAP (source: *Statgraphics 18* software)

Table 11 Regression model equations for ITS_{dry}

RAP content [%]	ITS_{dry}		
	Regression model equation	R^2 [%]	p -value [-]
0	$ITS_{dry,0}(BE) = 98.3843 + 113.314 \cdot BE - 7.711786 \cdot BE^2$	86	0.6705
30	$ITS_{dry,30}(BE) = -2.63286 + 155.648 \cdot BE - 10.9607 \cdot BE^2$	85	0.6243
50	$ITS_{dry,50}(BE) = -119.514 + 206.414 \cdot BE - 16.1286 \cdot BE^2$	89	0.9441
70	$ITS_{dry,70}(BE) = -166.419 + 251.726 \cdot BE - 21.0536 \cdot BE^2$	82	0.9316
90	$ITS_{dry,90}(BE) = 11.8486 + 178.381 \cdot BE - 16.0929 \cdot BE^2$	75	0.4199

Table 12 Regression model equations for ITS_{wet}

RAP content [%]	ITS_{wet}		
	Regression model equation	R^2 [%]	p -value [-]
0	$ITS_{wet,0}(BE) = 159.846 + 29.1371 \cdot BE$	77	0.8252
30	$ITS_{wet,30}(BE) = 7.74571 + 82.2443 \cdot BE - 3.57857 \cdot BE^2$	92	0.2744
50	$ITS_{wet,50}(BE) = -134.717 + 145.413 \cdot BE - 10.1071 \cdot BE^2$	93	0.9975
70	$ITS_{wet,70}(BE) = -241.536 + 227.054 \cdot BE - 19.6179 \cdot BE^2$	86	0.6628
90	$ITS_{wet,90}(BE) = -82.46 + 170.327 \cdot BE - 15.2786 \cdot BE^2$	94	0.0800

As reported in Table 11 and Table 12, the obtained R^2 coefficient values for all analysed datasets were higher or equal to 70%, which allows for a statement that the applied polynomial and linear regression models can be considered well-fitted to experimental data, with a satisfactory percentage of variation in the values of the dependent variable being explained by the variation in the independent variable. This is confirmed by the results of p -value calculations for lack-of-fit, where for all analysed cases, this value was higher than 0.05.

Since the threshold values for $ITS_{dry}=225$ kPa and $ITS_{wet}=100$ kPa, presented in Wirtgen Cold Recycling Manual [10] for BSM Class 1 mixtures have been exceeded regardless the content of RAP and bitumen emulsion, the designed bitumen emulsion content in the BSM-E mixtures was identified as the amount corresponding to the highest value of the ITS_{dry} parameter (maximum of a regression function for an individual mixture). Additionally, TSR equal to 70% (as presented in the AASHTO MP31-22 [S2] standard) was adopted as a BSM-E threshold verification value for the BE content determination. The calculated TSR values are presented in Figure 24.

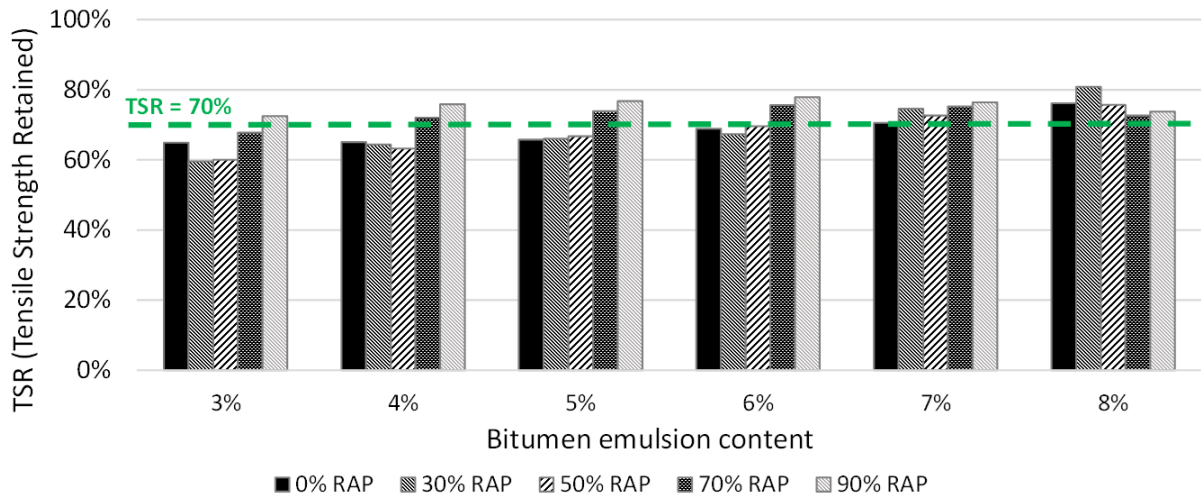


Figure 24 TSR values of the BSM-E mixtures.

It can be concluded that the average TSR values increased gradually with an increase of bitumen emulsion content for BSM-E mixtures containing from 0% to 50% RAP. For these mixtures, the TSR threshold values were obtained only when the bitumen emulsion content

ranged from 6% to 8% of the mixture mass. For the mixtures with 70% RAP and 90% RAP, TSR values higher than 70% were observed for lower bitumen emulsion contents – starting from 3% in the case of 90% RAP mixture and 4% for 70% RAP mixture. Based on the obtained ITS_{dry} and TSR values, the BE contents in BSM-E mixtures with different amounts of RAP were calculated as the values corresponding to the maximum value from the regression ITS_{dry} model equations. BE contents applied in the BSM-E mixtures are presented in Table 13.

Table 13 Determined bitumen emulsion content in BSM-E mixtures

RAP content [%]	Bitumen emulsion content [%]
0	7.4
30	7.1
50	6.4
70	5.7
90	5.5

As shown in Table 13, the RAP and BE contents in the designed BSM-E mixtures change together systematically, with the emulsion content decreasing with the increase in the RAP content. Based on the conducted mix design process, the following BSM-E mixtures presented in Table 14 were selected for the assessment of the cohesion and permanent deformation response.

Table 14 Mixture compositions of the designed BSM-E mixtures

No.	Mixture designation	Mineral mixture		Cement	BE	Water
1	0%R 7.4%E	100% VA		1%	7.4%	According to the OFC calculation
2	30%R 7.1%E	30% RAP	70% VA		7.1%	
3	50%R 6.4%E	50% RAP	50% VA		6.4%	
4	70%R 5.7%E	70% RAP	30% VA		5.7%	
5	90%R 5.5%E	90% RAP	10% VA		5.5%	

Based on the average ITS_{dry} and ITS_{wet} values with standard deviations (Table 15), it can be stated that the mixtures were characterized with similar indirect tensile strength in the unsoaked and soaked state, ranging from 502 kPa to 543 kPa and 371 kPa to 411 kPa, respectively. The air void content (V_m) ranged from 10.3% to 12.3%, which stays within the required 10% – 15% range [10].

Table 15 Designed BSM-E mixtures – mechanical and volumetric properties

No.	Mixture designation	ITS_{dry} – average [kPa]	ITS_{dry} – st.dev. [kPa]	ITS_{wet} – average [kPa]	ITS_{wet} – st.dev. [kPa]	V_m – average [%]	V_m – st.dev. [%]
1	0%R 7.4%E	507	21	371	29	12.3	0.9
2	30%R 7.1%E	543	29	403	21	11.0	0.8
3	50%R 6.4%E	539	24	382	19	10.8	0.5
4	70%R 5.7%E	540	28	411	25	10.3	0.7
5	90%R 5.5%E	502	22	380	10	10.7	0.5

5.1.1.2. Flow Number testing

Figure 25 presents the results of Flow Number testing performed directly after curing (FN_0) on the designed BSM-E mixtures. Based on the presented data, it can be stated that the mixtures with no RAP and 30% RAP were characterized by the highest FN_0 values. No statistically significant difference between these FN_0 group means was observed at the 95% confidence level, as confirmed in the ANOVA test ($p\text{-value} = 0.353$). For the mixtures with an intermediate to high RAP content (50%–90%), lower average FN_0 values ranging from 839 cycles (90%R_5.5%E mixture) to 1310 cycles (70%R_5.7%E mixture) were recorded. By performing ANOVA in a combination with the multiple sample comparison using the Fisher's LSD post-hoc test, it was confirmed that for this group of mixtures, the differences between the FN_0 means were statistically significant, resulting in treating these mix compositions as separate cases. Despite presenting similar mechanical and volumetric properties (Table 15), the 70%R_5.7%E mixture exhibited a 21% increase in the number of cycles to failure compared to the 50%R_6.4%E mixture, while BSM-E with the highest amount of RAP and 5.5% BE was considered the most susceptible to strain accumulation in dynamic creep testing.

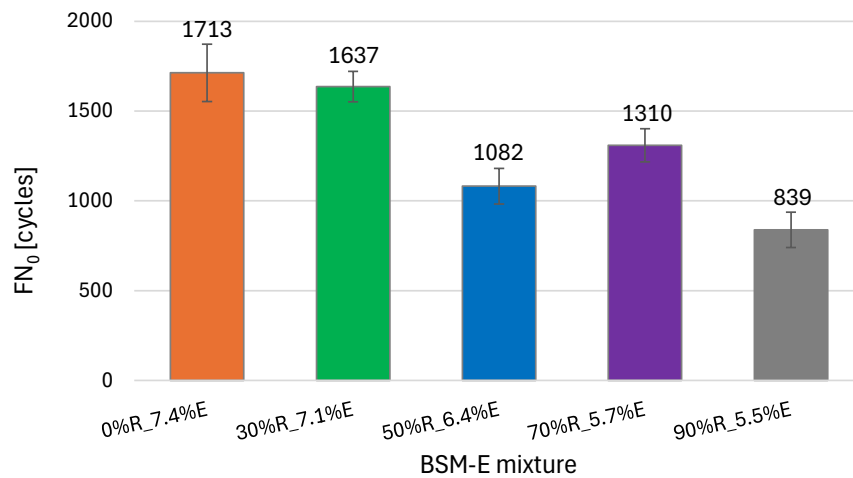


Figure 25 FN_0 values for the designed BSM-E mixtures at 0-day conditioning

Figure 26 presents the FN curves for the BSM-E mixtures with the Francken curve inflection points, corresponding to the Flow Number, marked in circles. The tests were performed until the accumulated permanent strain threshold of 3% was reached. However, for the 0%R_7.4%E mixture, testing was terminated at approximately 2.5% of the accumulated permanent strain due to rapid cracking and specimen disintegration in the final phase of testing. Analysis of the gathered data indicates that the low-RAP BSM-E mixtures exhibited a notably different performance compared to mixtures with intermediate and high RAP content. In addition to achieving higher FN values, significantly lower levels of the accumulated permanent

strain at failure ($\epsilon(FN_0)$) were observed for these mixtures, resulting in a reduced slope of the power-law segment of the Francken function (Figure 26). Moreover, although the average FN_0 values for the 0%R_7.4%E and 30%R_7.1%E mixtures were statistically equivalent, clear differences were observed in the rate and progression of permanent strain accumulation. In the first stage of testing, the 0%R_7.4%E mixture exhibited a lower accumulated permanent strain, indicating a reduced potential for the initial densification. However, during the subsequent stage characterised by a constant strain accumulation rate, the 0% RAP BSM-E mixture showed a more rapid increase in permanent strain compared to the BSM-E mixture containing 30% RAP. This eventually resulted in both mixtures reaching a similar number of cycles to shear failure and a comparable level of the accumulated permanent strain at FN.

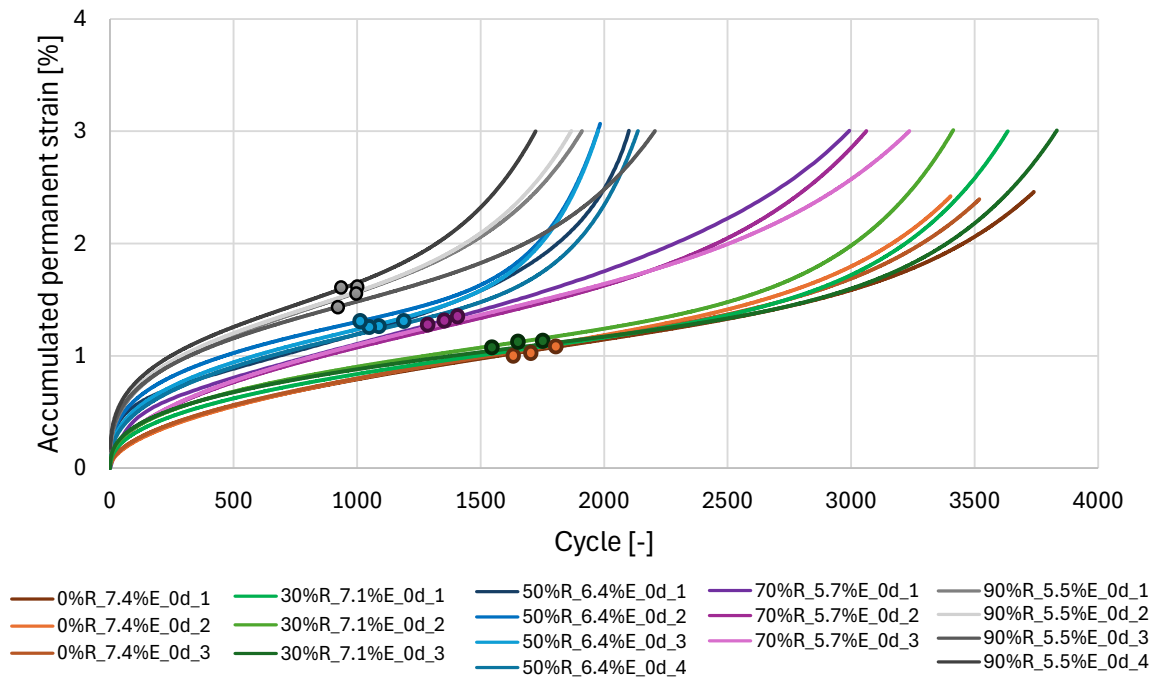


Figure 26 FN curves for the designed BSM-E mixtures at 0-day conditioning

Despite the fact that significant differences in FN_0 were observed for mixtures containing 50% and 70% RAP (Figure 25), the accumulated permanent strain levels at FN_0 were notably similar in both cases, as shown by the FN curves (Figure 26). In contrast, the 90%R_5.5%E mixture exhibited the most rapid increase in permanent strain during testing, ultimately resulting in the lowest number of cycles to shear failure among the tested mixtures and the highest accumulated permanent strain at FN. Therefore, due to the ambiguities in the FN_0 values and the characteristics of the accumulated permanent strain curves observed for specific BSM-E mixtures, the accumulated permanent strain at FN – $\epsilon(FN_0)$ – was introduced as an additional performance evaluation parameter. A material is considered more resistant to permanent deformation if it exhibits a higher FN value and/or a smaller $\epsilon(FN)$ value. The

proposed approach is also supported by findings from studies on HMA performance, which indicate that identical FN values can correspond to significantly different levels of permanent strain [173], [174]. Figure 27 presents the average $\epsilon(FN_0)$ values for the tested mixtures along with the results' dispersion.

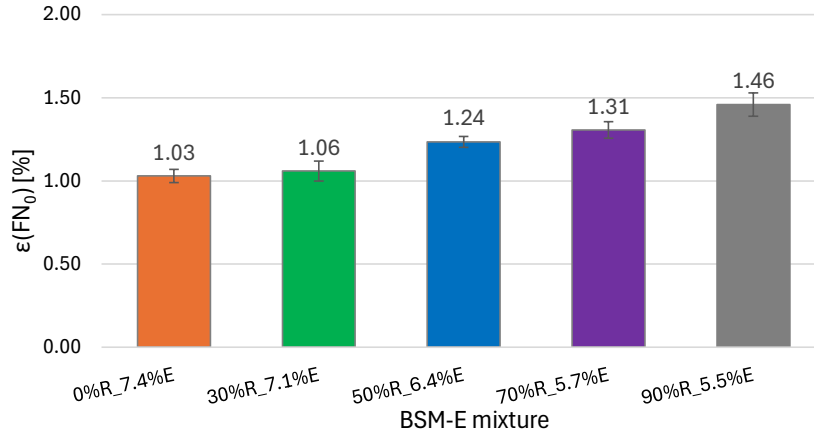


Figure 27 $\epsilon(FN)$ values for the designed BSM-E mixtures at 0-day conditioning

Based on the statistical analysis of the data presented in Figure 27, it can be stated that mixtures with the lowest RAP content were characterized by the lowest $\epsilon(FN_0)$ values, with no statistically significant difference determined in ANOVA test. The lowest level of accumulated permanent strain observed for mixtures with a low RAP content, combined with a high FN, may indicate that their significant resistance to permanent deformation may be – apart from the effect of the localised bituminous bonding – also a result of high internal friction within the mixture skeleton, which consists predominantly of virgin aggregate.

The determined level of the accumulated permanent strain at failure for the 0%R_7.4%E and 30%R_7.1%E mixtures can be considered relatively low as for mixtures with the highest “fresh” residual bitumen contents (approx.. 4.39% for 0%R_7.4%E and 4.22% for 30%R_7.1%E). In contrast, the mixture with 90% RAP content and approximately 3.27% residual bitumen content exhibited the greatest strain accumulation at shear failure ($\epsilon(FN_0) = 1.46\%$), corresponding to an increase of nearly 40% relative to the low-RAP mixtures. In the case of 50%R_6.4%E and 70%R_5.7%E mixtures, statistical analysis revealed that their average $\epsilon(FN_0)$ values can be considered equal ($p\text{-value} = 0.127$), simultaneously demonstrating an average 22% increase in the accumulated permanent strain at FN_0 compared to the low-RAP mixtures.

For a comprehensive assessment of the cohesion of BSM-E mixtures, an additional parameter was introduced – *FN index*, which combines the FN and $\epsilon(FN)$ parameters and

represents the average permanent strain increase per load cycle within the range of load cycles $N \in [1; FN]$. The lower the FN index value (i.e., smaller $\varepsilon(FN)$ and/or higher FN), the slower the accumulation of permanent strain in successive cycles, indicating that the material is more resistant to permanent deformation.

Figure 28 presents the calculated FN index values for the BSM-E mixtures.

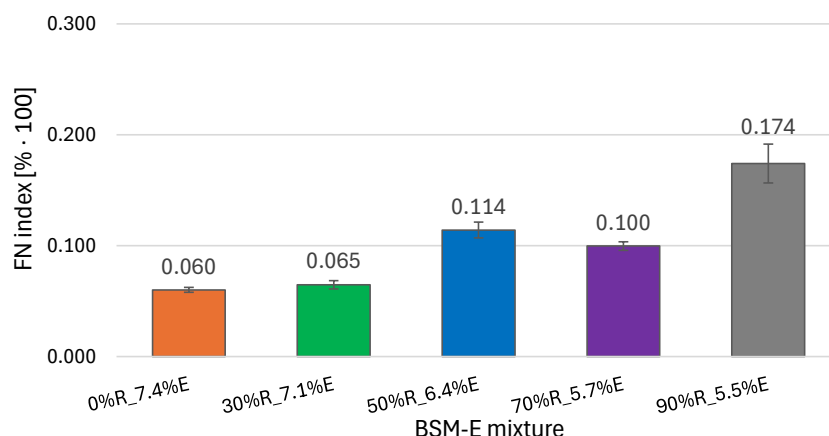


Figure 28 FN index for the designed BSM-E mixtures at 0-day conditioning

The analysis of the FN index confirmed a similar level of resistance to permanent deformation in the case of the 0%RAP and 30%RAP mixtures. With the average FN index value of 0.063 [%·100] resulting from high number of cycles to failure and low corresponding accumulated strain, mixtures of the mineral skeleton consisting mainly of virgin aggregates were assessed as the most resistant to deformation in dynamic creep testing. Analysis of the FN index for 50%RAP and 70%RAP mixtures, which exhibited intermediate FN and $\varepsilon(FN)$ values, revealed a differentiation in the rate of permanent deformation accumulation, characterizing the 50%R_6.4%E mixture as less resistant (a 14% increase in the FN index compared to the 70%R_5.7%E mixture), which is consistent with the conclusion made based on FN_0 . The FN index for the BSM-E mixture containing 90% RAP reached the highest value among the tested mixtures, indicating a nearly 3 times faster permanent deformation accumulation than in the case of 0%R_7.4%E and 30%R_7.1%E mixtures.

Due to the varying contents of both RAP and bitumen emulsion, an additional parameter was introduced to characterise the analysed mixtures – the total binder content (TBC), which includes the estimated content of aged RAP binder and the “fresh” residual bitumen from bitumen emulsion, expressed as a percentage of the mineral mixture mass.

Table 16 Total Binder Content in BSM-E mixtures

No.	Mixture	TBC [%]	Residual bitumen share in TBC [%]
1	0%R 7.4%E	4.39	100
2	30%R 7.1%E	5.66	75
3	50%R 6.4%E	6.20	61
4	70%R 5.7%E	6.75	50
5	90%R 5.5%E	7.59	43

Figure 29 presents plots of the fitted linear regression trends obtained in the *Statgraphics 18* software and describing the relationships between TBC and FN, $\epsilon(\text{FN})$ and FN index values. Based on the statistical analysis, it can be stated that strong correlations were observed for all permanent deformation performance parameters. In the case of FN (Figure 29 a)), a strong negative correlation ($r = -0.86$) was noted, indicating a general trend of the decrease in permanent deformation resistance with the increase in total binder content in the mixture. In contrast, the amount of binder in the mixture positively correlates with the accumulated permanent strain at FN (Figure 29 b)) ($r = 0.92$) and the FN index (Figure 29 c)) ($r = 0.87$).

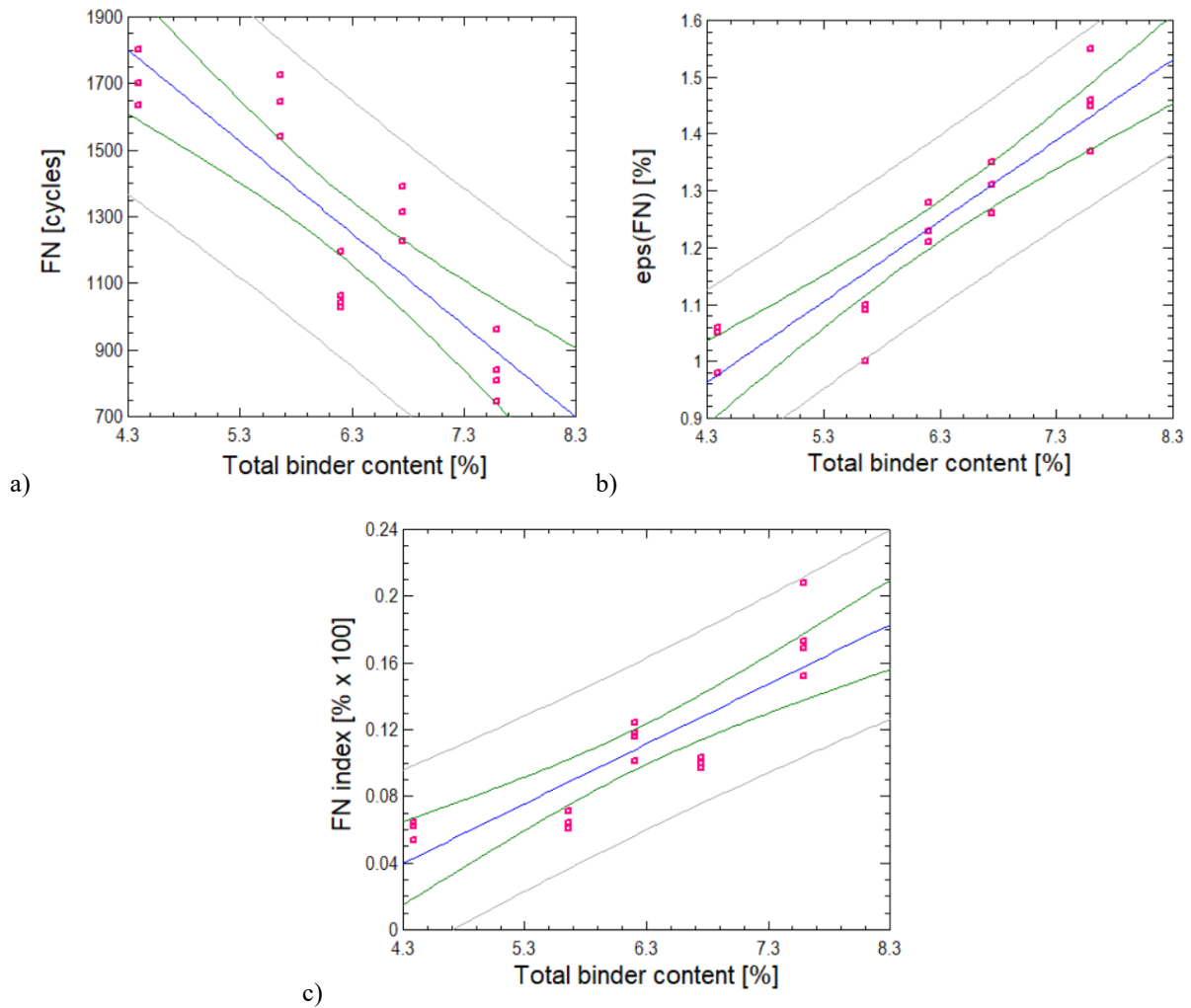


Figure 29 Permanent deformation performance parameters vs. total binder content: a) FN, b) $\epsilon(\text{FN})$, c) FN index (source: *Statgraphics 18* software).

However, it should be acknowledged that the low-RAP mixtures (0%R_7.4%E and 30%R_7.1%E) presented the same level of the permanent deformation resistance, based on FN_0 , $\epsilon(FN_0)$, and FN index, despite the 29% difference in the TBC (Table 16). These mixtures were also characterised by the highest share of the residual bitumen in TBC (>75%). In the case of mixtures with intermediate to high RAP content, the differences between the total binder contents, although less distinct (8.9% – 12.4%), allowed for a differentiation of their permanent deformation resistance. The observed tendencies allow the conclusion that, although RAP is introduced into the BSM-E mixture as a "black rock" and does not exhibit cohesive properties at ambient temperatures under impact loading (see section 4.2.1), it may still contribute to the development of cohesion. This effect is likely due to interactions between the aged binder and the residual bitumen, which might promote the formation of bituminous bonds.

Considering the permanent deformation development trends observed for the designed mixtures with varying RAP and bitumen emulsion content, it was decided to establish a control group of BSM-Es with a fixed emulsion content and a variable RAP content ranging from 0% to 90%, to solely investigate the influence of RAP content on the development of initial cohesion of BSM-Es.

5.1.2. Analysis of the influence of the RAP content on cohesion

To determine the impact of the RAP content on the BSM-Es' initial cohesion development, a constant BE content of 4% (by mineral mixture mass) was adopted for the whole set of mixtures, regardless of the TSR value (see Figure 24). Such BE amount is also recommended in the Wirtgen Cold Recycling Manual guideline for the active filler requirements determination based on the mixtures' grading curve (fine fractions amount of 7%–10% and the 0/4.75 mm aggregate share lower than 50%).

Table 17 presents the mixture compositions of the BSM-E mixtures with a constant BE content. The mechanical and volumetric properties of the mixtures are shown in Table 18.

Table 17 Mixture compositions of the designed BSM-E mixtures

No.	Mixture designation	Mineral mixture		Cement	BE	Water
1	0%R_4%E	100% VA		1%	4%	According to the OFC calculation
2	30%R_4%E	30% RAP	70% VA			
3	50%R_4%E	50% RAP	50% VA			
4	70%R_4%E	70% RAP	30% VA			
5	90%R_4%E	90% RAP	10% VA			

Table 18 BSM-E mixtures – mechanical and volumetric properties

No.	Mixture designation	ITS _{dry} – average [kPa]	ITS _{dry} – st.dev. [kPa]	ITS _{wet} – average [kPa]	ITS _{wet} – st.dev. [kPa]	V _m – average [%]	V _m – st.dev. [%]
1	0%R 4%E	430	27	280	35	13.7	0.5
3	30%R 4%E	444	28	286	23	13.6	0.9
5	50%R 4%E	452	26	285	19	12.7	0.8
7	70%R 4%E	487	22	350	24	12.0	0.6
9	90%R 4%E	475	22	359	8	11.6	0.7

Figure 30 presents the results of Flow Number testing performed directly after curing (FN₀) on BSM-E mixtures with a 4% bitumen emulsion and a reference FN value obtained for the HMA AC 22 mixture. By performing the ANOVA test in a combination with the multiple sample comparison using the Fisher's LSD post-hoc test, it was confirmed that the differences between the FN₀ group means for BSM-Es are statistically significant at the 95% confidence level, with an exception for a pair of mixtures: 50%R_4%E and 70%R_4%E (Table 19).

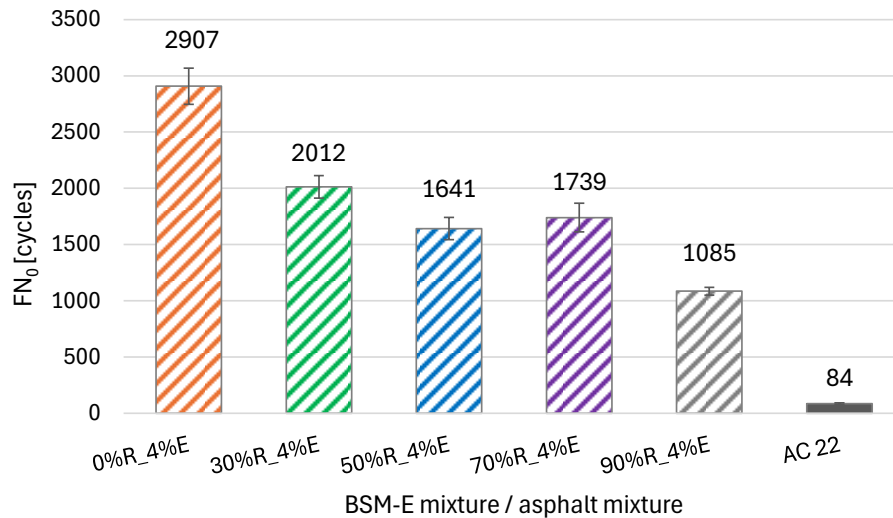


Figure 30 FN values for BSM-E mixtures with 4%BE and asphalt mixture AC 22 at 0-day conditioning

Table 19 Statistical analysis of FN values of BSM-E mixtures with 4%BE at 0-day conditioning

ANOVA	F-ratio	108.49	p-value	0.000
LSD Fisher	Limit difference ±202.707 [cycles]			
Mixture	Differences (FN value):			
30%R_4%E	850			
50%R_4%E	1266	416		
70%R_4%E	1168	318	-98	
90%R_4%E	1823	972	557	655
	0%R_4%E	30%R_4%E	50%R_4%E	70%R_4%E

Based on the obtained FN results, it can be stated that generally the Flow Number values decreased with the increase in the RAP content in BSM-Es, which was confirmed by the Pearson correlation coefficient analysis, revealing a strong negative linear relationship ($r = -0.94$) between these two variables. The 0%R_4%E mixture containing solely virgin

aggregates was characterized with the highest resistance to permanent deformation (FN = 2907 cycles), with the FN value 44% higher than the one obtained for the BSM-E mixture with 30% RAP content and nearly 2.7 times higher compared to the 90%R_4%E mixture. 50%R_4%E and 70%R_4%E mixtures were characterised by an intermediate level of permanent deformation resistance, equal to 1690 cycles on average, while BSM-E with the highest RAP addition exhibited the lowest number of cycles to failure.

To explain the observed influence of RAP content on the permanent deformation performance of the tested mixtures, it is important to consider how differences in the qualitative composition of the finest aggregate fractions (<0.063 mm) – the matrix for residual bitumen setting – might affect the dynamics of bitumen emulsion breaking. Whereas in the mixtures containing 0% and 30% RAP, the contribution of the finest fractions derived from reclaimed material is either zero or negligible, in the mixtures with the highest RAP content, the share of fines from RAP reaches approximately 10% (see section 4.2.1). The issue of achieving adequate adhesion at the RAP–new binder interface in CRMs is complex. While some studies report week bonding within this zone [175], as noted by Herrington and Henning [176], the bitumen undergoing oxidative aging, exhibits changes in its polarity. Considering that the dynamics of emulsion breaking depends on, among other factors, the surface properties of aggregates (porosity, surface area and surface charge) [177], the RAP particles might be expected to provoke faster emulsion setting than virgin limestone and dolomite aggregates. This can be attributed to the fact that the polarity of bitumen in RAP increases due to the formation of carbonyl, sulfoxide and hydroxyl groups [176], which are either negatively charged or behave polar. For the cationic BEs, when in contact with oppositely charged particle surfaces, the electrostatic attraction promotes rapid coalescence and breaking of the emulsion, resulting in a more effective bonding of the residual bitumen to the aged binder on the RAP surface and, therefore, providing conditions for the cohesion development through the binder integration at the RAP surface. The positive charge present on the limestone (CaCO_3) and dolomite ($\text{CaMg}(\text{CO}_3)_2$) aggregates, can hinder the adhesion of cationic BEs due to electrostatic repulsion, potentially delaying the emulsion breaking process when in contact with carbonate aggregates. On the other hand, RAP particles might also exhibit the tendency to absorb water from emulsion at the initial stage of mixture production due to the expected presence of microcracks, greater surface area and an increased porosity of the aged aggregates, which might negatively influence the ability of the residual bitumen to distribute within the mineral skeleton, which can hinder the initial cohesion performance, as observed in this study. However, the application of an accelerated curing procedure involving oven conditioning at 40°C may disrupt

the processes described above and diminish the influence of the electrostatic effects, particularly in the surface layer of the specimens and as curing progresses over time. Nevertheless, the observed trends may still be relevant during the stages of mixing, compaction, and the initial phase of accelerated curing. As curing advances, progressive water evaporation may enhance the influence of physical factors on the formation of bituminous bonding through binder coalescence, potentially increasing the significance of parameters such as mixture grading, as well as the content and structure of air voids within the material.

The significantly lower average FN value obtained for asphalt mixture (84 cycles) indicates notable differences in the mechanisms of the permanent deformation accumulation present in BSM-E and HMA mixtures, which result from the characteristics of the bituminous bonding (e.g., binder film continuity within the mineral skeleton variously formed in hot and cold production processes). These findings can be confirmed by the differences in the accumulated permanent strain levels ($\epsilon(FN_0)$) observed for these mixtures (Figure 31, Figure 32).

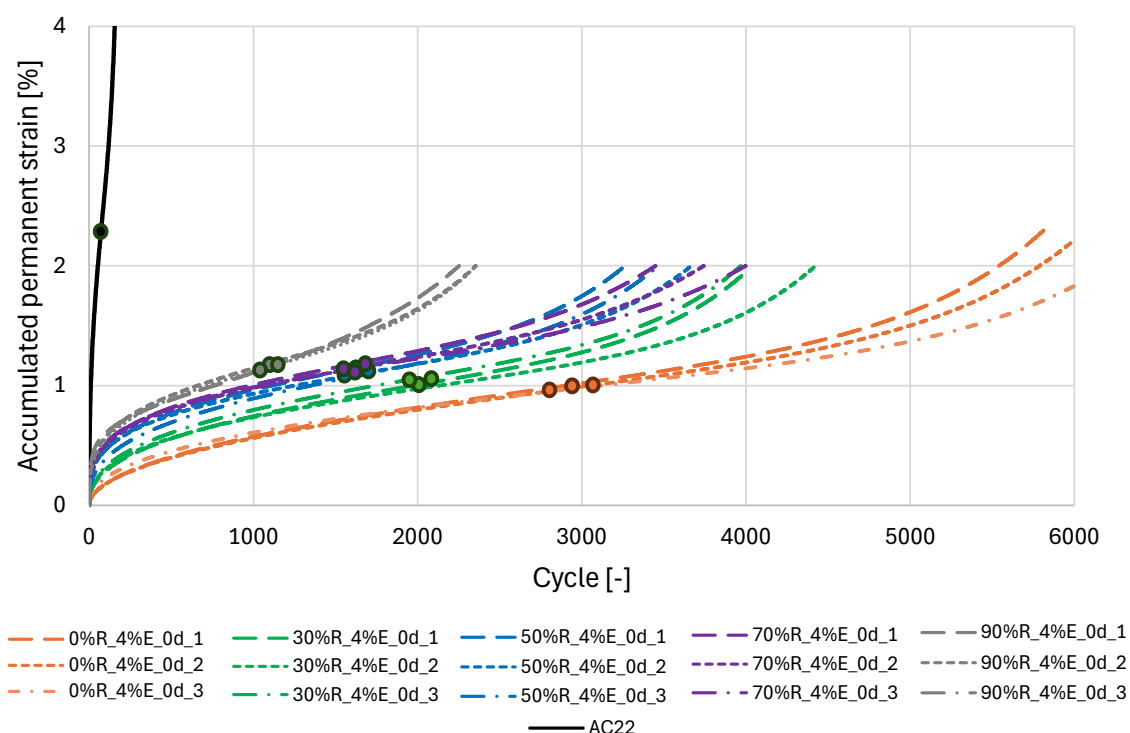


Figure 31 FN curves for BSM-E mixtures with 4%BE and asphalt mixture AC 22 at 0-day conditioning

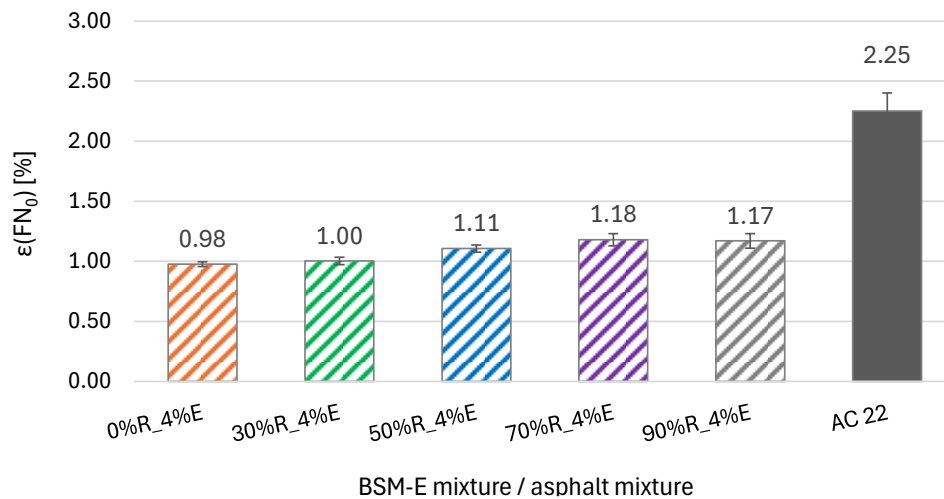


Figure 32 Accumulated permanent strain at FN of BSM-E mixtures with 4%BE and asphalt mixture AC 22 at 0-day conditioning

For BSM-E mixtures without RAP and containing 30% RAP, the obtained $\epsilon(FN_0)$ values were approximately 2.3 times lower than in the case of AC 22. This indicates a different failure mechanism of the specimens – BSM-E mixtures exhibited cracking within the specimen with only minor height reduction during testing, while in HMA mixtures, deformation was primarily associated with aggregate rearrangement while maintaining mixture cohesion through continuous asphalt mastic, allowing for exhibiting greater deformation during testing. BSM-Es with the intermediate to high-RAP contents presented higher $\epsilon(FN_0)$ values (1.11%–1.17%) than the low-RAP ones. Based on this observation, an assumption can be made that an increase in $\epsilon(FN_0)$ values may suggest that the mixture's properties are evolving toward a more viscoelastic behaviour. Even though a strong positive correlation between $\epsilon(FN_0)$ and RAP content was noted ($r = 0.86$), no statistically significant differences were observed for $\epsilon(FN_0)$ between the 70%R_4%E and 90%R_4%E mixtures, as well as the 0%R_4%E and 30%R_4%E mixtures, despite significant differences in their FN_0 . $\epsilon(FN_0)$ was also not a distinguishing parameter for the performance of the 50%R_4%E mixtures compared to the 70%R_4%E and 90%R_4%E mixtures (Table 20). For this reason, the FN index was additionally analysed to classify the mixtures' resistance to permanent deformation.

Table 20 Statistical analysis of $\epsilon(FN)$ values of BSM-E mixtures with 4%BE at 0-day conditioning

ANOVA		F-ratio	13.74	p-value	0.001
LSD Fisher		Limit difference ±0.080 [%]			
Mixture	Differences (eps(FN) value):				
30%R_4%E	-0.017				
50%R_4%E	-0.130	-0.113			
70%R_4%E	-0.200	-0.183	-0.073		
90%R_4%E	-0.190	-0.173	-0.063	0.01	
	0%R_4%E	30%R_4%E	50%R_4%E	70%R_4%E	

Figure 33 presents the FN index values. Based on the data summarized in Table 21, the significant variation of the BSM-Es' rate of permanent deformation accumulation depending on the RAP content in the mixture was confirmed.

Table 21 Statistical analysis of FN index values of BSM-E mixtures with 4%BE at 0-day conditioning

ANOVA	F-ratio	85.59	p-value	0.000
LSD Fisher	Limit difference ±0.009 [% x 100]			
Mixture	Differences (FN index value):			
30%R_4%E	-0.015			
50%R_4%E	-0.034	-0.019		
70%R_4%E	-0.035	-0.020	0.000	
90%R_4%E	-0.074	-0.059	-0.040	-0.039
	0%R_4%E	30%R_4%E	50%R_4%E	70%R_4%E

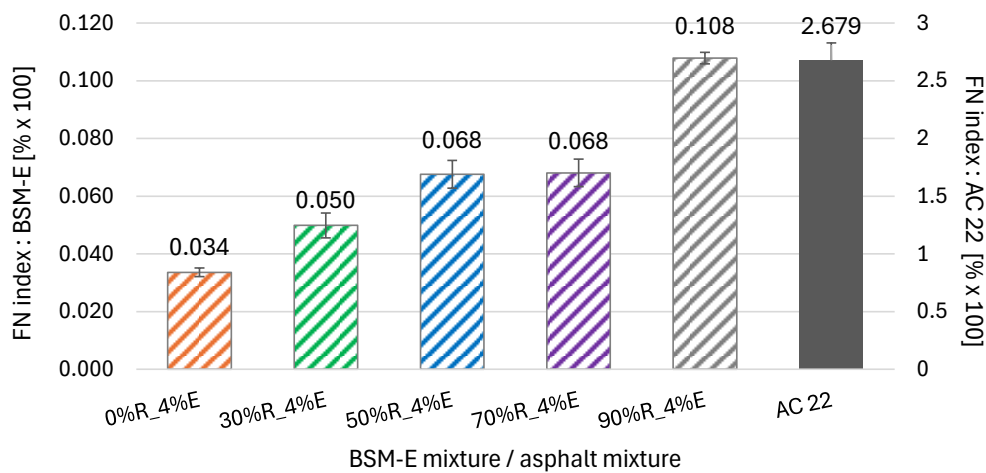


Figure 33 FN index of BSM-E mixtures with 4%BE and asphalt mixture AC 22 at 0-day conditioning

A general trend of the increase in the FN index values with an increase in RAP content was observed with a strong positive correlation described by the Pearson coefficient $r = 0.85$. The low-RAP content mixtures were characterized with the slowest strain increase until shear failure, with the increase rate for the 0%R_4%E mixture being 2 times smaller than in the case of BSM-E mixtures with 50% and 70% RAP addition, which reached the same FN index value of 0.068 [% x 100]. The BSM-E mixture with the highest RAP content exhibited the fastest strain accumulation rate, showing an even greater distinction compared to the 0%R_4%E mix when the FN_0 value was analysed (3.18 times lower value). Based on the FN index results obtained for the BSM-E mixtures and the reference asphalt mixture (AC 22), a clear difference can be observed in the nature of the permanent deformation resistance and the cohesion of these materials. While the BSM-E mixtures reached FN index values in the range of 0.034–0.108 [% x 100], the AC 22 mixture exhibited a value of 2.679 [% x 100], i.e., approximately 25–80 times higher. This substantial disparity reflects the fundamentally different mechanisms governing the performance of both types of mixtures, owing to the differing characteristics of

the bitumen phase, air void content, and internal structure. The high permanent deformation resistance observed in the BSM-E mixtures containing cement and characterised by an air void content of approximately 14% can be largely attributed to the internal friction of the mineral skeleton. However, the role of bituminous bonding characteristics in controlling the material response becomes increasingly evident with higher RAP contents (increase in FN index), as demonstrated in the analysed case where the residual bitumen content remains constant at a relatively low level (approx. 2.4%). In contrast, due to the continuous bituminous bonding present in HMA, the cohesion and integrity of the mixture are primarily determined by the presence of the asphalt mastic, along with a low air void content (4.5%) and a continuous aggregate grading.

Figure 34 and Figure 35 present exemplary X-ray CT scans of cross-sections (on the left) and their binary images (on the right) for mixtures 30%R_4%E and 90%R_4%E, respectively. The images were captured at the mid-height of the gyratory-compacted 150-mm high specimens prepared for Flow Number testing. In the binary images, the air voids were presented as the black areas, while the remaining composite structure was marked in white.

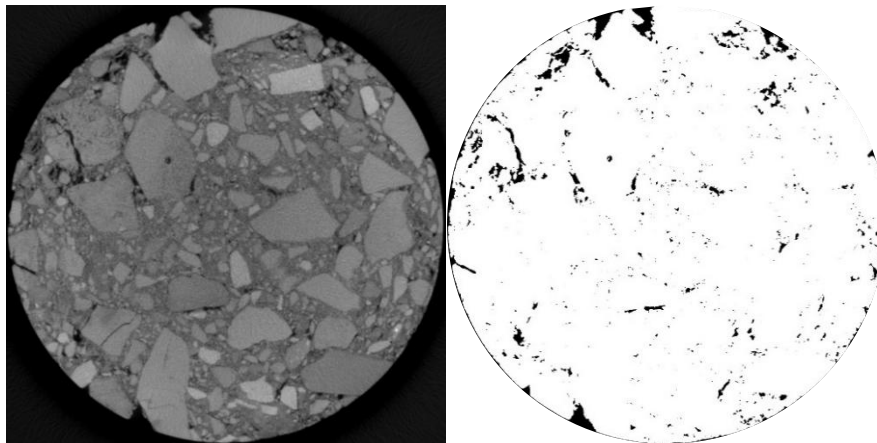


Figure 34 X-ray CT scan of the 30%R_4%E sample cross-section with binary image

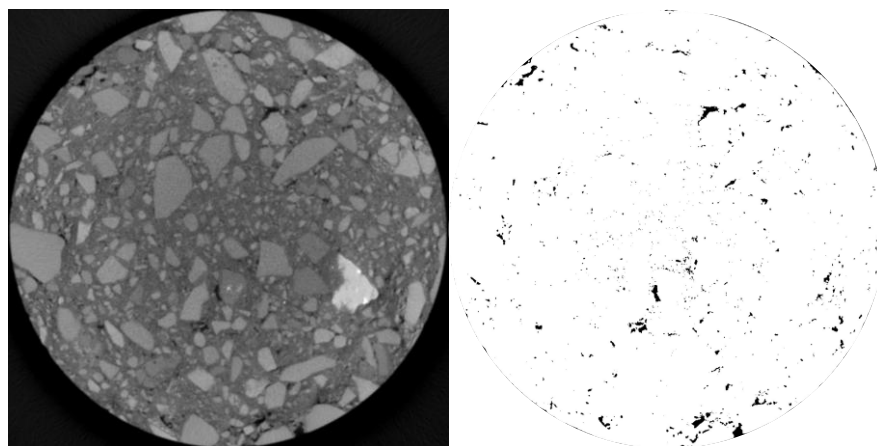


Figure 35 X-ray CT scan of the 90%R_4%E sample cross-section with binary image

Based on the selected scans, it can be concluded that the differences in the mineral mixture composition of BSM-Es containing 30% and 90% RAP influence the distribution of air voids within the composite. A qualitative assessment of the binary images indicates that in the mixture with lower RAP content the air voids exhibited less uniform distribution across the cross-section, with larger clusters located near the coarsest aggregate particles (Figure 34). This is most likely a result of the limited compactability of mixtures with such mineral skeleton characteristics. The presence of locally concentrated voids may lead to areas within the composite where interparticle bituminous bonding is weakened, thereby reducing the effectiveness of the skeleton in transferring loads and diminishing the overall material cohesion. Even though this observation was not reflected in the FN values, the obtained low $\varepsilon(FN_0)$ levels and observed tendency for surface cracking of the specimens during testing might indicate different mechanical characteristics of the mixtures with lower RAP content compared to the high-RAP mixtures. In contrast, in the 90%R_4%E mixture the air void distribution was more homogeneous, with a higher proportion of small, discontinuous voids more evenly dispersed across the cross-section (Figure 35). This phenomenon is favourable for enhancing material cohesion and promoting effective load transfer through interparticle contact within the aggregate skeleton and localised bituminous bonds.

5.1.3. Bitumen emulsion content discussion in terms of shear failure

To assess the influence of varying bitumen emulsion content on the initial mixture cohesion, a comparison was made of the FN_0 , $\varepsilon(FN_0)$, and FN index parameters for corresponding BSM-E mixtures with successive RAP contents. As presented in Figure 36, all mixtures with a 4%BE content, regardless of the amount of RAP, exhibited higher number of cycles to shear failure compared to the corresponding mixtures with higher BE contents. It can be related to the fresh residual bitumen content in the mixture, which might influence the characteristics of the bituminous bonding in the mixture and consequently promote the development of the BSM-Es' susceptibility to permanent deformation, especially notable at the applied testing temperature of 50°C. The biggest difference in FN_0 values was observed for BSM-Es with 0% RAP (an approximately 70% increase in the case of 0%R_4%E mixture), for which the BE content varied the most. For mixtures with 90% RAP content, the observed differentiation between FN_0 values was the smallest, which corresponds to the smallest difference in the BE content in the mixture (4.0% vs. 5.5%). No clear relationship was observed between differences in the obtained FN_0 values and variations in air void content among the analysed mixture pairs, since

statistically insignificant differences were noted in both cases of mixtures without RAP and those with the highest RAP amount (Figure 37).

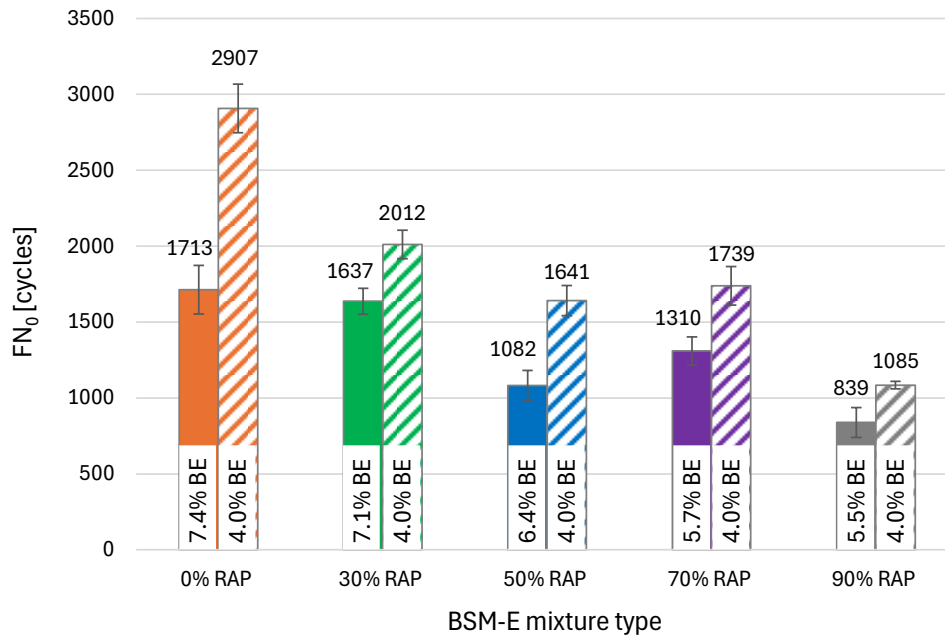


Figure 36 Comparison of FN_0 values for BSM-E mixtures with corresponding BE contents

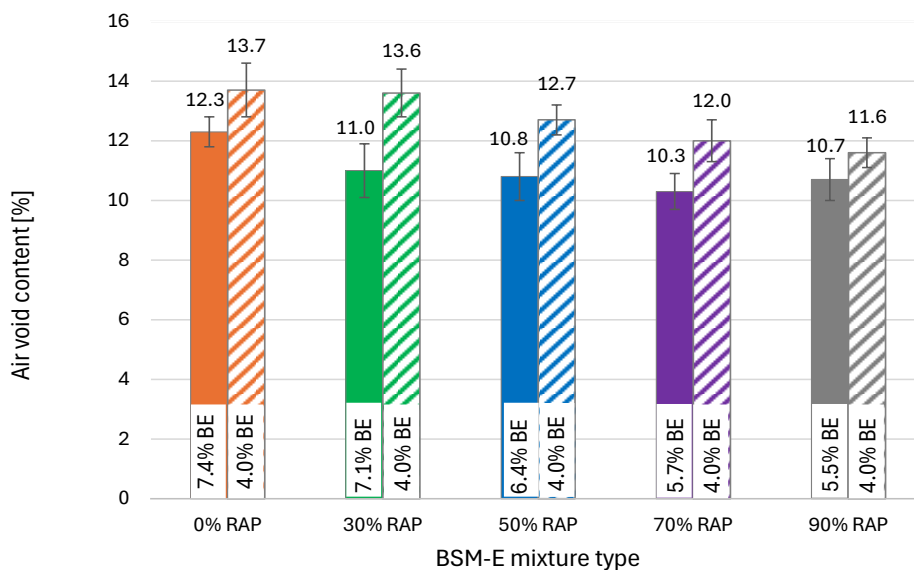


Figure 37 Comparison of air void content for BSM-E mixtures with corresponding BE contents

For BSM-Es containing 30% to 70% RAP, the mixtures with a 4% BE addition were characterised by a higher air void content compared to the variants with higher amount of emulsion. This may be attributed to the proportionally increased water content in these mixtures, which is more likely to be extracted during sample compaction and to evaporate during curing, resulting in a greater formation of air voids within the structure. Based on the obtained results, it can be stated that BSM-Es with BE contents determined in the mix design

process exhibited improved compactability. Nevertheless, the increased percentage of voids combined with a 4%BE content (resulting in approximately 2.4% of residual bitumen) did not have a negative influence on the particle interlock and bonding within the internal structure of the analysed BSM-Es, as reflected in FN_0 values. Higher FN_0 values obtained for these mixtures indicate reduced susceptibility to permanent deformation. However, this effect may not be primarily due to the presence of residual binder and its contribution to inter-particle bonding that enhances the integrity of the composite. Instead, it is more likely attributed to the internal friction between the particles within the mineral skeleton. Increasing the BE content in the corresponding mixtures, accompanied by a reduction in air void content, resulted in a decreased resistance to permanent deformation. To further investigate this phenomenon, the values of cumulative strain at failure – $\epsilon(FN_0)$ – were compared for the mixtures, as illustrated in Figure 38.

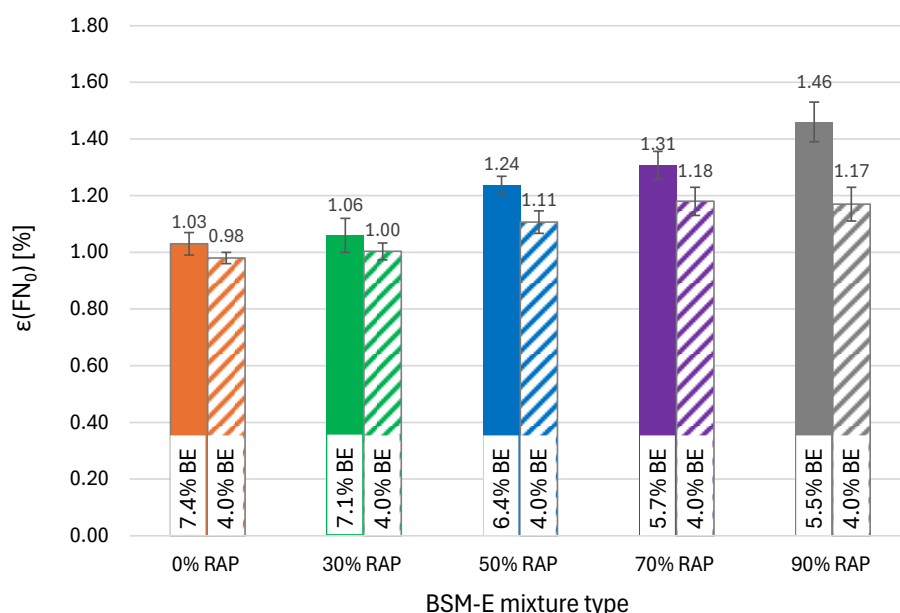


Figure 38 Comparison of $\epsilon(FN_0)$ values for BSM-E mixtures with corresponding BE contents

Analysis of the $\epsilon(FN_0)$ allows for a conclusion that the statistical significance of the differences in values of the accumulated permanent strain at FN between pairs of BSM-Es was primarily determined by the RAP content in the mixture, and thus by the structure of the mineral skeleton, rather than by the change in the BE content alone. Notably, the mixtures with the smallest difference in fresh residual binder content exhibited the largest difference in the accumulated permanent strain level (i.e. 90%R_4%E and 90%R_5.5%E), while no statistically significant changes were observed for mixtures with 0% and 30% RAP content. In these cases, considerable differences in the amount of BE in the mix composition did not result in different level of deformation after reaching the point of shear failure.

Analysis of the FN index, facilitating the simultaneous assessment of FN_0 and $\varepsilon(FN_0)$, allowed for a differentiation of the tested BSM-Es in terms of the accumulated permanent strain increase rate in the cycle range of $[1; FN]$ (Figure 39). The observed differences in the FN index values, similarly to those noted for $\varepsilon(FN_0)$, may indicate a synergistic interaction between RAP and bitumen emulsion affecting the nature of the initial cohesion within the material. In the case of low-RAP BSM-Es, mixtures with the designed BE contents - 0%R_7.4%E and 30%R_7.1%E - presented higher potential for permanent deformation reflected by an increase in the FN index compared to the 4% BE compositions (76% and 30%, respectively). Such variations in the FN index among mixtures with comparable RAP contents – resulting from significant differences in FN values at nearly identical levels of the accumulated permanent strain – suggest a different failure mechanism in the analysed mixtures. Achieving a similar level of deformation with a lower number of loading cycles may be associated with the softening of the residual bitumen present in the localised bituminous bonds under the assumed testing conditions. Mixtures with lower residual binder content and low RAP content may therefore be considered less sensitive to temperature effects, indicating a similarity in behaviour to that of granular materials.

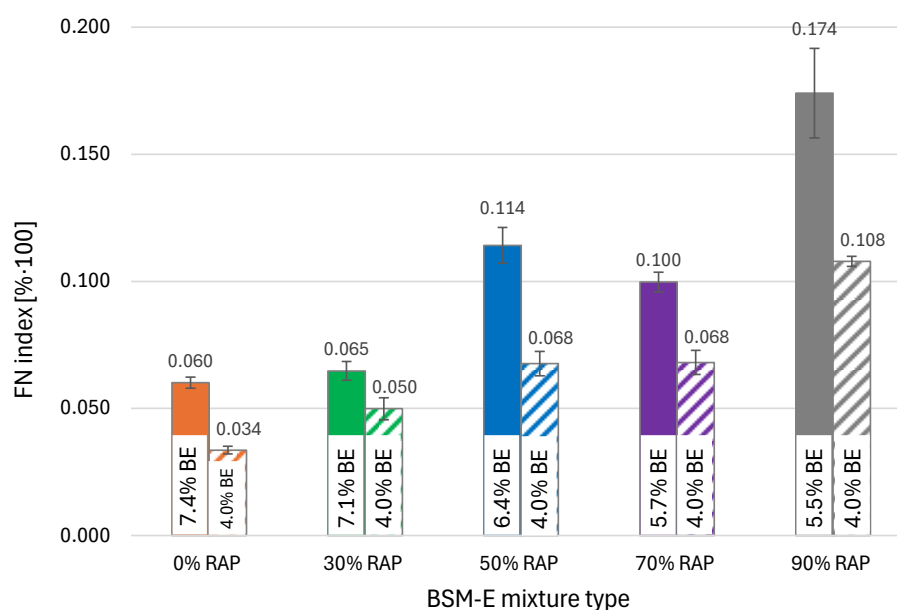


Figure 39 Comparison of FN index values for BSM-E mixtures with corresponding BE contents

In the case of mixtures with medium to high RAP content, the differences observed in FN_0 and $\varepsilon(FN_0)$ values were also reflected in variations in the FN index. Increases in the FN index of 68%, 47%, and 61% were recorded for mixtures with the designed emulsion contents and RAP contents of 50%, 70%, and 90%, respectively. The increase in the BE content in these mixtures thus influenced the material's permanent deformation resistance characteristics.

However, significant changes in the accumulated permanent strain became evident only when the RAP content in the mixture exceeded 50%.

The analysis of the Flow Number performance parameters at 0-day conditioning (FN_0 , $\epsilon(FN_0)$ and $FN\ index$) allowed for a comprehensive description of the initial cohesion and shear failure characteristics of BSM-E mixtures with a specific focus on analysing the internal structure integrity of the mixture, as well as influence of the RAP and BE content on the permanent deformation potential. The observed consistency of the FN_0 and $FN\ index$ parameters in reflecting the differentiation of the cohesive properties of BSM mixtures determined their use for further evaluation of the cohesion evolution in time.

5.2. Cohesion evolution

The first stage of data analysis involved assessing the statistical significance of the differences in the mean values of the FN and FN index parameters, measured after 0, 7, 14, 28, 90, and 120 days of conditioning, by means of ANOVA. Using statistical analysis methods, cohesion development patterns over time were examined for 10 designed BSM-E mixtures. This enabled the subsequent identification of short-term and long-term conditioning periods and allowed for the identification of mixtures exhibiting cohesion development over time, along with the quantification of changes in FN and FN index values. It was assumed that the expected increase in the BSM-E's cohesion would be associated with a statistically significant increase in FN values and simultaneously decrease in FN index over the conditioning period.

5.2.1. Preliminary assessment of cohesion changes

Table 22 summarizes the results of the ANOVA results assessing the statistical significance of differences in mean FN and FN index values across the 120-day conditioning period. The analysis considered six result groups corresponding to the following time points: 0, 7, 14, 28, 90, and 120 days. The detailed results of the Flow Number tests are provided in Appendix 2.

Table 22 Statistical significance of differences between Flow Number means for the 120-day conditioning period

BSM-E mixture \ p-value	FN_{0-120} diff.	$FN\ index_{0-120}$ diff.	Cohesion change in time
0%R 4%E	0.412	0.685	not present
30%R 4%E	0.955	0.978	not present
50%R 4%E	0.223	0.603	not present
70%R 4%E	0.003	0.003	present
90%R 4%E	0.000	0.000	present
0%R 7.4%E	0.582	0.923	not present
30%R 7.1%E	0.381	0.498	not present
50%R 6.4%E	0.001	0.002	present
70%R 5.7%E	0.000	0.000	present
90%R 5.5%E	0.000	0.000	present

Based on the calculated *p-values*, it can be stated that half of the BSM-E mixtures did not exhibit statistically significant changes in the permanent deformation performance, among which mixtures with low to intermediate RAP content were distinguished: 0%R_4%E, 30%R_4%E, 50%R_4%E, 0%R_7.4%E, and 30%R_7.1%E. Given the positive verification of changes in the cohesive performance of the remaining mixtures, an analysis was conducted on the patterns underlying the changes in their permanent deformation performance throughout the 120-day conditioning period. ANOVA, combined with Fisher's LSD multiple comparison procedure, was employed to identify statistically homogeneous groups based on the FN values, regarded as the primary standardised parameter for evaluating the mixture properties. The results of the statistical analysis are presented in Table 23.

Table 23 Homogeneous groups of FN means for the 120-day conditioning period
(BSM-Es with confirmed cohesion changes)

Time point (days)	Homogeneous groups of FN values														
	70%R_4%E			90%R_4%E			50%R_6.4%E			70%R_5.7%E			90%R_5.5%E		
0	X			X			X			X			X		
7	X			X			X			X			X		
14	X			X			X			X			X		
28	X				X		X				X			X	
90			X			X			X			X			X
120			X			X			X			X			X

Based on the results of the statistical analysis of the FN values (Table 23), it can be stated that, for three out of five analysed BSM-Es (90%R_4%E, 70%R_5.7%E, 90%R_5.5%E), three homogeneous groups of means were identified: (1) values obtained from day 0 to day 14 of conditioning (FN₀₋₁₄); (2) values obtained on day 28 (FN₂₈); and (3) values obtained from day 90 to day 120 of conditioning (FN₉₀₋₁₂₀). For the 70%R_4%E and 50%R_6.4%E mixtures, two homogeneous groups were identified, corresponding to the periods from day 0 to day 28 and from day 90 to day 120 of conditioning. This observation indicates that the temporal changes in FN values for these mixtures were less pronounced compared to the three previously discussed ones. On this basis, the 70%R_4%E and 50%R_6.4%E mixtures can be classified as borderline cases between mixtures exhibiting cohesion development over time and those showing no such development.

The overall analysis of the results shows that the change in cohesion can be analysed by dividing the conditioning period into short-term and long-term phases with the division established based on two variability periods corresponding to the FN parameter, in which the cohesive properties of the material can be considered unchanged. A short-term conditioning

period was identified, covering the range from day 0 to day 28 with a sub-period ranging from day 0 to day 14, as well as a long-term conditioning period spanning from day 90 to day 120.

5.2.2. Short-term evaluation

According to the conclusions presented in section 5.2.1, no significant changes in FN were observed for any of the analysed BSM-Es during the day 0 and day 14 of conditioning. Table 24 presents the ANOVA test results assessing differences in the mean values of FN and FN index at three time points: 0, 7, and 14 days, for five mixtures with confirmed changes in cohesion. Figure 40 and Figure 41 illustrate the mean FN and FN index values over the short-term conditioning period for the analysed mixtures, accompanied by the corresponding data dispersion.

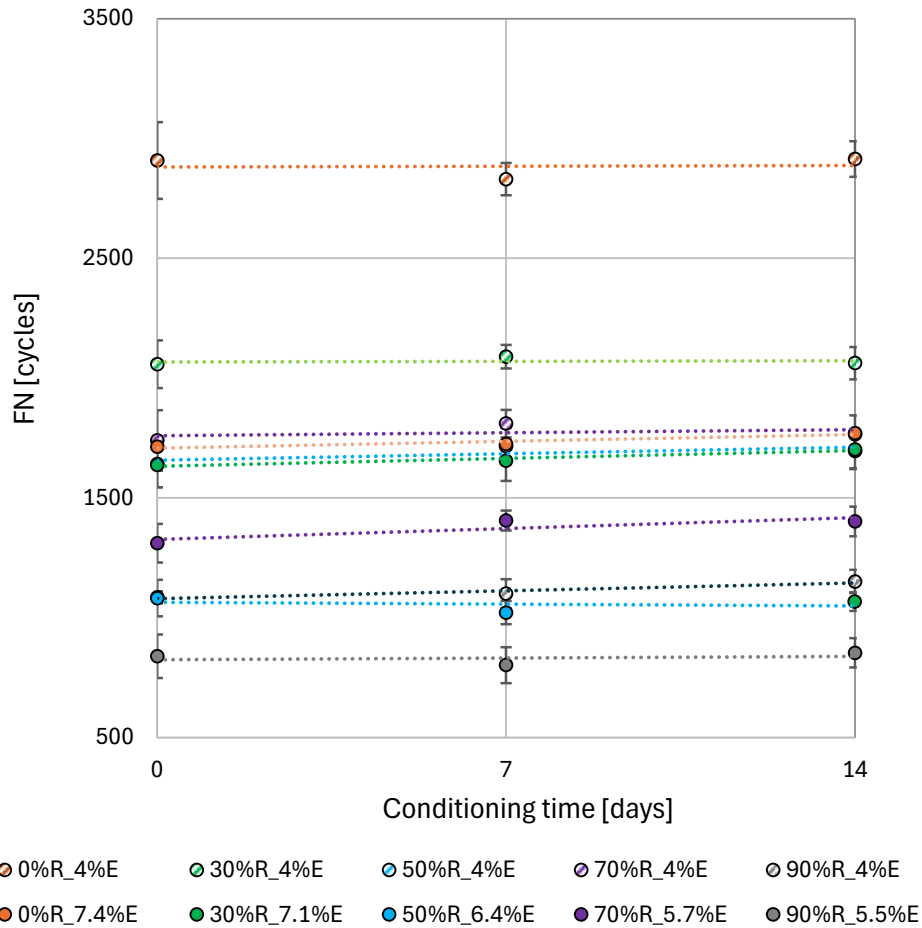


Figure 40 Changes in FN values during the 0–14 day conditioning period (all BSM-E mixtures)

Table 24 ANOVA test results for Flow Number means in the 0-14 day conditioning period
(BSM-Es with confirmed cohesion changes)

BSM-E mixture	p-value	FN ₀₋₁₄ diff.	FN index ₀₋₁₄ diff.
70%R 4%E		0.642	0.449
90%R 4%E		0.274	0.104
50%R 6.4%E		0.406	0.581
70%R 5.7%E		0.155	0.161
90%R 5.5%E		0.678	0.892

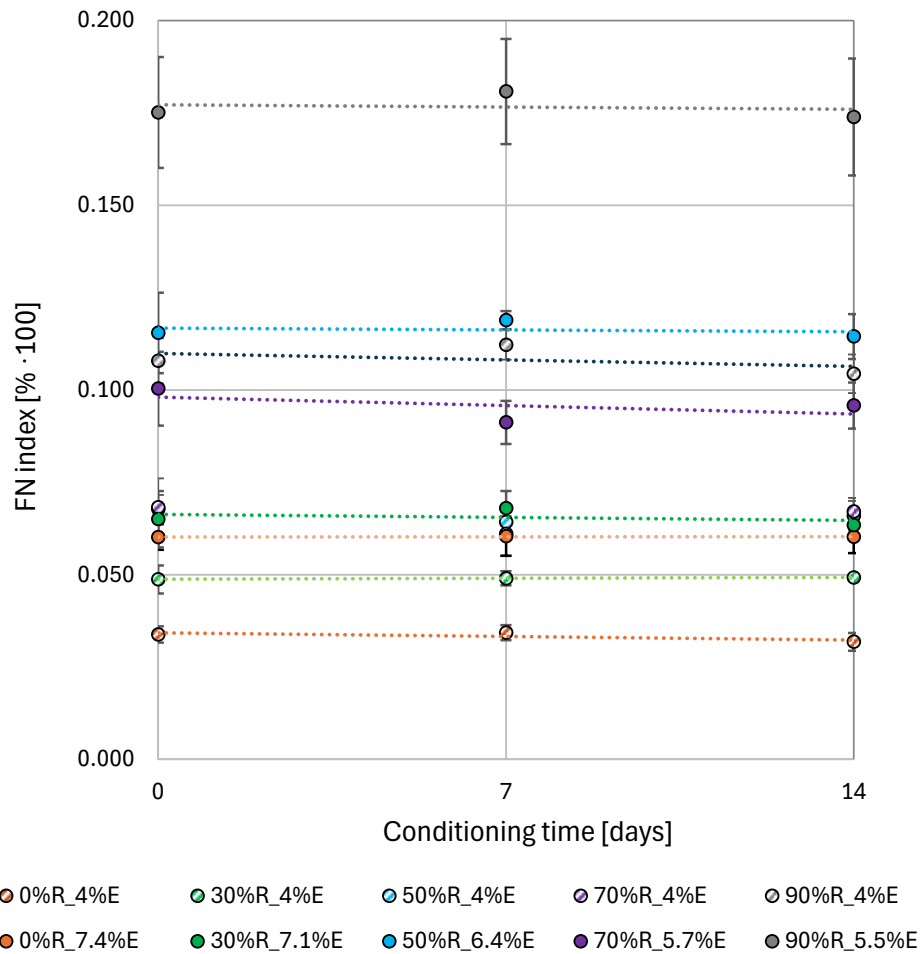


Figure 41 Changes in FN index values during the 0–14 day conditioning period (all BSM-E mixtures)

To investigate the progression of changes in FN and FN index parameters over time, the analysis performed for days 0 to 14 was extended to include data from day 28 of conditioning. Figure 42 illustrates the average FN values for BSM-Es with no cohesion development. The highlighted coloured regions represent the overall dispersion of results for these mixtures, based on specimens tested at four consecutive time points, and indicate the absence of significant changes in the mixtures' permanent deformation performance.

Based on the data presented in section 5.2.1 (Table 23), it can be stated that the day 28 of conditioning can be identified as a distinguishing point differentiating the course of cohesion

development over time in the case of three mixtures: 90%R_4%E, 70%R_5.7%E, and 90%R_5.5%E. This is confirmed by the ANOVA p-values indicating the statistical significance of differences in FN and FN index means between the 0–14 day conditioning period and day 28, as presented in Table 25. However, it must be stated that the observed changes in FN index values were strictly connected to changes in the number of cycles to failure, since the level of accumulated permanent strain at FN – $\varepsilon(\text{FN})$ – remained unchanged in the course of the 28-day conditioning for all tested BSM-Es.

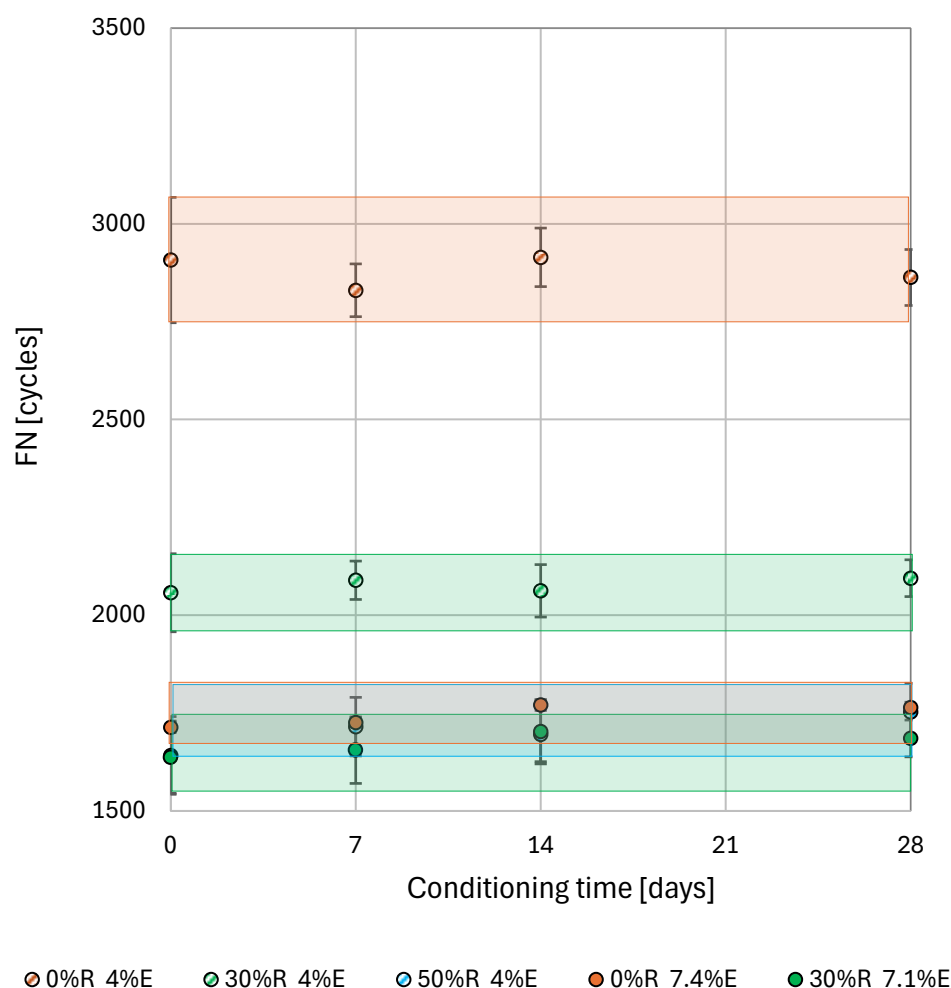


Figure 42 Changes in FN values during the 0–28 day conditioning period (BSM-Es with no cohesion changes)

Table 25 ANOVA test results for FN and FN index means in the 28-day conditioning period
(BSM-Es with confirmed cohesion changes)

BSM-E mixture \ p-value	$\text{FN}_{0-14} - \text{FN}_{28}$ diff.	$\text{FN index}_{0-14} - \text{FN index}_{28}$ diff.	$\varepsilon(\text{FN})_{0-14} - \varepsilon(\text{FN})_{28}$ diff.
70%R_4%E	0.119	0.561	0.285
90%R_4%E	0.001	0.004	0.806
50%R_6.4%E	0.231	0.191	0.607
70%R_5.7%E	0.002	0.054	0.399
90%R_5.5%E	0.002	0.001	0.149

Figure 43 depicts the changes in the mean FN values for the mixtures up to day 14 of conditioning (with the average level marked by a constant line), compared to the mean value recorded on day 28. For all analysed mixtures, an increase in FN values was observed on the 28th day of conditioning. However, for mixtures 70%R_4%E and 50%R_6.4%E, the observed increase in the mean FN value was not statistically significant, which justifies further analysis of changes in cohesive parameters over a longer conditioning period.

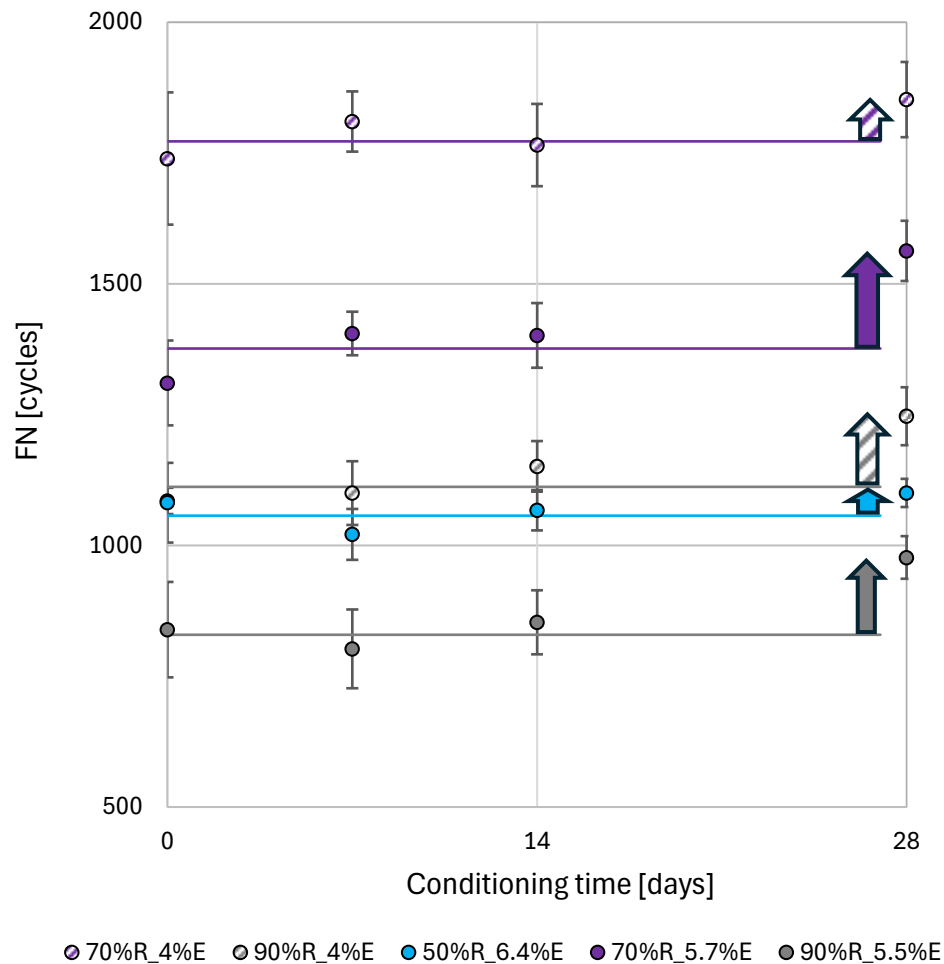


Figure 43 Changes in FN in the 0–28 day conditioning period (BSM-Es with cohesion changes)

Changes in FN index in the 0–28 day conditioning period for the same group of mixtures are presented in Figure 44. For all analysed mixtures, a decrease in the FN index value was observed, indicating a reduction in the rate of permanent strain accumulation within the [1, FN] cycle range. However, according to the data presented in Table 25, at this stage, these changes were statistically significant only for the two mixtures with the highest RAP content (90%R_4%E and 90%_5.5%E).

Figure 45 shows the percentage parameter changes between the short-term conditioning sub-period (0-14 days) and the 28th day of conditioning. The most pronounced changes were

recorded for the 90%R_5.5%E mixture, which exhibited a 17.8% increase in FN and a 17.8% decrease in the FN index values. A comparable level of cohesion change over time was observed for the 90%R_4%E and 70%R_5.7%E mixtures, for which statistically significant increases in FN values exceeding 12% were noted.

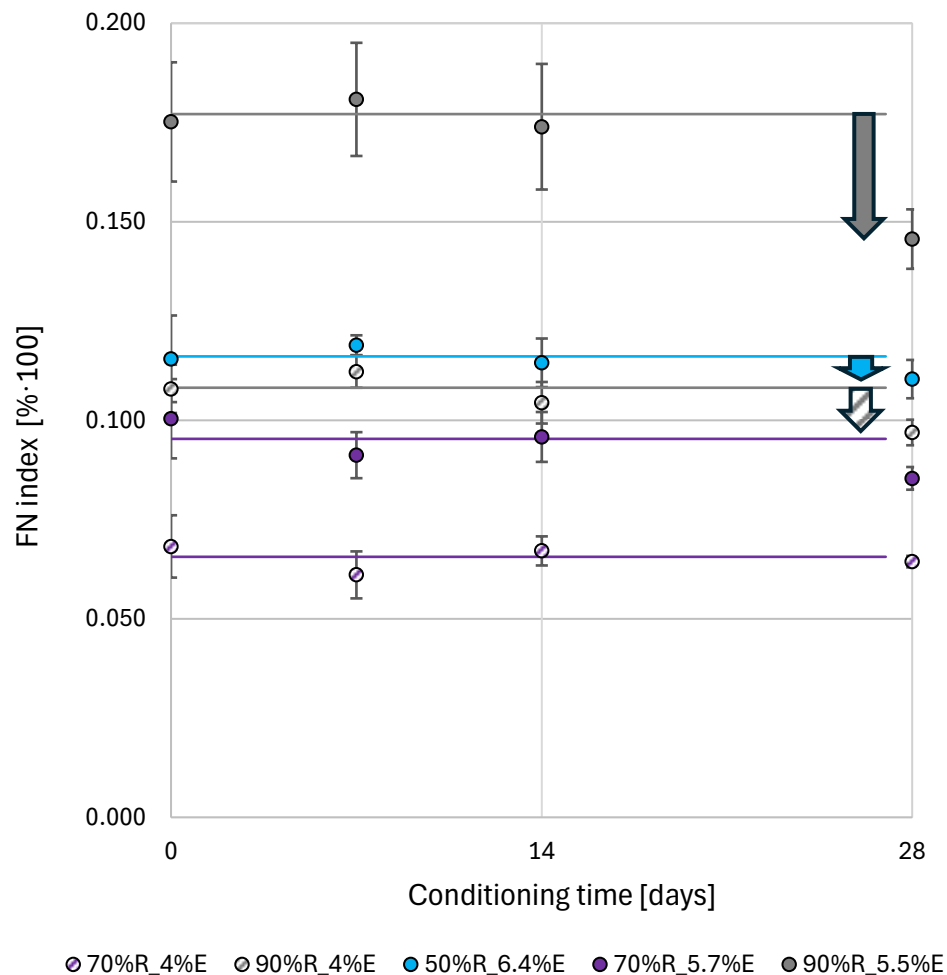


Figure 44 Changes in FN index in the 0–28 day conditioning period (BSM-Es with cohesion changes)

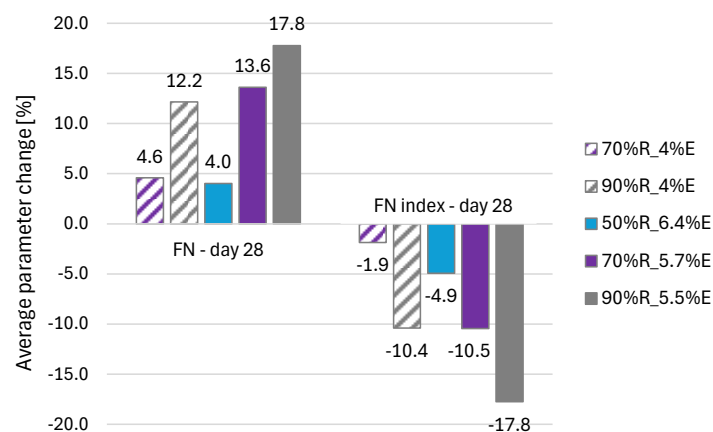


Figure 45 Percentage changes in FN and FN index between 0–14 and 28 days of conditioning.

The increase in FN values indicates a higher number of cycles required to reach shear failure, which, in the context of cohesion development analysis, is a favourable phenomenon. The accompanying decrease in the FN index value reflects a reduction in the increase rate of accumulated permanent strain. During the first month of conditioning, both parameters exhibited statistically significant changes only in the case of mixtures with the highest RAP content. However, the identified trends and percentage changes in FN and FN index values, supplemented by the results of the statistical analysis (Table 23, Table 25), indicate the occurrence of noticeable changes in the permanent deformation performance of the tested mixtures during the conditioning period.

5.2.3. Long-term evaluation

The long-term evaluation of the cohesion development involved an analysis of changes in FN and FN index values, taking into account the results obtained during the conditioning period beyond day 28.

As shown in Figure 46, which presents changes in FN values throughout the conditioning period, an increase in FN values was observed on days 90 and 120 for all analysed mixtures, interpreted as an indication of further cohesion development in the material. The ranges of the short- and long-term conditioning periods are also marked in the figure, in accordance with the previously conducted analyses. The results of the statistical analysis presented in section 5.2.1 (Table 23) indicate that the Flow Number test results from days 90 and 120 can be treated as a single group for the purpose of mean value analysis, due to the absence of statistically significant differences. Based on the ANOVA test results presented in Table 26, in all analysed cases the FN and FN index values obtained on day 28 were significantly different from those recorded at the later stages of testing. In contrast, no significant changes in $\epsilon(\text{FN})$ values were noted, except for the 70%R_4%E mixture, showing that the values of accumulated permanent strain at FN did not change during the long-term conditioning period.

Table 26 ANOVA test results for FN and FN index means in the 120-day conditioning period
(BSM-Es with confirmed cohesion changes)

BSM-E mixture \ p-value	FN₂₈ – FN₉₀₋₁₂₀ diff.	FN index₂₈ – FN index₉₀₋₁₂₀ diff.	$\epsilon(\text{FN})_{28}$ – $\epsilon(\text{FN})_{90-120}$ diff.
70%R_4%E	0.005	0.001	0.046
90%R_4%E	0.003	0.001	0.360
50%R_6.4%E	0.005	0.004	0.053
70%R_5.7%E	0.001	0.001	0.958
90%R_5.5%E	0.001	0.001	0.360

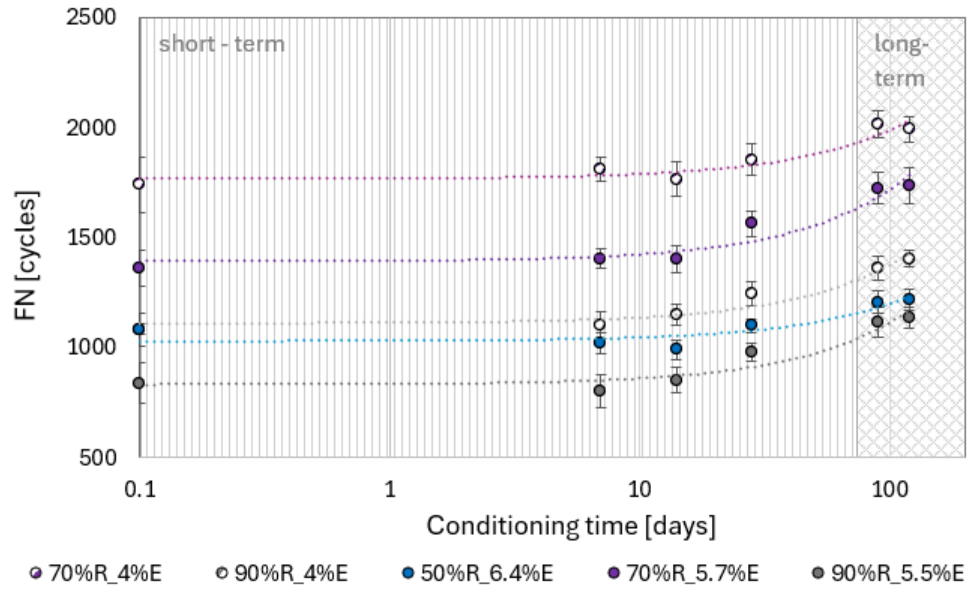
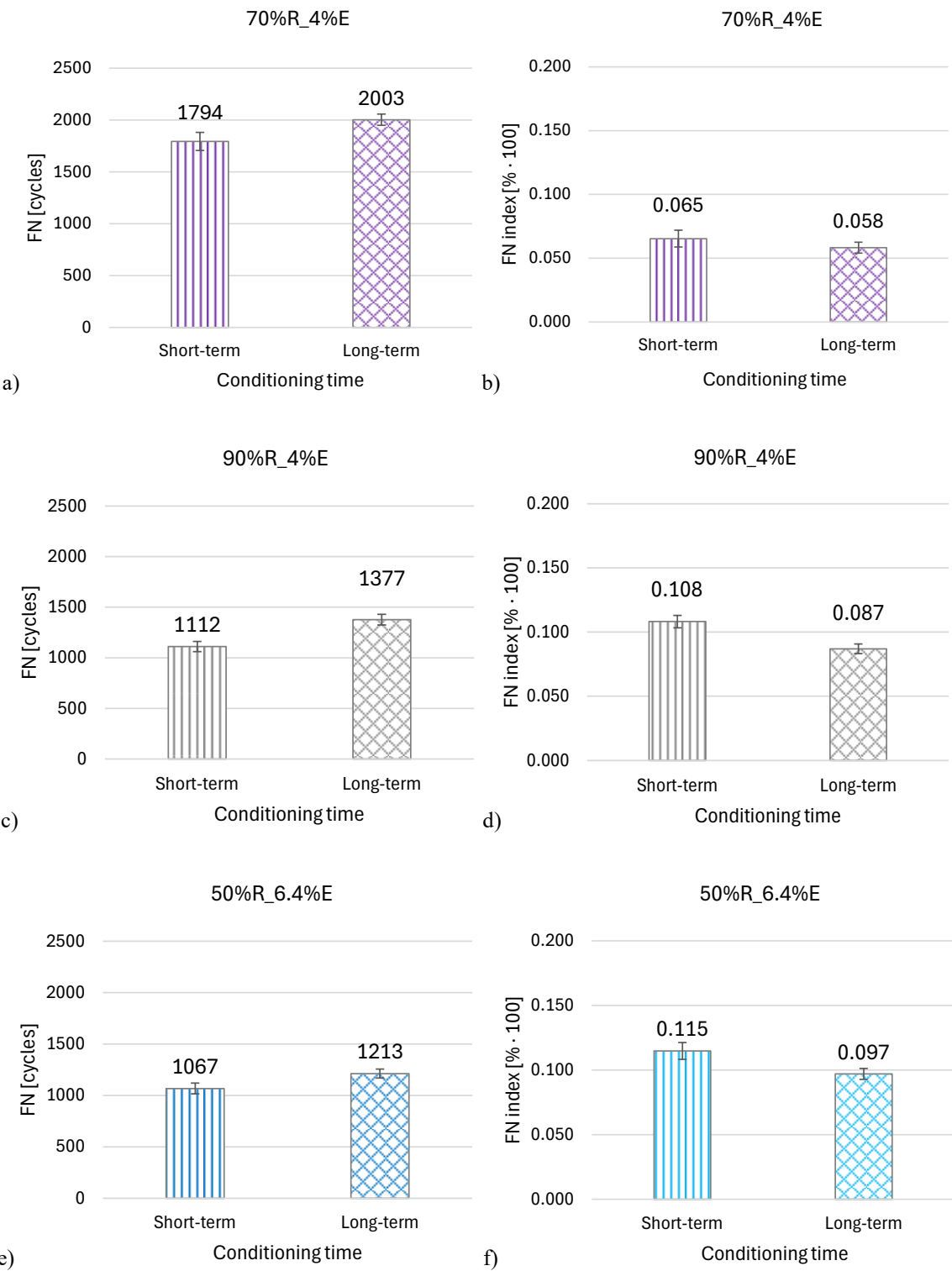


Figure 46 FN changes during the 120-day conditioning period

The observed homogeneity between the results from days 90 and 120 may indicate a stabilization of the cohesive properties of the tested mixtures over the longer period after curing. The increase in FN values, noticeable for selected mixtures as early as day 28, may be attributed to internal structural changes in the specimens induced by conditioning factors, such as lateral confinement and vertical loading simulating the pressure exerted by overlying pavement layers.

To assess the dynamics of changes in FN and FN index values throughout the entire conditioning period, mean values of these parameters were analysed with a distinction between the short- and long-term stages. The values for the short-term stage were calculated based on the homogeneity of result groups within the 0–28 day conditioning period, according to Table 23. Analysing the data presented in Figure 47 a) – j), it can be concluded, that the 90%R_5.5%E mixture was characterized with the most significant changes in cohesion during the conditioning period. In this case, a 35.6% increase in the average FN value was observed and a 29.1% decrease in the average FN index value was observed. The mixture exhibiting the second highest increase in FN during conditioning was 70%R_5.7%E, which demonstrated a 33.7% increase during long-term conditioning, accompanied by a 23.8% decrease in the FN index. In contrast, results obtained for the corresponding mixture with 4%BE (70%R_4%E) indicate the lowest tendency for FN and FN index changes among the analysed mixtures (increase by 11.6% and decrease by 10.9%, respectively). The 90%R_4%E mixture was characterized with an increase in the average FN value by 23.8% compared to short-term conditioning time, while the FN index value decreased by 19.6%. The 50%R_6.4%E mixture

was characterized by a second lowest level of FN index reduction (15.5%) and an increase in the Flow Number value equal to 13.6%.



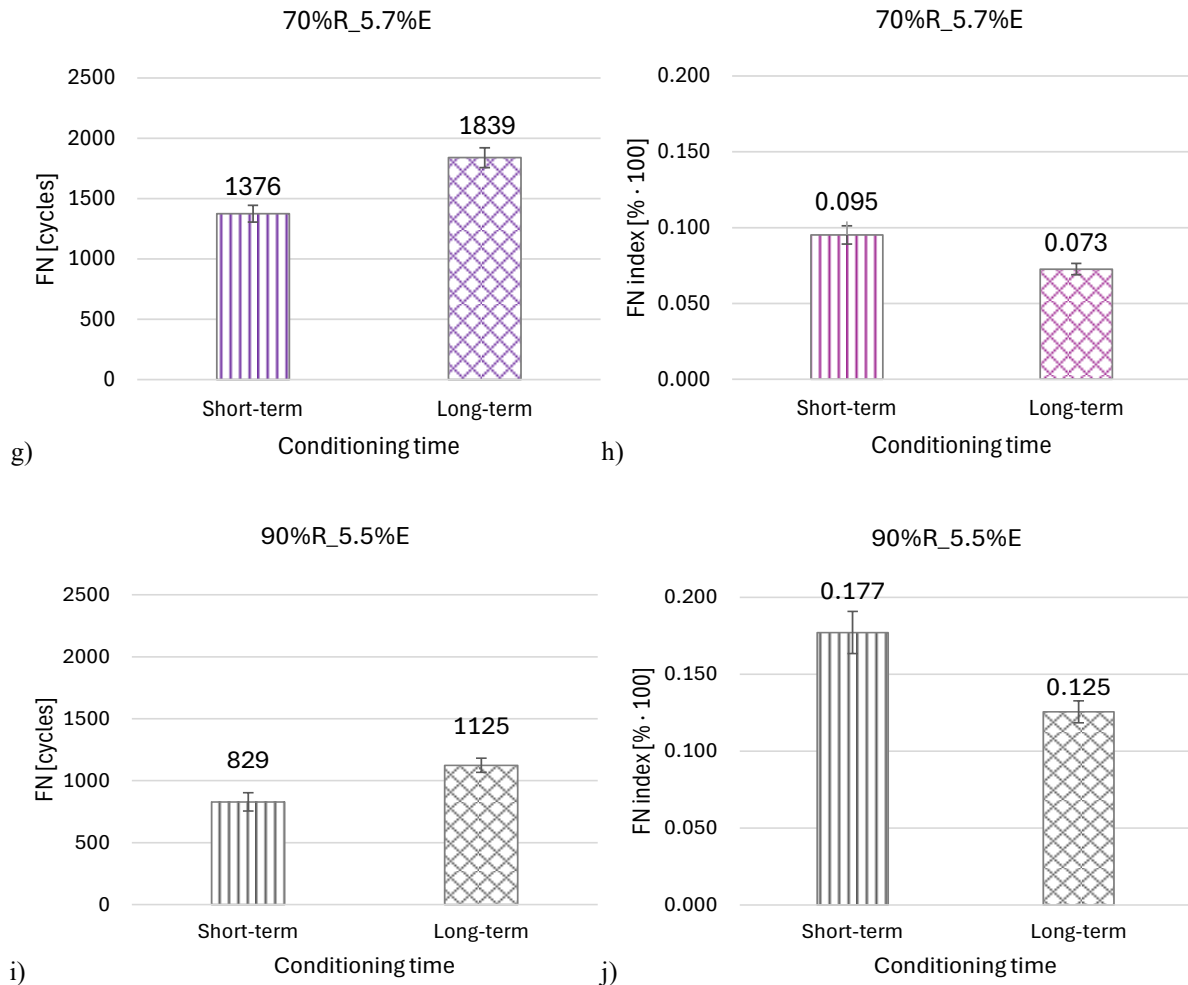


Figure 47 Changes in FN and FN index for BSM-Es with cohesion evolution: a),b) 70%R_4%E; c),d) 90%R_4%E; e),f) 50%R_6.4%E; g),h) 70%R_5.7%E; i),j) 90%R_5.5%E.

5.3. Discussion on the short- and long-term cohesive performance in terms of viscoelasticity

The objective of the experimental program was to examine how the selection of BSM-E components (RAP, bitumen emulsion) influences the cohesive properties of the material, and whether the cohesion understood as composite integrity can change over time given specific external conditions and conditioning procedures. The applicability of the Flow Number test to quantify the changes in BSM-Es' performance at the initial testing stage and over time was positively verified. The obtained results serve to identify mixtures that potentially exhibit viscoelastic behaviour. The analysis of the results presented and discussed in sections 5.1 and 5.2 aims to identify patterns of cohesion development and to address the research question of whether, and to what extent, cohesion is related to the viscoelastic properties of the material.

Based on the analysis of the initial cohesion test results, it can be stated that the performance of BSM-Es is material-related. A clear relationship was identified between the

RAP content in BSM-E mixtures and their resistance to permanent deformation immediately after the curing process (0-day conditioning). In general, with a constant bitumen emulsion content, an increase in RAP content led to a noticeable decrease in the FN value, while simultaneously increasing the FN index value – the latter incorporating information on the accumulated permanent strain at FN – which implies an increased susceptibility of the mixtures to permanent deformation. These trends were also observed in the group of mixtures selected in the mix design process, characterized by simultaneous variation in both RAP and bitumen emulsion content. A comparison of corresponding mixtures with constant RAP content and varying bitumen emulsion content indicated a reduction in the number of cycles to failure and an increase in the FN index value with an increase in the BE amount across the analysed variants.

The influence of the material-related factors was evident not only at the initial stage of the permanent deformation resistance testing, but was also reflected in the patterns of cohesion development over time in the tested BSM-E mixtures. As concluded in section 5.2, cohesion development is possible, but it does not follow a linear trend – within the analysed conditioning period, distinct short-term and long-term stages can be identified, each characterised by different levels of the evaluated parameters. Figure 48 and Figure 49 present the ranking of all tested mixtures based on the values of the FN and FN index, calculated for both short- and long-term conditioning periods. Mixtures exhibiting statistically significant changes in the performance parameters have been marked in red boxes.

The analysis of the character and dynamics of changes in the cohesion quantified based on changes in FN and FN index parameters, allowed for identification of distinct performance patterns for specific groups of BSM-Es. Firstly, mixtures with zero or low RAP content and 4% BE content, which did not exhibit statistically significant changes in cohesion over time, were characterized by the highest FN and the lowest accumulated strain levels at FN, both in the initial stage of testing and after the long-term conditioning period. The contrasting group consisted of mixtures with medium to high RAP contents (50%–90%) and medium emulsion contents (4%–6.4%), which were particularly predisposed to exhibit changes in permanent deformation susceptibility. For these mixtures, a significant increase in FN values and decrease in FN index values were observed, indicating evolution in the cohesion properties, but still both of their performance parameters were ranked in the same order, presenting the lowest resistance to permanent deformation among the analysed mixtures. Only the changes in FN and FN index values observed for mixtures containing 70% RAP introduced differences in the overall ranking of the mixtures. A significant increase in the FN value for the

70%R_5.7%E mixture – placing it, after long-term conditioning, at a level comparable to the 30%R_4%E and 70%R_4%E mixtures – combined with a concurrent FN index change that still classifies it among mixtures with higher susceptibility to permanent deformation, may indicate that this mixture exhibits behaviour characteristic of viscoelastic materials. Changes in parameter values for the 70%_4%E mixture were less pronounced – however, a notable shift in the ranking was observed for the FN index, indicating a reduction in the rate of accumulated permanent deformation growth over time following long-term conditioning. This positioned the mixture behind mixtures 0%R_7.4%E, 30%R_7.1%E, and 50%R_4%E, which did not exhibit any changes in cohesive properties (Figure 49).

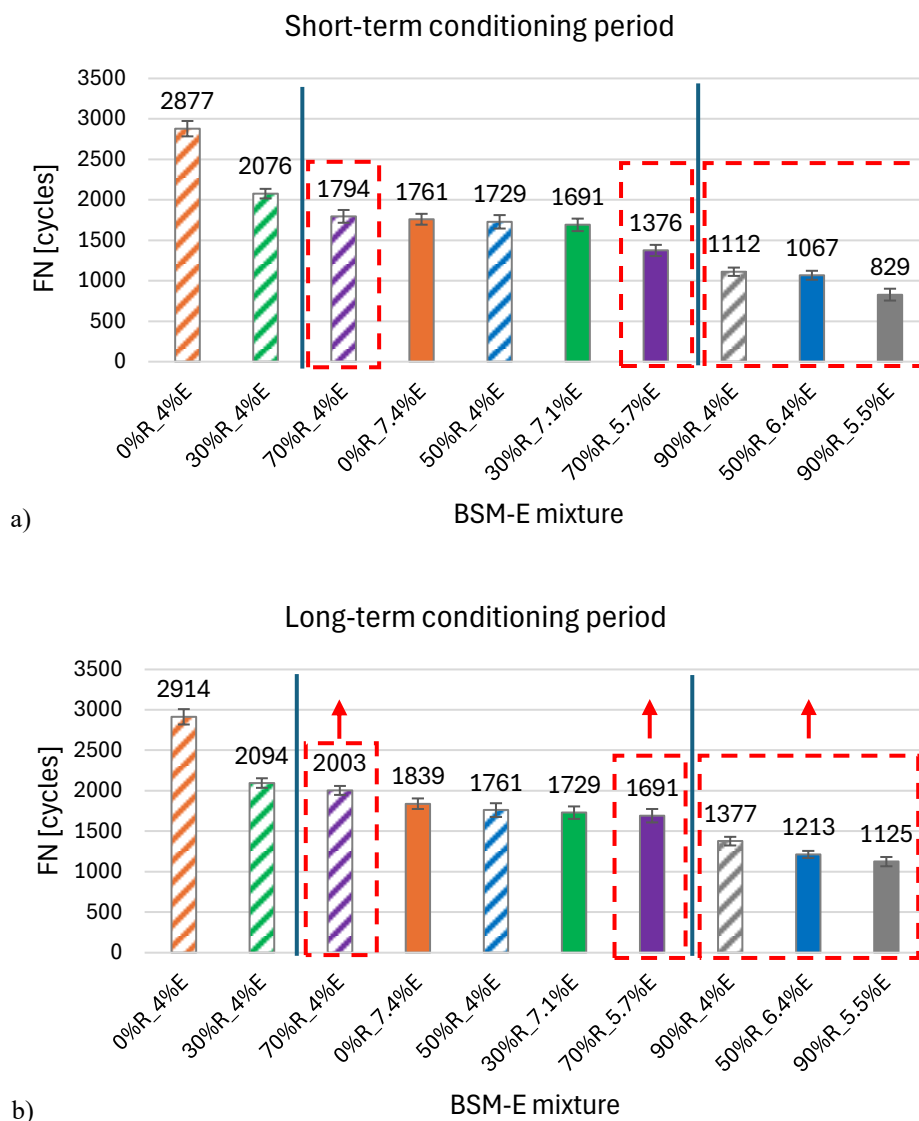


Figure 48 Comparison of FN for BSM-E mixtures with respect to the: a) short-term and b) long-term conditioning periods.

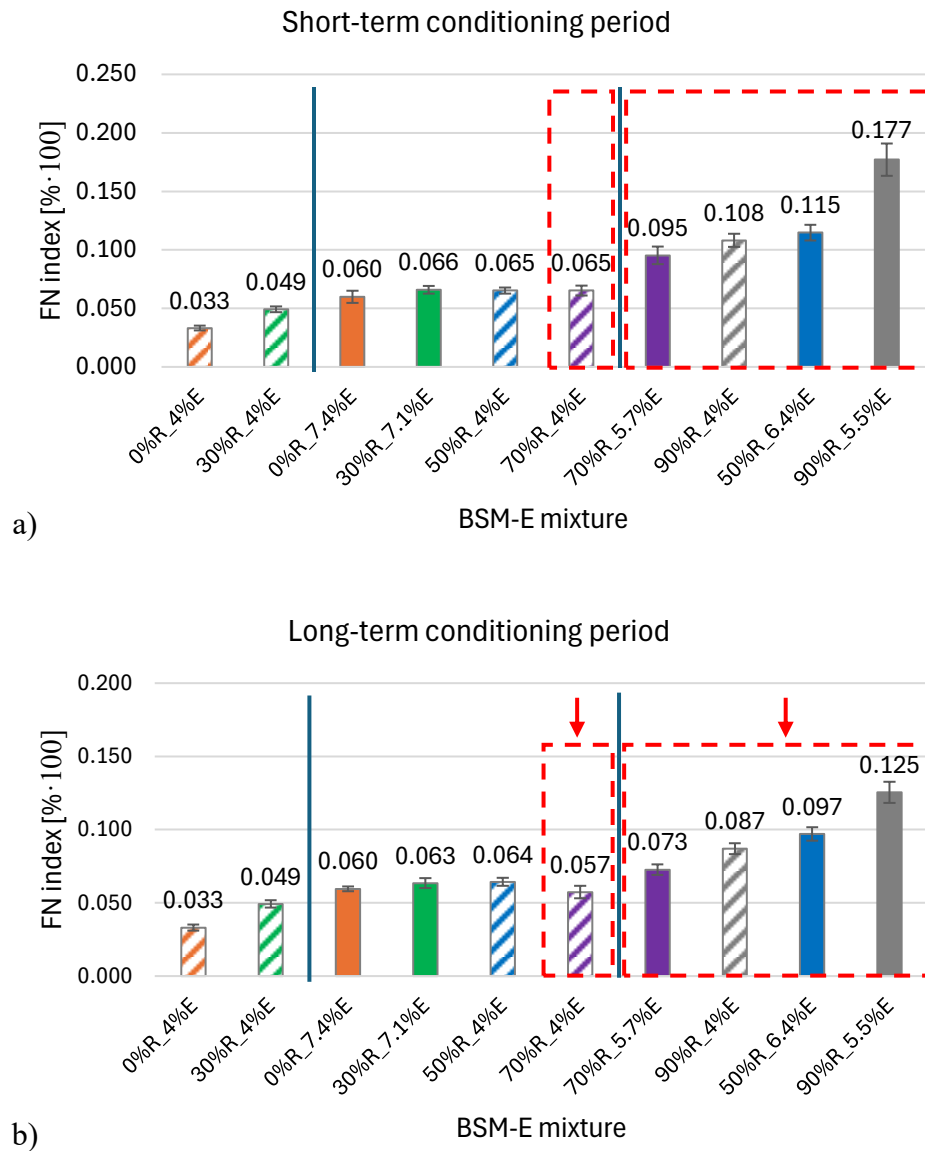


Figure 49 Comparison of FN index for BSM-E mixtures with respect to the: a) short-term and b) long-term conditioning periods.

To address the research question concerning whether – and to what extent – the cohesion of BSM-Es is related to the material’s viscoelastic properties, a classification of the investigated mixtures was undertaken considering their expected mechanical performance in pavement structure. Criteria were also established for selecting mixtures for dynamic modulus testing to evaluate their viscoelastic behaviour. To this end, the results of Flow Number tests (FN, $\varepsilon(\text{FN})$ and FN index) conducted at the initial stage of conditioning (0-day) were gathered and assigned to mixtures exhibiting specific patterns in terms of both initial cohesion and its development over time (Figure 50). These analyses considered both material-specific factors and external influences that affect the development of the material’s internal structure. Three groups of mixtures were distinguished based on the recorded test parameter values and ranked according to the FN index values.

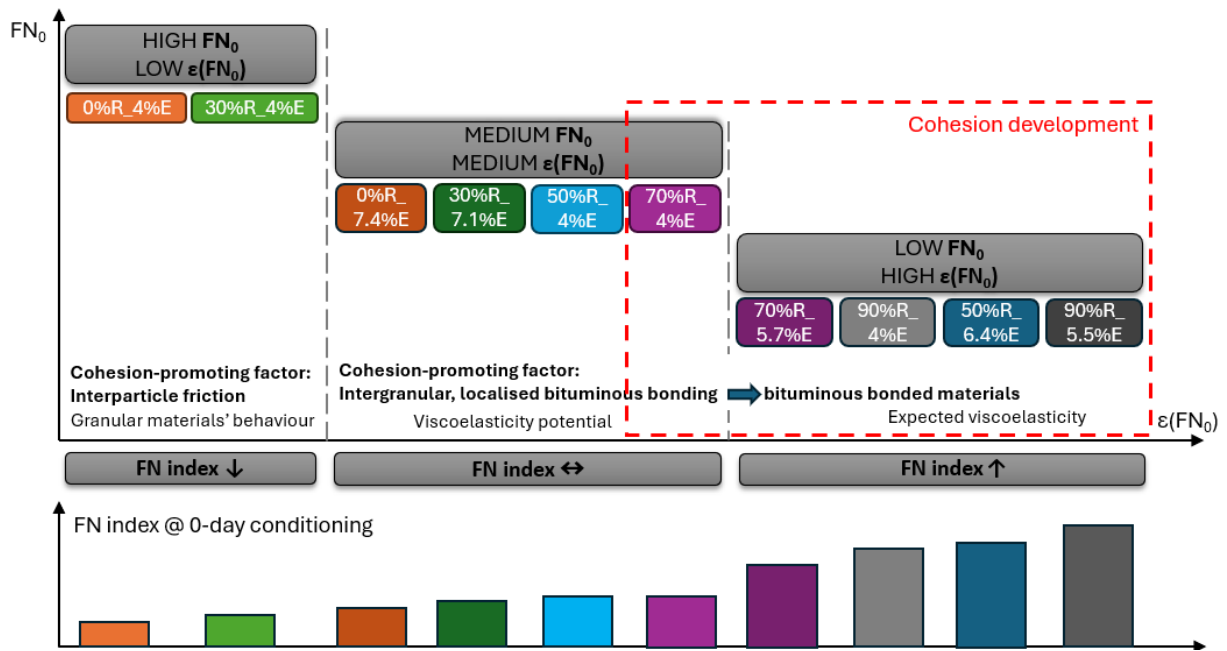


Figure 50 BSM-Es' cohesive performance based on Flow Number testing parameters at 0-day conditioning

The first identified group consisted of mixtures characterised by a high number of cycles to failure combined with low accumulated strain levels at shear failure, as demonstrated by mixtures containing zero or low amounts of RAP and a low BE content (4%) – 0%R_4%E and 30%R_4%E. For these mixtures, the lowest values of the FN index at 0-day conditioning were recorded. By comparing the Flow Number test parameter values obtained for these mixtures with those obtained for the reference AC 22 base course mixture (as discussed in section 5.1.2), it can be inferred that the primary mechanism responsible for the resistance of these mixtures to permanent deformation is interparticle friction. This highlights the dominant role of the mineral skeleton in load transfer, while the influence of bituminous bonds with aggregate particles appears to play a lesser role in the material's performance. These observations lead to the conclusion that such mixtures are likely to exhibit behaviour characteristic of granular materials, and their mechanical properties are unlikely to be significantly affected by factors influencing the behaviour of viscoelastic materials, such as temperature or loading time (frequency).

The next group consisted of four mixtures – 70%R_5.7%E, 90%R_4%E, 50%R_6.4%E, and 90%R_5.5%E – exhibiting properties that were distinctly different from those of the first group, namely high $\epsilon(FN)$ values and low numbers of cycles to failure (FN), which resulted in significantly higher FN index values compared to the other analysed mixtures. This indicates an accelerated rate of permanent deformation development relative to the remaining BSM-Es. However, for all mixtures in this group, an increase in cohesion over time was observed,

evidenced by an extended test duration to shear failure (increased FN) without a corresponding increase in accumulated permanent strain. This suggests an improvement in the internal structural integrity of the mixtures over time and an associated enhancement in resistance to permanent deformation. These BSM-Es were characterized by medium to high RAP content (50%–90%) and medium BE content (4%–6.4%). Given the high total binder content in these mixtures, and a relatively high share of fresh residual binder, it can be assumed that for this BSM-Es group, the role of interparticle bituminous bonding in load transfer is greater than in the first group, showing the potential to obtain continuous bonds within the structure. This observation, complemented by the recorded changes in the mechanical response of the material over time, may therefore indicate the capacity of these mixtures to exhibit viscoelastic behaviour within the pavement structure.

The last group comprised mixtures exhibiting intermediate properties between those of groups 1 and 2, as evidenced by the intermediate values of all measured parameters in the Flow Number test. This group included mixtures with low RAP content and high BE content, as well as mixtures with medium RAP content (50%, 70%) and low BE content (4%). Based on the obtained test results, the 70%R_4%E mixture was also classified as a BSM-E material, which demonstrated cohesion development over time. The Flow Number test results obtained for these mixtures may indicate a potential increase in the role of interparticle localised bituminous bonding in the material compared to the mixtures from group 1. The similar values of the FN index suggest a comparable response of these mixtures to loading in terms of the rate of the accumulated permanent strain increase. It was assumed that, in the case of these mixtures, the factors determining their potential to exhibit viscoelastic behaviour may include either a high BE content (in the case of 0%R_7.4%E and 30%R_7.1%E), or a high RAP content (for mixtures 50%R_4%E and 70%R_4%E).

Based on the data presented in Figure 50, it can be also stated that particular attention should be given to the interpretation of the performance parameters originally used for characterizing HMA and WMA in Flow Number testing, especially in terms of the FN index. Since it was observed that BSM-Es with the highest initial FN index values, exhibited cohesion development over time measured by an increase in resistance to permanent deformation over the conditioning period, it is vital to consider whether the FN index can be simply interpreted as a parameter enabling comparison and classification of the mixtures' permanent deformation resistance based on the rate of accumulated permanent strain increase within the [1, FN] cycle range. It should be noted that in CRMs, the FN index may not only provide information on the rate of the permanent strain accumulation, but may also serve as an indicator of changes in

material characteristics, namely the transition of mechanical properties between those typical of granular materials and those resembling viscoelastic materials. Moreover, when analysing the changes in material cohesion, the $\varepsilon(\text{FN})$ values generally remained constant throughout the conditioning period for all mixtures, making the changes in the FN index dependent solely on the variations in the FN value over time.

Based on the obtained mean FN index values and the observed data scatter, ranges of this parameter were proposed to characterize three groups of mixtures with an expectedly different mechanical performance. For the calculations, mean values from the individual time points of the short-term conditioning period were adopted, for which no statistically significant differences were recorded with respect to the 0-day of conditioning. This approach was applied in order to increase the statistical sample size, enhance the reliability of the predictions, and remain consistent with the initial FN index values, which may serve as the first verification parameter of the mixture performance.

The following FN index ranges were distinguished:

- Range I: FN index ≤ 0.050 [% · 100] – expected granular material behaviour, low chances of cohesion evolution,
- Range II: FN index (0.065 ± 0.005) [% · 100] – mixtures show potential for viscoelastic behaviour driven by different material-related factors (such as high BE content or high RAP content). However, this value should be considered as a threshold with respect to predicting cohesion development in the material.
- Range III: FN index ≥ 0.090 [% · 100] – mixtures are expected to demonstrate viscoelastic behaviour and cohesion development over time.

On the basis of the results obtained in this study, no mixtures were identified with FN index values within the intermediate ranges of (0.055 ± 0.005) [% · 100] and (0.080 ± 0.010) [% · 100]. Nevertheless, considering the upward trend of the FN index values accompanied by the observed changes in the cohesive properties of the mixtures, it may be assumed that in the first case (0.055 ± 0.005) [% · 100], the viscoelastic behaviour of the material and its potential for time-dependent changes cannot yet be fully ascertained. In the second case (0.080 ± 0.010) [% · 100], by analysing the properties of the mixtures falling within Range II and Range III, it can be assumed that mixtures with such FN index values are likely to have an increased potential for both cohesion development and viscoelastic behaviour.

It can therefore be concluded that the FN index ranges defined in this manner may serve as a preliminary indicator of whether a mixture exhibits predominantly granular or viscoelastic

performance, and may also be regarded as a predictor of cohesion development within the material.

By analysing the influence of material-related factors and external conditions on the permanent deformation response of BSM-Es and their cohesion development potential in relation to the prediction of viscoelastic behaviour, the selection of BSM-E mixtures for further analysis was carried out using a decision tree method based on the following three hierarchical criteria:

1. RAP content: high (i.e., $\geq 70\%$),
2. Cohesion development in time: existing,
3. BE content: high (i.e., $\geq 5.5\%$).

Figure 51 depicts the decision process and presents the seven BSM-E mixtures selected for mechanical testing. Three out of ten analysed mixtures with the lowest BE content and low to intermediate RAP content (0%R_4%E, 30%R_4%E, 50%R_4%E) did not fulfil the identified criteria and were excluded from the viscoelasticity testing.

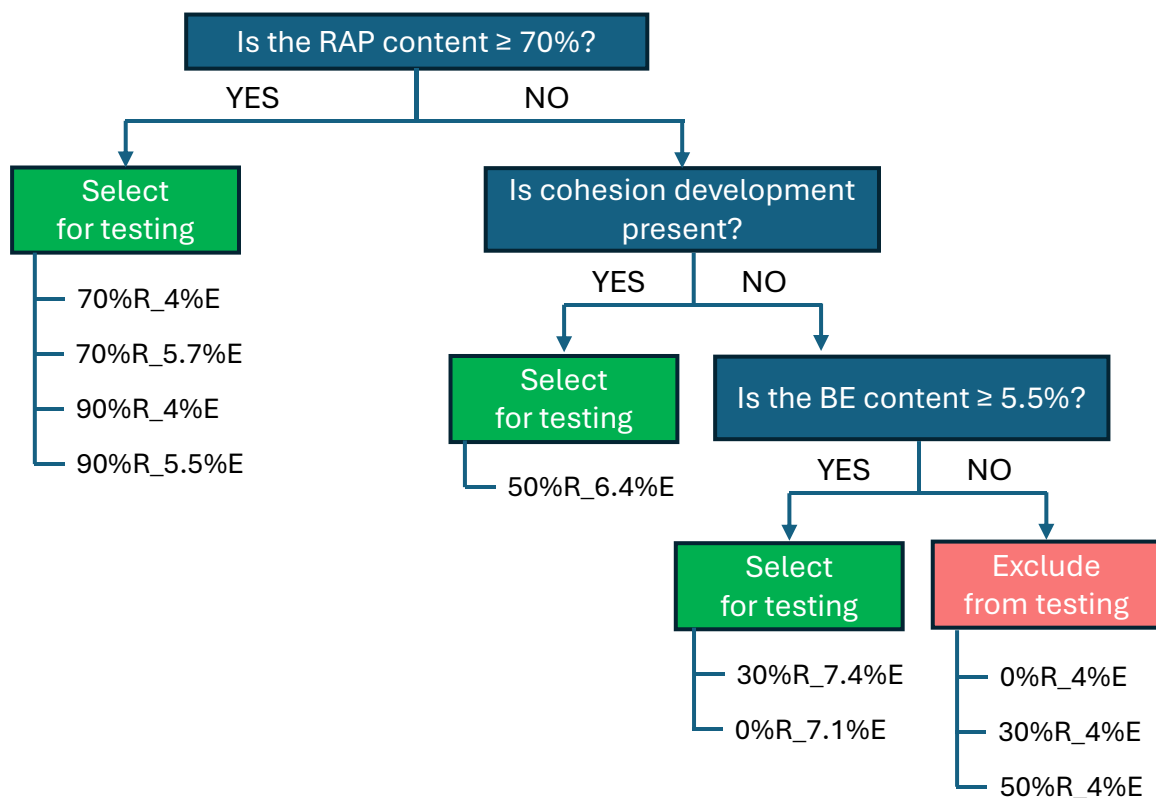


Figure 51 Decision tree of the BSM-E selection process for viscoelasticity assessment

6. Viscoelasticity assessment

The aim of the analyses presented in this chapter was to determine whether the threshold contents of BE and RAP can be established to ensure a viscoelastic material response, and to investigate the extent to which the cohesive properties of BSM-Es are related to their viscoelastic behaviour. The assessment of BSM-E viscoelasticity was conducted using performance modelling approaches typically applied to asphalt mixtures.

For this purpose, dynamic modulus testing was carried out on long-term conditioned specimens. The BSM-E mixtures selected based on the cohesion test results (section 5.3) were subjected to dynamic mechanical testing performed at several predefined combinations of temperature and frequency, in accordance with the procedure described in section 4.3.3.3. In the first stage of the analysis, the material characteristics of the BSM-E mixtures obtained from dynamic testing – specifically, the arithmetic means of dynamic modulus and phase angle values – were compared with the results for the reference hot mix asphalt (HMA) mixture. Additionally, the rate of change in material parameters was evaluated to preliminarily assess the applicability of the time–temperature superposition principle for describing the mechanical performance of BSM-E mixtures.

Building on these findings, the next step involved the analysis of the interdependence between dynamic modulus and phase angle across the full range of test temperatures and frequencies. These data were used to construct Black curves, Cole–Cole diagrams, and subsequently dynamic modulus and phase angle master curves for the tested mixtures. The qualitative and statistical analysis of this combined dataset served to characterise the viscoelastic response of BSM-Es and to support the evaluation of the relationship between cohesive properties and viscoelastic behaviour.

6.1. Evaluation of the viscoelastic response in dynamic testing

The preliminary analysis of the viscoelastic properties of the BSM-E mixtures included a comparison of average dynamic modulus values (Figure 52) and phase angle values (Figure 53) between the BSM-E mixtures and the AC mixture, tested at a frequency of 10 Hz across four temperatures: 5°C, 10°C, 20°C, and 40°C. In the presented bar charts, the reference values for the asphalt mixture are indicated by a black line, and the result variability is represented by a grey shaded area.

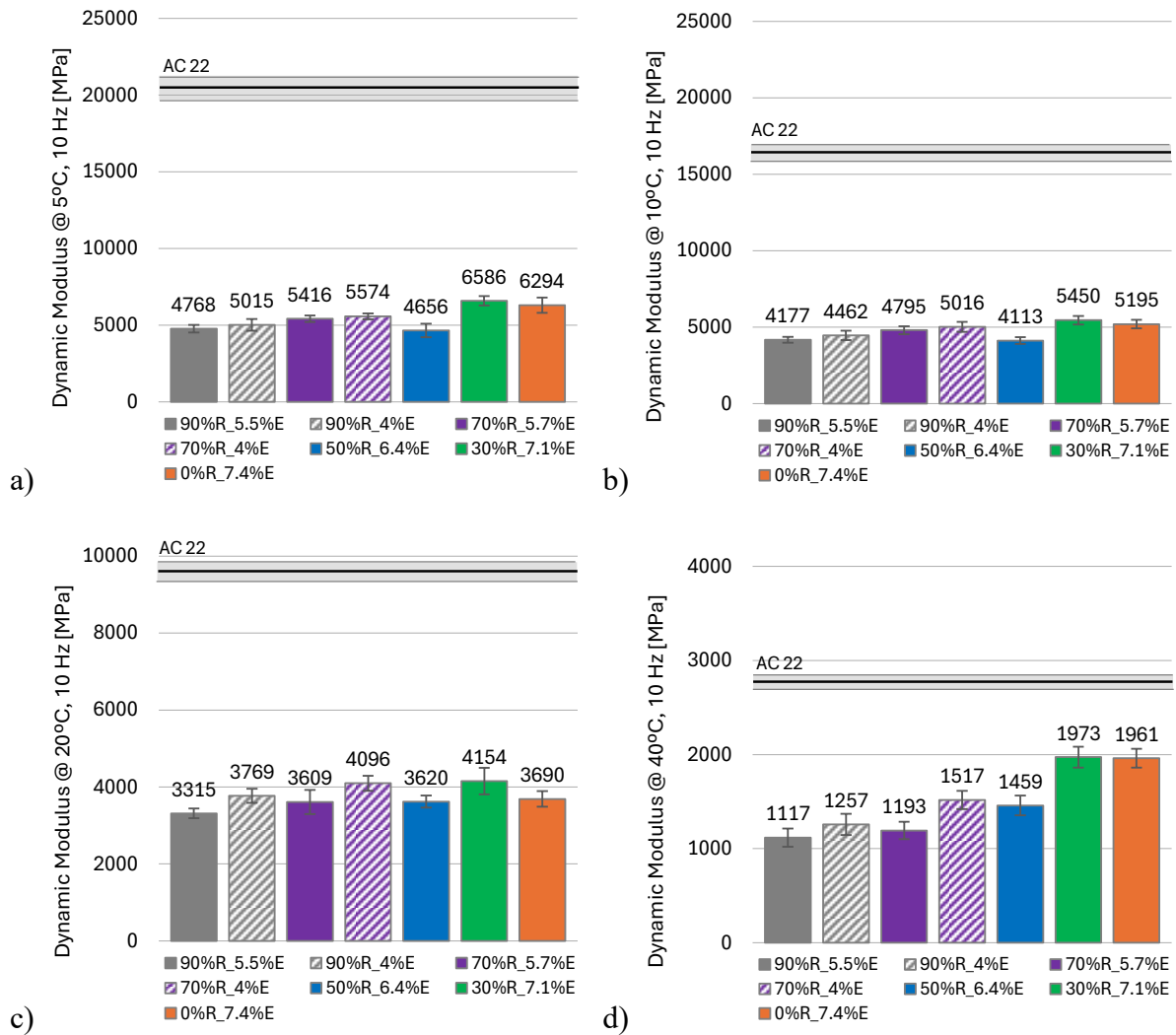
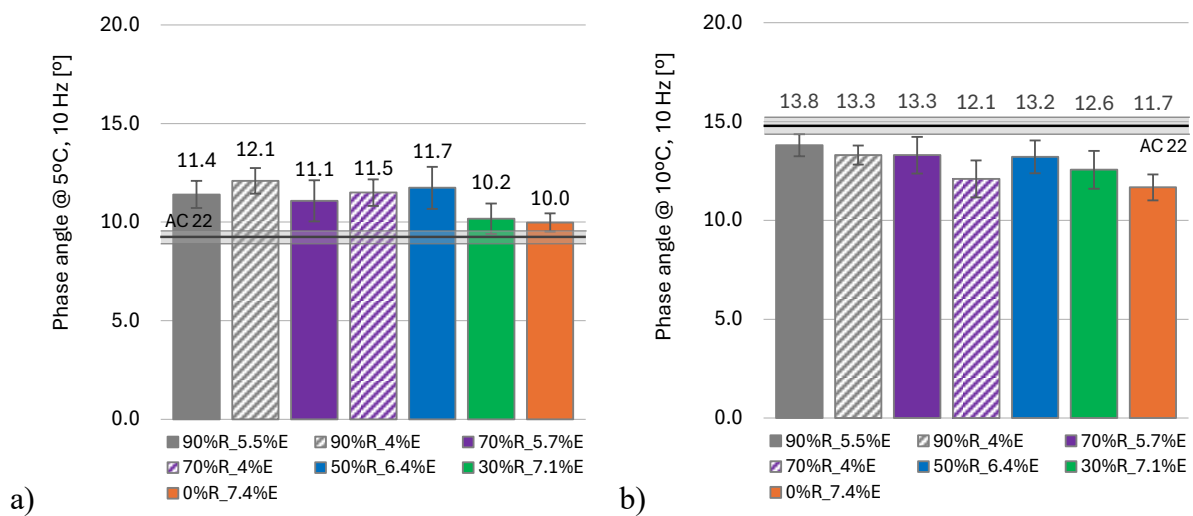


Figure 52 Dynamic modulus of BSM-E and AC mixtures at: a) 5°C, b) 10°C, c) 20°C, d) 40°C.



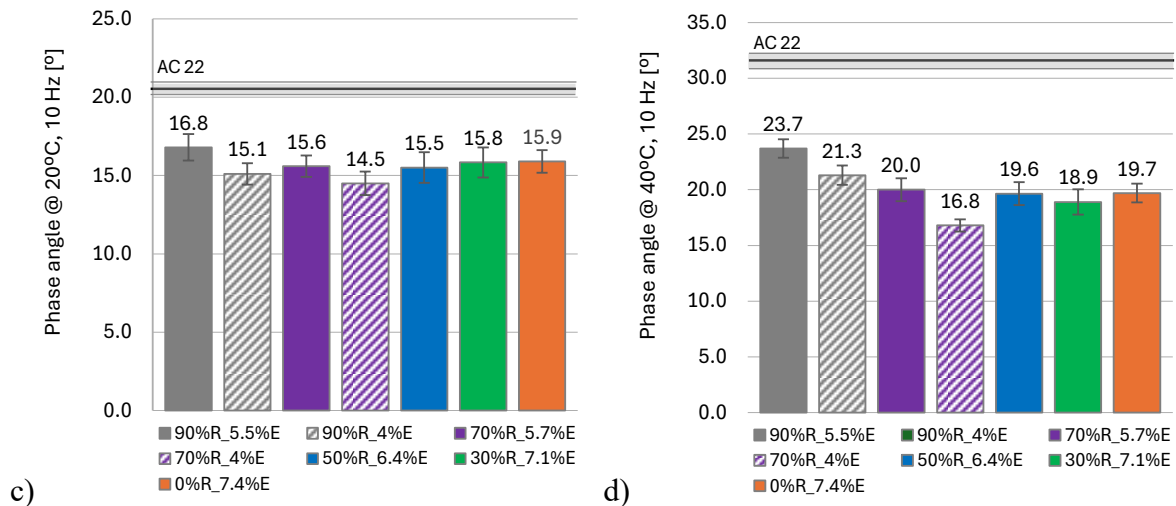


Figure 53 Phase angle of BSM-E and AC mixtures at: a) 5°C, b) 10°C, c) 20°C, d) 40°C.

Based on the results presented in Figure 52, it can be concluded that the BSM-E mixtures exhibited lower levels of dynamic modulus compared to AC mixture at all testing temperatures. The most significant percentage differences were observed at the lowest test temperatures (5°C and 10°C), for which cold recycling mixtures exhibited on average 3.5 – 3.7 times lower values than HMA, while the smallest differences were observed at 40°C. This may be related to the inherent temperature dependency of bitumen in terms of stiffness and viscosity – characterised by stiffening at low temperatures and softening at high temperatures – which, however, manifests differently in these two types of materials. This observation highlights a distinction in the structure and bituminous binder distribution between BSM-E and AC mixtures, particularly notable under low to intermediate temperature conditions. The reduced stiffness can be attributed to the lower continuity of the asphalt mastic phase in BSM-Es. In AC mixtures, the well-developed and cohesive mastic phase ensures effective stress transfer between aggregate particles, even at low temperatures when the binder exhibits stiffening, which results in high dynamic modulus values.

Despite the lower levels of dynamic modulus, the differences between the phase angle values of the analysed BSM-E mixtures and the AC mixture at 5°C were considerably less pronounced. Based on the ANOVA and LSD test results, within the 95% confidence interval, no statistically significant differences in phase angle were observed for the mixtures with the highest BE contents (30%R_7.1%E and 0%R_7.4%E), and thus the highest proportion of fresh residual bitumen (approximately 4.26% and 4.44%). For the remaining mixtures, slightly higher phase angle values were recorded compared to HMA, suggesting a relatively more pronounced viscous response. This may be due to the presence of softer fresh binder in localised regions, which, although not contributing significantly to overall stiffness, still affects the phase

relationship between stress and strain. Additionally, the absence of a fully continuous mastic phase in BSM-Es may lead to increased micro-slippage at the aggregate-binder interfaces under cyclic loading, further increasing energy dissipation. Relatively smaller differences in phase angle values compared to the differences in dynamic modulus between BSM-E and AC mixtures were also observed at a temperature of 10°C. In this case, no statistically significant differences in phase angle values were observed for all BSM-E mixtures (p -value = 0.055) and the average 13% decrease compared to the average phase angle of the AC mixture was noted. Therefore, it can be summarised, that at lower temperatures, the tested BSM-E mixtures demonstrated reduced stiffness, accompanied by a slightly increased or similar contribution of viscous behaviour compared to HMA. This mechanical response suggests a likely different pattern of load transfer through bituminous bonds present in BSM-E mixtures. The presence of localised bituminous bonds and the partial coating of coarse aggregate particles with residual bitumen – expected for BSM with bitumen emulsion – may respond independently under mechanical loading, resulting in a more heterogeneous and less structurally integrated viscoelastic behaviour of the material.

At temperatures of 20°C and 40°C, both the dynamic modulus and phase angle values of the BSM-E mixtures were consistently lower than those observed for AC. While at 20 °C no clear trend was observed in the dynamic modulus values of the BSM-E mixtures, at the highest test temperature a distinctly different pattern emerged. Mixtures with the lowest RAP content and the highest bitumen emulsion content exhibited significantly higher modulus values, forming a statistically homogeneous group (LSD test, p -value = 0.895). The percentage difference between this group and the average value recorded for the asphalt mixture was approximately 29%, whereas the remaining BSM-E mixtures showed reductions in modulus ranging from 45% to 60%. The increase in testing temperature also resulted in a more significant differentiation in the average values of phase angle compared to the lower test temperatures. This was confirmed by the results of the ANOVA mean values comparison, indicating statistically significant differences between the means of the seven BSM-E mixtures at the 5% significance level (p -value = 0.013 for 20°C, and p -value = 0.000 for 40°C).

Due to the material variability of the tested BSM-E mixtures in terms of RAP and bitumen emulsion content, their viscoelastic behaviour was evaluated based on the assessment of their temperature sensitivity. The comparison was carried out based on the average percentage changes in dynamic modulus (Table 27) and phase angle values (Table 28) within specific temperature ranges of the test. Statistically significant differences in parameter values at the 95% confidence level were marked in green, while non-significant differences in red.

Table 27 Percentage changes in dynamic modulus values of BSM-E mixtures in relation to test temperature

Mixture	AC 22	90%R_5.5%E	90%R_4%E	70%R_5.7%E	70%R_4%E	50%R_6.4%E	30%R_7.1%E	0%R_7.4%E
Temperature	Average decrease in dynamic modulus @ 10 Hz							
5°C - 10°C	19%	12%	11%	11%	10%	12%	17%	17%
10°C - 20°C	42%	21%	16%	25%	18%	12%	24%	29%
20°C - 40°C	71%	66%	67%	67%	63%	60%	53%	47%

Table 28 Percentage changes in phase angle values of BSM-E mixtures in relation to test temperature

Mixture	AC 22	90%R_5.5%E	90%R_4%E	70%R_5.7%E	70%R_4%E	50%R_6.4%E	30%R_7.1%E	0%R_7.4%E
Temperature	Average increase in phase angle @ 10 Hz							
5°C - 10°C	61%	21%	10%	20%	5%	13%	24%	17%
10°C - 20°C	38%	22%	14%	17%	20%	17%	26%	36%
20°C - 40°C	55%	41%	41%	28%	16%	27%	19%	24%

Considering changes in dynamic modulus values between 5°C and 10°C, it can be stated that all of the BSM-E mixtures exhibited a less significant viscoelastic response to temperature changes compared to HMA. For mixtures containing 4% bitumen emulsion and the highest RAP content (70%R_4%E and 90%R_4%E), no statistically significant differences in mean values were identified. This may be attributed to the lower proportion of "fresh" residual bitumen, which is more thermally responsive than the aged RAP binder and plays a more prominent role in determining material stiffness at low temperatures. In contrast, for the remaining mixtures with BE contents exceeding 5.5%, statistically significant stiffness changes were observed, ranging from 11% to 17%.

In the two subsequent ranges (10 °C–20 °C and 20 °C–40 °C), all tested BSM-E mixtures exhibited a statistically significant decrease in average dynamic modulus values with the increase in temperature. Within the 10 °C–20 °C range, no clear trend was observed with respect to BE content, in contrast to the behaviour noted at lower temperatures. Similarly, no consistent pattern related to RAP content was identified. The percentage changes in modulus within this range varied between 12% and 29%. At the highest test temperatures (20 °C–40 °C), the greatest percentage reductions in modulus values (ranging from 60% to 67%) were recorded for mixtures with intermediate to high RAP content (50%–90%), regardless of the emulsion content.

Analysing the changes in phase angle values, it can be stated that this parameter was relatively less sensitive to temperature variations than the dynamic modulus. Statistically significant changes in average phase angle values between 5°C and 10°C were observed only for mixtures with the high bitumen emulsion content (0%R_7.4%E and 30%R_7.1%E), and mixtures with high RAP content (90%R_5.5%E, 70%R_5.7%E). Nevertheless, the observed range of changes (17%–24%) was markedly lower than that recorded for the AC mixture, which reached 61%. The level of change in average phase angle values at 10°C and 20°C for most mixtures was similar to that observed at lower temperatures, ranging from 14% to 26%. The highest increase – comparable to that recorded for the AC mixture (36%) – was observed for the BSM-E mixture with the highest bitumen emulsion content and no RAP (0%R_7.4%E). In the highest temperature range of the study (20°C–40°C), the greatest increases in phase angle – indicating the most pronounced transition of the material toward a viscous state – were observed for the mixtures with the highest RAP content (90%R_5.5%E and 90%R_4%E).

Based on the presented data, it can be concluded that, in general, BSM-E mixtures exhibited changes in the average dynamic moduli and phase angle in relation to the test temperature. Nevertheless, it was found that all tested BSM-Es exhibited lower percentage decreases in modulus as well as smaller increases in phase angle compared to HMA. However, while the absolute changes in the parameter values indicate a lower BSM-Es' temperature sensitivity compared to the reference viscoelastic material, the BSM-Es' thermal response is generally consistent enough to produce statistically significant differences. This, in consequence, confirms the relevance of further analysing the viscoelastic properties of these materials, taking into account the results obtained in the frequency-sweep testing mode.

6.2. Application of the time–temperature superposition principle to the mechanical behaviour of BSM-E mixtures

The dynamic modulus and phase angle of viscoelastic materials, such as asphalt mixtures, are both strongly dependent on loading frequency and temperature. Therefore, it was assumed that in the case of BSM-E mixtures exhibiting viscoelastic properties, the material will exhibit a more elastic behaviour as frequency increases, resulting in an increase in the modulus and a decrease in the phase angle. Conversely, at lower frequencies, the dynamic modulus was expected to decrease and the phase angle to increase, indicating a greater contribution of the viscous component. With the increase in temperature, a notable reduction in the BSM-Es' dynamic modulus and an increase in phase angle was assumed, as the material softens and behaves in a more viscous manner. In contrast, for lower temperatures, an increase in stiffness

and reduction in the phase angle, should be observed, promoting a more elastic response of the composite.

To verify whether the response of BSM-E mixtures aligns with, and to what extent it resembles, the behaviour of viscoelastic materials, their properties were compared with those of a HMA mixture type AC 22, which served as a reference for evaluating variations in the measured parameters. The first step of the analysis was the qualitative assessment of the Black curves and Cole-Cole diagrams developed for the analysed BSM-E mixtures as initial indicators of the viscoelastic character of the analysed BSM-Es. The interpretation was carried out by comparing the results obtained for BSM-E and AC mixtures in terms of the shape and position of the curves, allowing for a visual and analytical evaluation of the linear viscoelastic domain and the degree to which each material exhibits elastic–viscous balance. This comparative approach provided further insight into the viscoelastic nature of BSM-E mixtures and the relevance of their cohesive performance in defining their mechanical performance. For the positively verified mixtures, master curves of the dynamic modulus were constructed and the goodness-of-fit of the experimental data to the calculated values was assessed.

The Black curve, representing the relationship between phase angle and the logarithm of the complex modulus, serves as a useful tool for evaluating the viscoelastic behaviour of asphalt mixtures. The shape of the Black curve effectively illustrates how stiffness and energy dissipation co-evolve across a range of frequencies and temperatures. A smooth and continuous curve is generally indicative of linear viscoelastic behaviour, material homogeneity, and consistent rheological properties. In contrast, any deviations from the expected curve shape – such as irregularities and discontinuities – may point to issues such as material damage, nonlinearity, phase separation, or poor reproducibility of the experimental data.

The Cole–Cole diagram is a parametric representation of the complex modulus, where the imaginary component (loss modulus E'') is plotted against the real component (storage modulus E') across a range of loading frequencies. It provides insight into the viscoelastic nature of materials by illustrating how energy storage and dissipation interact under dynamic loading. For ideal linear viscoelastic materials, the diagram forms a semi-circular arc, indicating a uniform distribution of relaxation mechanisms. However, in the case of asphalt mixtures, which are inherently heterogeneous and composed of mineral aggregates and bituminous binder, the curve typically deviates from this ideal shape. The resulting plots are often flattened, asymmetric, or distorted, reflecting the combined contribution of the elastic mineral skeleton and the viscous binder phase. As such, for the purpose of this study, the Cole–Cole diagram

served as a complementary tool to the Black diagram for the qualitative assessment of viscoelastic behaviour in BSM-Es.

The subsequent step of the data analysis was the construction of master curves for mixtures exhibiting viscoelastic performance. The concept behind constructing master curves for asphalt mixtures is based on defining a continuous relationship between the complex modulus and frequency at a selected reference temperature, using partial data obtained from dynamic modulus testing conducted under several predefined temperature–frequency combinations. For the analysis performed in this study, a reference temperature of 20°C was adopted, and a commonly used Standard Logistic Sigmoidal model (14) was applied for the mathematical representation of the curve. The value of the reduced frequency (f_r), which links the value of the modulus to the temperature, was calculated according to the equation (17). To calculate the value of the shift factor a_T , the Arrhenius equation (16) was used, which is based solely on temperature values. The final values of the curve-fitting parameters ($\delta, \beta, \gamma, \Delta E_a$) were obtained through iterative optimisation of the equation (14). The optimisation procedure entailed iterative adjustment of the fitting parameters with the objective of minimising the sum of squared deviations between the experimentally obtained values and those predicted by the model.

The phase angle master curve was fitted according to the improved Gauss model [178], applying the same shift factors (a_T) calculated for the dynamic modulus master curves. The Gauss model is shown in equation (18):

$$\varphi = \varphi_0 + Ae^{\frac{(\log(fr)-fc)^2}{2f^2}} \quad (18)$$

where:

φ – phase angle,

f_r – frequency at the reference temperature,

f – spread width of the Gaussian curve (fitting parameter),

f_c – frequency at peak of the phase angle (fitting parameter),

φ_0 – baseline phase angle (fitting parameter),

A – phase angle amplitude – peak rise above baseline (fitting parameter).

The analysed master curve goodness-of-fit parameters included: R^2 and S_e/S_y ratio. The S_e/S_y ratio, defined as the quotient of the standard error of the estimate (S_e) and the standard deviation of the observed values (S_y) reflects the relative magnitude of the model's fitting error in comparison to the inherent variability of the data. An S_e/S_y value close to 0

indicates a very good fit, as the model closely reproduces the observed data. Values below 0.5 are generally considered acceptable, while values approaching or exceeding 1 suggest that the model fails to explain the variability in the data.

6.2.1. Evaluation of the reference viscoelasticity – asphalt mixture performance

Figure 54 a),b) presents the Black curve and Cole–Cole diagram constructed from the test results obtained for a representative specimen of the reference asphalt concrete mixture AC 22.

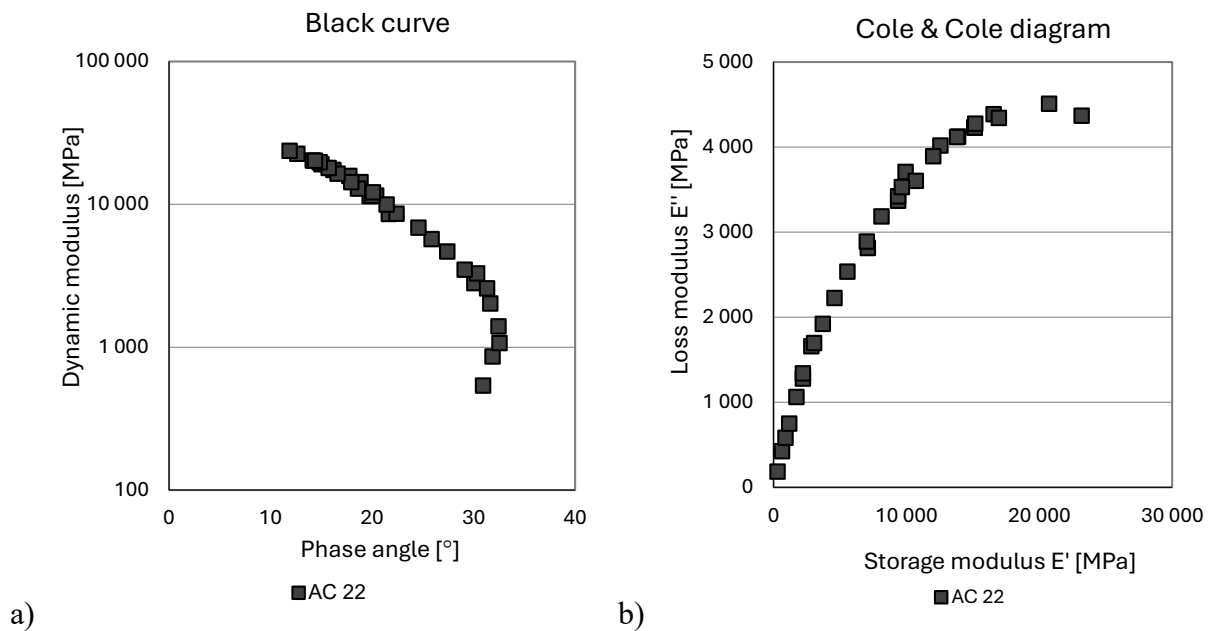


Figure 54 Viscoelasticity assessment of the AC 22 mixture: a) Black curve, b) Cole & Cole diagram.

The Black curve (Figure 54 a)) presents a smooth and continuous trend, with the dynamic modulus decreasing progressively as the phase angle increases, confirming the validity of the time–temperature superposition principle for the tested asphalt concrete. The absence of any discontinuities suggests an internal homogeneity and stable structural integrity throughout the tested temperature and frequency range. The curve spans a wide stiffness domain, ranging from approximately 500 MPa to 25000 MPa, while the phase angle varies from approximately 9° to 36°, reflecting a transition from predominantly elastic to increasingly viscous responses at lower stiffness levels. The corresponding Cole–Cole diagram (Figure 54 b)) exhibits an arc-like curve, illustrating the relationship between the loss and storage modulus, deviating from the ideal semicircular form expected for viscoelastic asphalt materials. The peak in the loss modulus was observed at approximately 4500 MPa.

Figure 55 presents the dynamic modulus master curve for the reference HMA mixture constructed using time–temperature superposition principles. The maximum limiting modulus

of the master curve, representing its upper asymptote, was estimated at 29000 MPa based on literature data and experimental experience. The experimental data were fitted with a sigmoidal SLS model curve, providing a smooth representation of the material's viscoelastic behaviour across a wide frequency spectrum. The curve exhibits a typical S-shaped profile, indicating a transition from a predominantly viscous response at low reduced frequencies to a predominantly elastic response at high reduced frequencies. The obtained high values of R^2 and S_e/S_y demonstrate a statistically robust correspondence between the measured and calculated values of the dynamic modulus (Table 29).

Table 29 Fitting parameters and goodness-of-fit measures of the dynamic modulus master curve for AC 22

β [-]	δ [-]	γ [-]	ΔE_a [J/mol]	R^2	S_e/S_y
-0.6352	2.3989	-0.5234	224774	0.996	0.071

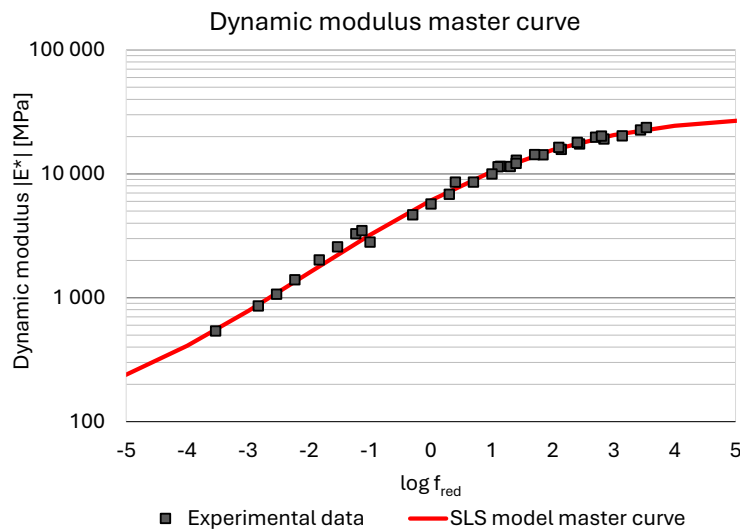


Figure 55 Dynamic modulus master curve for AC 22 mixture

The phase angle master curve for the AC mixture constructed by fitting the Gauss-based mathematical to the experimental data is presented in Figure 56, while the values of the fitting parameters and the goodness-of-fit indicators are summarized in Table 30. Based on the analysis of the data, it can be stated that the master curve is characterized by a typical bell shape consistent with phase angle trends in asphalt mixtures – exhibiting higher phase angles in the range of the lower reduced frequencies and presenting more elastic response at high loading frequencies. The calculated R^2 and S_e/S_y statistical characteristics show that the model explains about 97.6% of the variation in the experimental phase angle values, while the prediction error is equal to approximately 17% of the variability in the experimental data, both indicating a high-quality model fit.

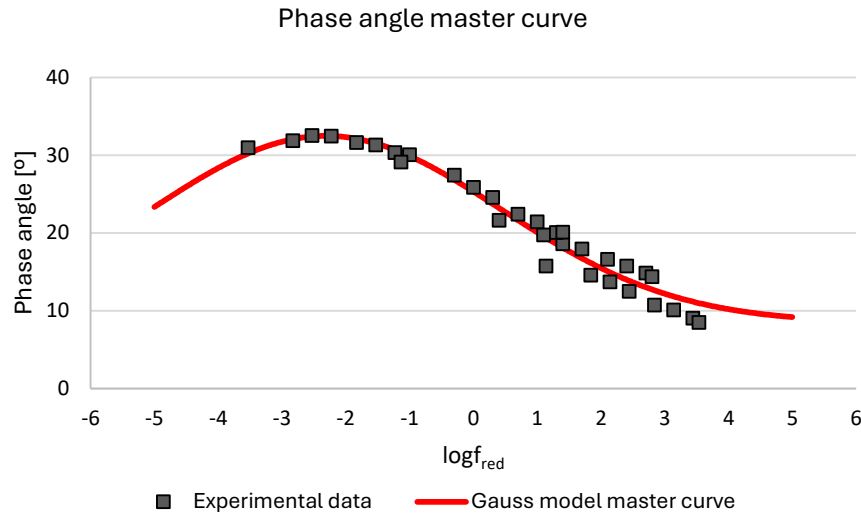


Figure 56 Phase angle master curve for AC 22 mixture

Table 30 Fitting parameters and goodness-of-fit measures of the phase angle master curve for AC 22

δ_0 [°]	A [°]	fc [-]	f [-]	R^2	S_e/S_y
8.485	23.989	-2.313	2.746	0.976	0.163

The presented analysis of the dynamic test results for the AC mixture demonstrates that the qualitative evaluation of graphical outputs such as the Black curve and Cole-Cole diagram (Figure 54), as well as the construction of master curve models (Figure 55, Figure 56) combined with a qualitative assessment of their fit to the experimental data, provide a useful tool for assessing the viscoelasticity of materials. Since clear evidence that the analysed AC 22 reference mixture exhibits rheological characteristics typical of viscoelastic materials has been provided, its use as a benchmark in the comparative analyses involving BSM-E mixtures has been validated.

6.2.2. Analysis of the BSM-E mixtures' mechanical response

6.2.2.1. Qualitative assessment

The viscoelastic properties of BSM-E mixtures exhibiting favourable cohesion-related characteristics were analysed. These characteristics resulted from specific material conditions as well as the combined influence of RAP and bitumen emulsion (BE) content and the structural changes occurring in the material over time due to external factors. Figure 57 a),b) presents Black curve and the Cole-Cole diagram for representative specimens of mixtures with the highest RAP addition.

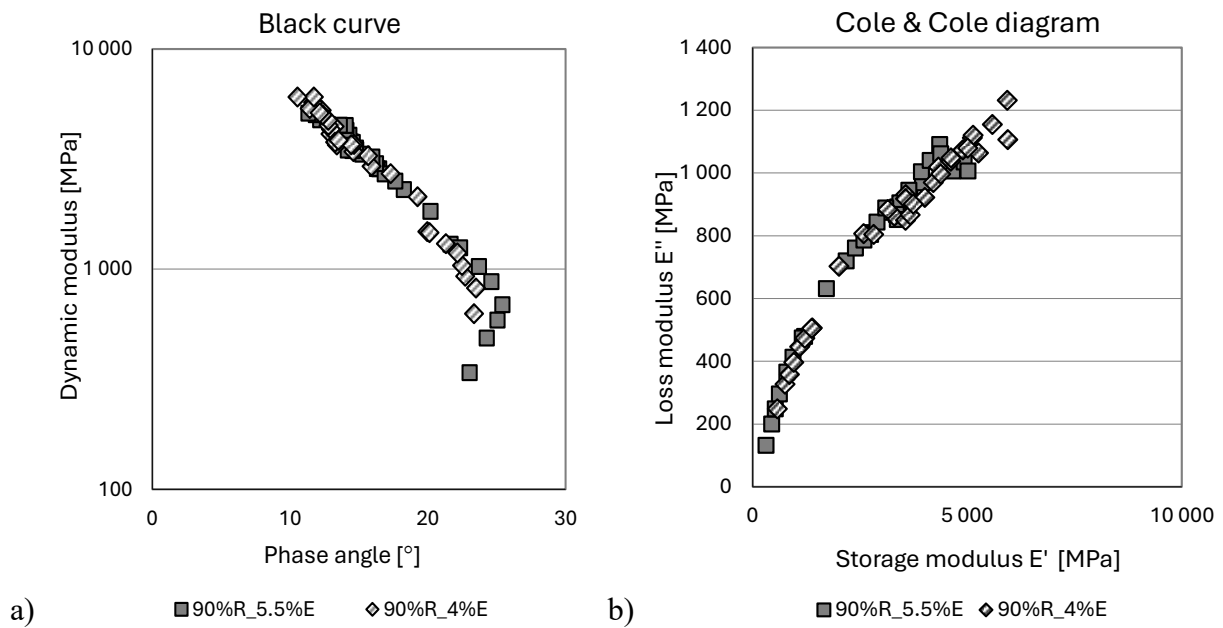


Figure 57 Viscoelasticity assessment of the 90%RAP mixtures: a) Black curve, b) Cole & Cole diagram.

Based on the analysis of the presented graphs, it can be stated that both 90%R_5.5%E and 90%R_4%E mixtures exhibit a typical viscoelastic material profile. The Black curves (Figure 57 a)) demonstrate smooth, continuous trends for both mixtures, with the dynamic modulus decreasing monotonically with increasing phase angle. After reaching the phase angle peak at approximately 23–25°, both curves show a decline in phase angle as the dynamic modulus continues to decrease. These data provide insight into the conditions under which these materials begin to lose their viscoelastic properties, transitioning into a viscous state. The Cole–Cole diagrams (Figure 57 b)) for both mixtures form well-defined arcs, further indicating a viscoelastic response and suitability for time–temperature superposition analysis. While the overall rheological response of the two mixtures remains similar, some differences in their performance are notable. The 90%R_5.5%E mixture demonstrated a broader dynamic modulus range and a wider phase angle span, while the 90%R_4%E mixture showed a higher minimum dynamic modulus value and a slightly narrower phase angle range, indicative of a stiffer and more elastic response, likely due to its lower binder content. The 90%R_4%E mixture tends to exhibit slightly lower phase angles, suggesting a modestly more elastic character, whereas the 90%R_5.5%E mixture shows a slightly higher tendency toward energy dissipation, reflecting a marginally greater viscous contribution. These trends align with the tested cohesive properties of the mixtures and are likely influenced by the varying emulsion content. However, the distinctions are not pronounced and both mixtures can be considered to fall within the same general category of cohesive, viscoelastic BSM-E materials.

Black curve and the Cole-Cole diagram for mixtures with a 70% RAP content and confirmed cohesion development are presented in Figure 58.

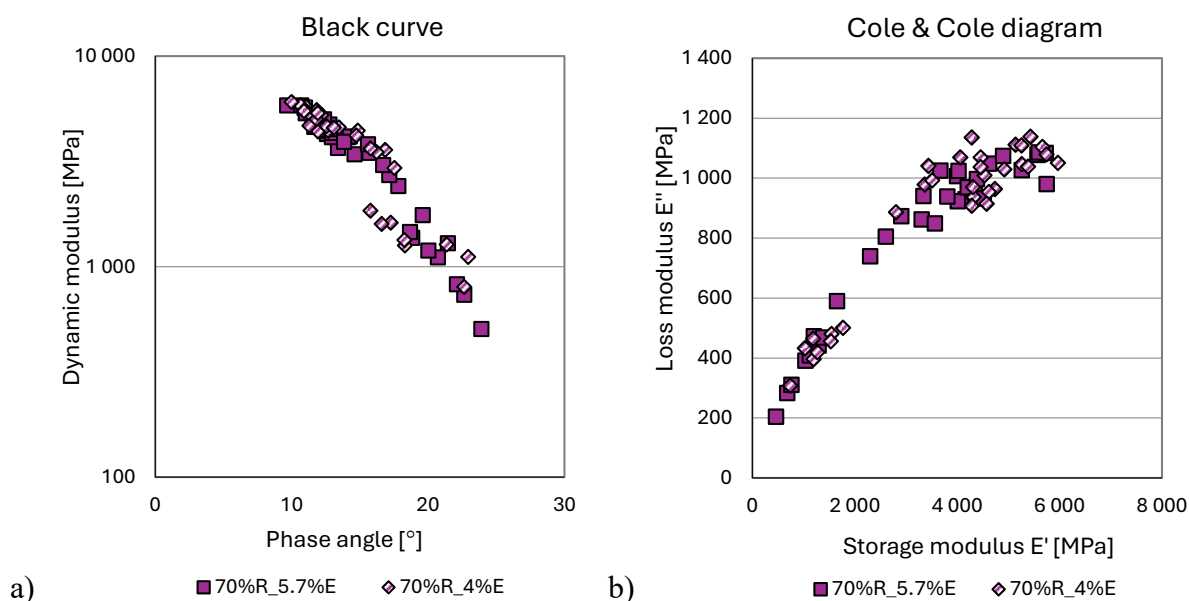


Figure 58 Viscoelasticity assessment of the 70%RAP mixtures: a) Black curve, b) Cole & Cole diagram.

Based on the data presented in Figure 58 a),b), it can be stated that both BSM-E mixtures with a 70% RAP content exhibited general trends in the dynamic modulus and phase angle characteristic for the viscoelastic behaviour. However, in the case of the 70%R_4%E mixture, local discontinuities and data clustering have been observed, most likely resulting from the low dynamics in modulus and phase angle changes within the lowest test temperatures (5°C – 10°C) – as presented for the 10 Hz test results in Table 27 and Table 28. Slightly wider ranges of phase angle and dynamic modulus determined for the 70%R_5.7%E mixture at the applied temperatures and loading frequencies might indicate that incorporating higher amounts of the fresh residual binder from BE results in a material more responsive to changes in loading conditions. In the Cole–Cole diagram (Figure 58 b)), both mixtures follow arc-like, upward-sloping curves characteristic of viscoelastic materials. However, the 70%R_4%E mixture exhibited scatter in the results, presenting a non-continuous curve, conversely to the 70%RAP BSM-E mixture with a higher BE content. Therefore, for the 70%R_4%E mixture, a more in-depth investigation of its potential viscoelastic properties based on master curves appears particularly justified.

Test results obtained for the representative specimens from the last group of BSM-E mixtures selected for viscoelasticity assessment are presented in Figure 59 – Figure 61. For these mixtures, characterised by low to intermediate RAP content (0%–50%) and high BE content (6.4%–7.4%), no cohesion development in time was observed.

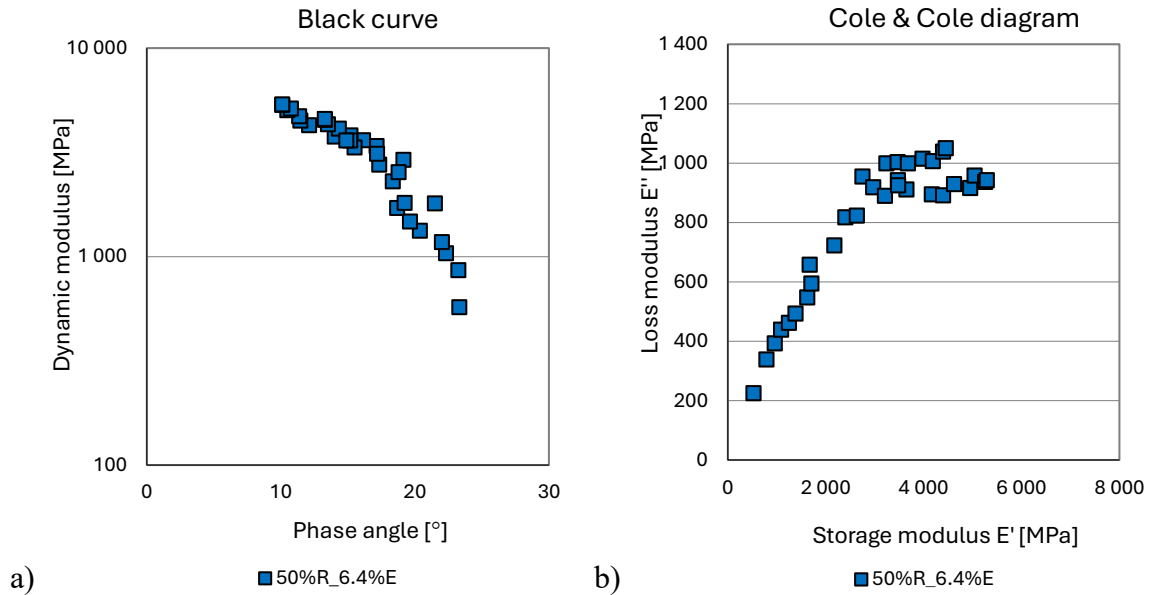


Figure 59 Viscoelasticity assessment of the 50%RAP mixture: a) Black curve, b) Cole & Cole diagram.

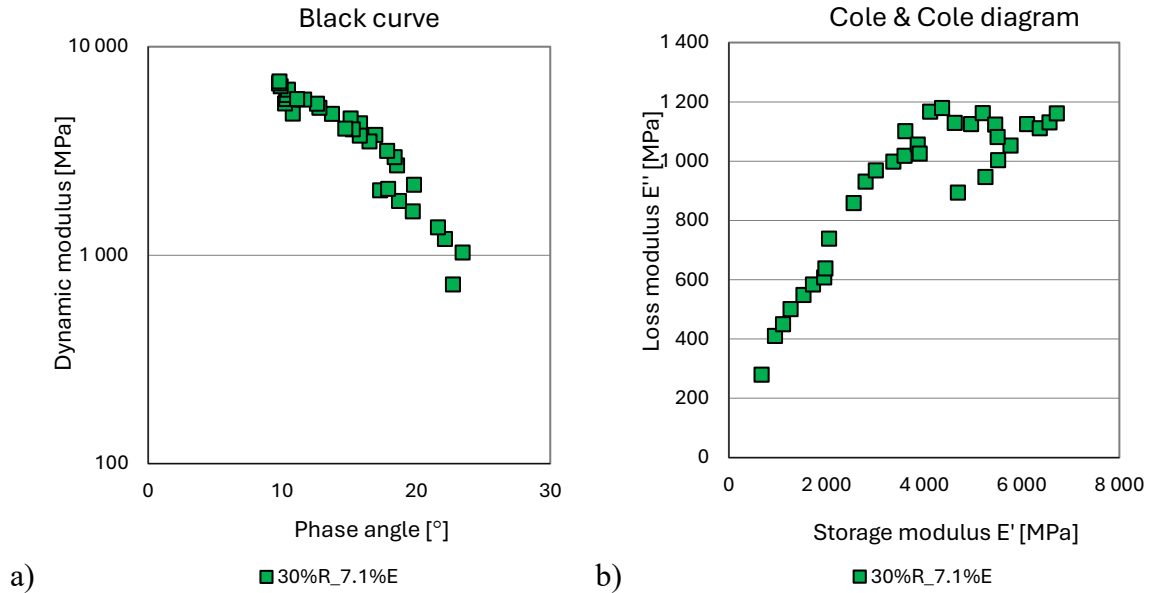


Figure 60 Viscoelasticity assessment of the 30%RAP mixture: a) Black curve, b) Cole & Cole diagram.

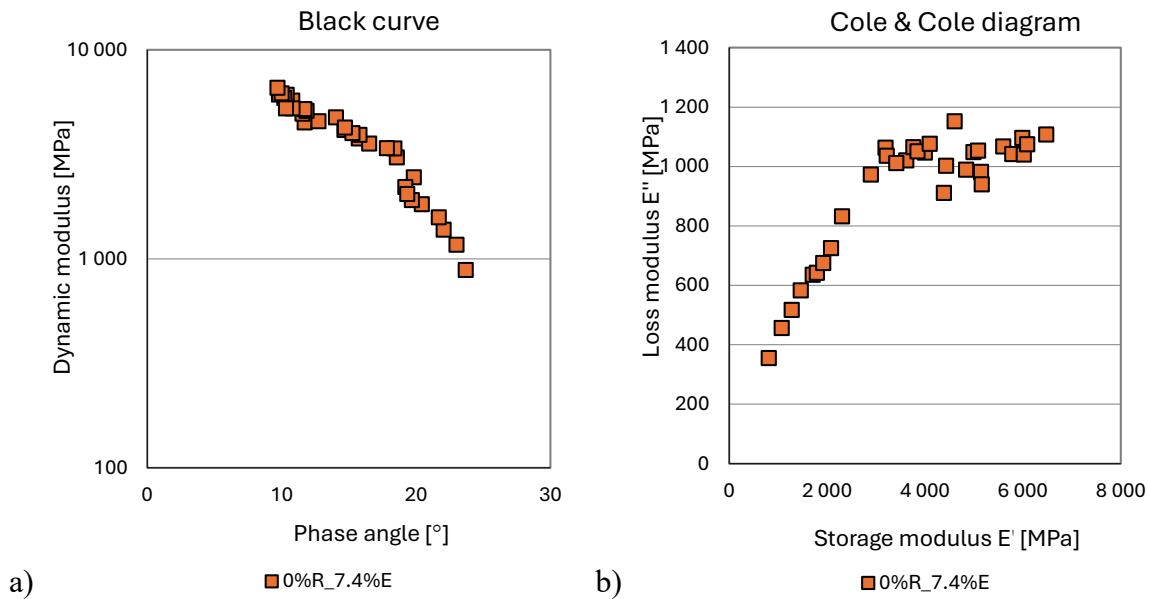


Figure 61 Viscoelasticity assessment of the 0%RAP mixture: a) Black curve, b) Cole & Cole diagram.

Based on the results presented in Figure 59 – Figure 61, it can be stated that all three mixtures demonstrated a similar variation in phase angle values under the adopted test conditions. For the dynamic modulus, the ranges obtained for mixtures with 0% and 30% RAP were wider compared to the mixture with a 50% RAP addition. A qualitative assessment of the curve profiles obtained for representative samples of the tested mixtures indicates that, in all cases, some degree of scatter and local clustering of results occurred - particularly within the low-temperature testing range. This corresponds to low phase angle values and high dynamic modulus values, which are indicative of the elastic behaviour of the material. In the case of the 50%R_6.4%E mixture, the Cole-Cole diagram (Figure 59 b) revealed overlapping result series and an almost parallel course of the calculated loss and storage moduli at test temperatures of 5°C and 10°C. The observed profiles in both the Black and Cole-Cole diagrams (Figure 59 a),b)) for this mixture are consistent with the relatively low percentage changes in modulus and phase angle values between individual test temperatures at a frequency of 10 Hz (Table 27, Table 28), indicating low temperature sensitivity of this mixture despite its high bitumen emulsion content. For mixtures with lower RAP content (0% and 30%), which – due to the high BE content – would be expected to exhibit sensitivity to temperature and loading frequency, the irregularities were observed in the profiles of both curves, especially in terms of the results obtained for the low-temperature conditions (ranges of the highest values of the loss and storage moduli). For the mixture containing 30% RAP, a low sensitivity of the phase angle to changes in testing frequency at 5°C was observed, as evidenced by the clustering of results in the region of low phase angle values of the Black diagram (Figure 60 a)). A slight horizontal shift relative to the main trend of the Black curve was also noted for the results obtained at

40°C, suggesting lower than expected phase angle values for the given dynamic modulus values. A more pronounced deviation from the main trend in the Cole-Cole diagram (Figure 60 b)) was observed for the results recorded at 5°C and the lowest testing frequencies (0.1–1 Hz). Similar tendencies were observed for the 0%R_7.4%E mixture; however, both the Black and Cole-Cole curves were more uniform in shape. The aggregation of results was most evident in the 5–10°C temperature range. These findings justify further analysis of the viscoelastic behaviour of the tested mixtures through the development and comparison of master curves, enabling a more comprehensive interpretation of their time–temperature dependent performance.

6.2.2.2. *Master curve fitting*

The master curve fitting for BSM-Es was carried out using an approach commonly applied to asphalt mixtures. For comparative purposes in evaluating the accuracy of the fit to the experimental data, a simplification was introduced in the determination of fitting parameters. Specifically, the lower and upper asymptotes of the dynamic modulus master curve were set based on the average minimum (δ) and maximum ($|E^*|$) modulus values obtained for each type of tested BSM-E mixture. For the phase angle master curves, a fixed baseline phase angle (φ_0) was adopted, corresponding to the average minimum value observed in the test results. This approach to constructing master curves was intended to yield mathematical models with fitting parameters that are credible in the context of typical properties of mixtures containing bituminous binders. It also reflects the characteristics of the applied models – Gauss models with a free φ_0 parameter tend to over-adapt to steep gradients, which can lead to overfitting in the curve tails and result in unrealistically low baseline phase angle values. Furthermore, limiting the temperature range for analysing minimum and maximum values of modulus and phase angle to the actual test temperatures (5°C to 40°C) is justified not only by the need to apply consistent comparison criteria, but also by the fact that BSM-E mixtures, when used in base layers, are exposed to a narrower temperature range within pavement structures (the deeper the layer placement, the smaller the daily and annual temperature variations).

Figure 64 presents dynamic modulus master curves constructed for the analysed BSM-E mixtures in comparison to the master curve calculated for the asphalt mixture. The fitting parameters and the goodness-of-fit measures for the SLS curve models are summarised in Table 31.

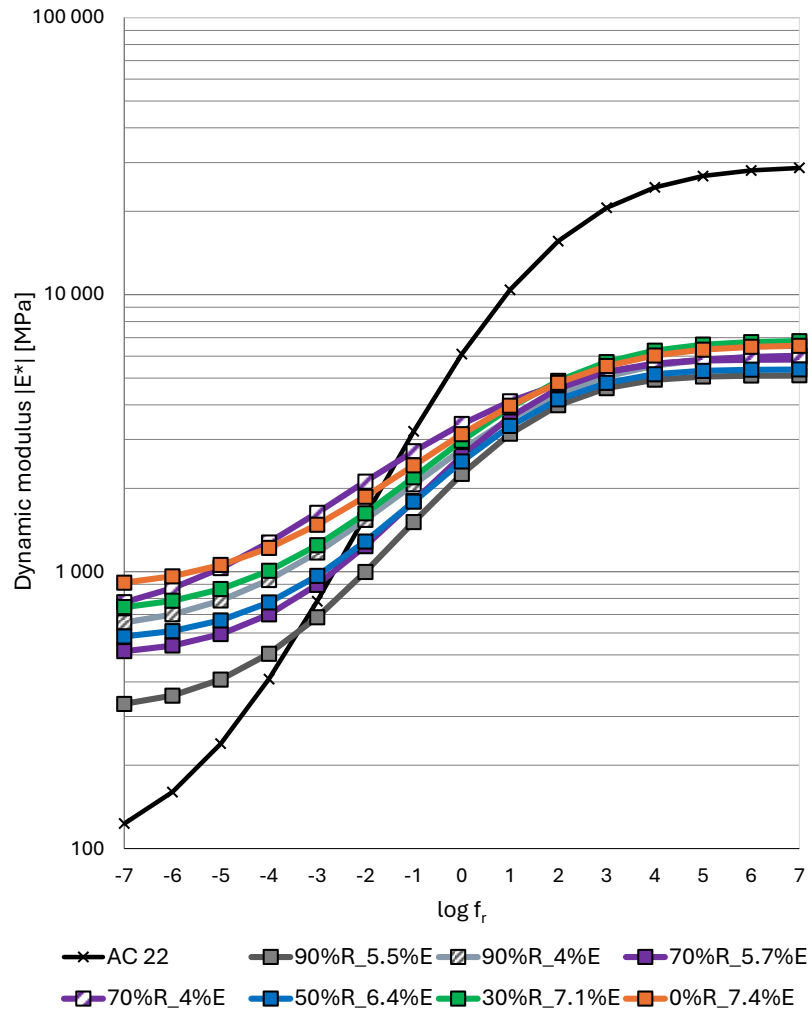


Figure 62 Dynamic modulus master curves of the selected BSM-Es and AC 22 mixture

Based on the presented master curve of the dynamic modulus $|E^*|$ as a function of reduced frequency $\log(f_r)$, several conclusions can be drawn regarding the viscoelastic behaviour of the tested materials and the applicability of the time–temperature superposition principle. All tested mixtures, including AC 22 and BSM-Es, exhibited an S-shaped curve characteristic of viscoelastic materials, confirming a typical dependence of stiffness on frequency (i.e., indirectly on loading time and temperature). The AC 22 mixture demonstrates the highest variability in dynamic modulus across the frequency spectrum, which reflects its strong sensitivity to time–temperature factors due to the presence of a continuous asphalt mastic phase. In contrast, the BSM-E mixtures show lower ranges of modulus variation, indicating a more attenuated response and reduced sensitivity to temperature and loading duration. However, the smooth and continuous shape of the BSM-E master curves supports the applicability of TTSP for describing their mechanical behaviour. This is confirmed by the calculated goodness-of-fit measures (Table 31). The obtained values of the coefficient of

determination R^2 , ranging from 0.970 to 0.988, indicate a very good agreement between the model and the experimental data. Additionally, the low values of the goodness-of-fit indicator S_e/S_y (ranging from 0.114 to 0.183) further confirm the high quality of the model fit to the measured results.

Table 31 Fitting parameters and goodness-of-fit measures of the dynamic modulus master curve for BSM-Es

Mixture	β [-]	δ [-]	γ [-]	ΔE_a [J/mol]	R^2	S_e/S_y
90%R 5.5%E	-0.6324	2.6332	-0.6605	223942	0.983	0.135
90%R 4%E	-0.4955	2.8320	-0.5284	302275	0.984	0.134
70%R 5.7%E	-0.2751	2.9195	-0.6966	243429	0.985	0.130
70%R 4%E	-0.8949	2.8699	-0.4847	326689	0.970	0.183
50%R 6.4%E	-0.4583	2.8296	-0.6440	236366	0.988	0.114
30%R 7.1%E	-0.3862	2.8916	-0.5514	278683	0.986	0.124
0%R 7.4%E	-0.4101	2.9654	-0.5215	280172	0.980	0.149

The orderly distribution of data along the reduced frequency axis suggests that constructing master curves by horizontal shifting of the frequency-temperature dynamic modulus data is valid. Although BSM-E mixtures exhibit a less pronounced viscoelastic character compared to conventional HMA, their mechanical response remains consistent and measurable, justifying the application of the TTSP.

Considering solely the BSM-E mixtures, the divergence in dynamic modulus values within the range of low reduced frequencies ($\log(f_r)$ from 0 to -7) and the convergence of the curves at higher frequencies was observed, which provides important insights into the viscoelastic behaviour of these materials. Low frequencies correspond to long loading times and/or elevated temperatures, which are conditions under which the viscous component of the material response becomes dominant. Differences in dynamic modulus values in this range suggest that the tested mixtures exhibit varying susceptibility to creep and permanent deformation, likely resulting from differences in cohesive structure, RAP and bitumen emulsion content, as well as the interaction between residual binder and the mineral skeleton. In contrast, the convergence of the modulus curves at higher reduced frequencies – associated with short loading times or low temperatures – indicates that the materials respond in a comparable manner under quasi-elastic conditions. Therefore, the divergence at low frequencies emphasizes the importance of the viscous component in long-term pavement performance, while the convergence at high frequencies confirms consistent elastic response across the tested BSM-E variants.

Figure 63 presents the phase angle master curves for the reference AC 22 mixture and the selected BSM-E mixtures. Consistently with the trends noted in the dynamic modulus master curves, the broadest range of phase angle variability was observed for the asphalt concrete mixture. However, in this instance, a greater variation in the shape and distribution of

the curves was observed specifically within the group of BSM-E mixtures, indicating increased heterogeneity in their viscoelastic response in this parameter domain. The differences in the shape of the master curves were quantified using the fitting parameter values presented in Table 32. In general, the analysed cold recycling mixtures were characterised with the lower values of curve peaks shifted towards lower frequencies ($\log(f_r)$) in the range of -3 to -2) compared to the HMA, which corresponds to longer loading times and/or higher temperatures.

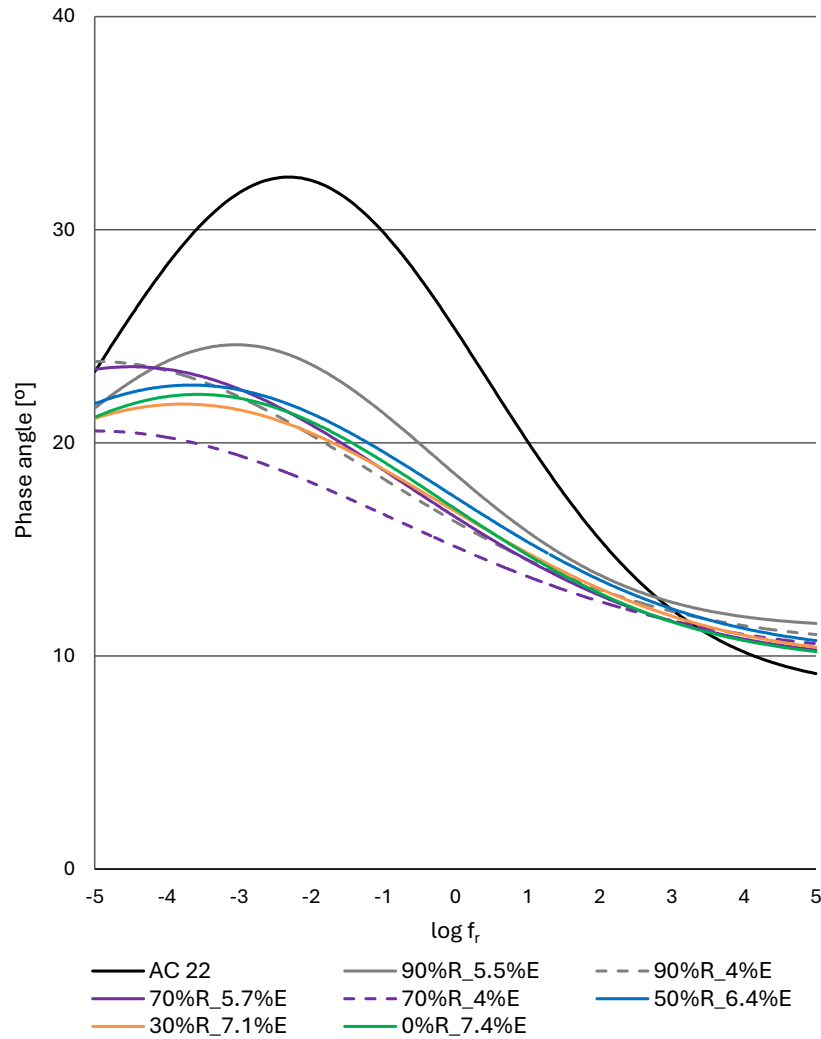


Figure 63 Phase angle master curves of the selected BSM-Es and AC 22 mixture

Table 32 Fitting parameters and goodness-of-fit measures of the phase angle master curve for BSM-Es

Mixture	δ_0 [°]	A [°]	f_c [-]	f [-]	R^2	S_e/S_y
90%R_5.5%E	11.35	13.26	-3.044	2.746	0.965	0.195
90%R_4%E	10.55	13.28	-4.994	3.859	0.991	0.099
70%R_5.7%E	9.70	13.88	-4.493	3.770	0.977	0.158
70%R_4%E	9.99	10.57	-5.000	4.163	0.885	0.351
50%R_6.4%E	10.11	12.61	-3.666	3.524	0.907	0.316
30%R_7.1%E	9.77	12.05	-3.774	3.626	0.875	0.366
0%R_7.4%E	9.53	12.58	-3.572	3.383	0.941	0.251

The master curve most closely resembling the characteristics of the HMA curve was observed for the 90%R_5.5%E mixture, presenting the highest phase angle peak and the widest range of the phase angle values. For mixtures containing 0% to 50% RAP and high bitumen emulsion content, specifically 0%R_7.4%E, 30%R_7.1%E, and 50%R_6.4%E, similarly shaped master curves were observed. However, compared to the 90%R_5.5%E mixture, significantly lower phase angle peak values were noted with their simultaneous shift to the $\log(f_r)$ range of -3 to -4. On the other hand, master curves fitted for mixtures with a 4% BE content (70%R_4%E and 90%R_4%E) and the 70%R_5.7%E mixture did not present a full Gauss shape within the analysed frequency range, reaching the peak at the lowest $\log(f_r)$ values.

Based on the obtained statistical fitting parameters for the phase angle master curves (Table 32), it can be concluded that most of the analysed mixtures exhibited a good agreement between the model and the experimental data. This is confirmed by high values of the R^2 ranging from 0.941 to 0.991 and the values of the S_e/S_y indicator below 0.26. However, for the mixtures 70%R_4%E, 50%R_6.4%E, and 30%R_7.1%E the model fit was less accurate, resulting in with lower R^2 values (ranging from 0.875 to 0.907) and noticeably higher S_e/S_y values (≥ 0.316). This is reflected in the conclusions presented in section 6.2.2.1, where attention was drawn to the variability of phase angle distribution in certain mixtures and its deviation from the typical viscoelastic behaviour pattern. Overall, the results confirm the adequacy of the modelling approach for describing the phase angle behaviour of the tested materials, while indicating the need for careful interpretation in cases where statistical agreement is lower.

To highlight the difference in the viscoelastic response, Figure 64 presents the distribution of dynamic modulus and phase angle values for BSM-E mixtures with the lowest RAP contents (0% and 30%) and BE content (4%), and consequently the lowest total binder content. According to the classification presented in Figure 50, these mixtures were identified as those most likely to exhibit the granular material-like behaviour.

The Black curve (Figure 64 a) reveals a limited range of phase angle values (approximately 8° – 17°), which indicates a relatively small contribution of the viscous component in the materials' mechanical response. The analysed mixtures exhibit relatively low phase angle values, indicating a behaviour that is more elastic than viscoelastic – characteristic for materials with a discontinuous binder structure. Moreover, the data points do not form a clearly defined curve, and their local aggregation of the data as well as the scattered distribution

suggests inconsistent or nonlinear viscoelastic behaviour, likely influenced by the dominant role of the mineral skeleton in the overall mixture response. Subsequently, the Cole–Cole diagram shows an irregular, scattered pattern of points that deviates from the shape typical of viscoelastic materials. The absence of a smooth and continuous curve may also suggest that the energy dissipation mechanism is limited and may be disrupted by the predominant granular structure.

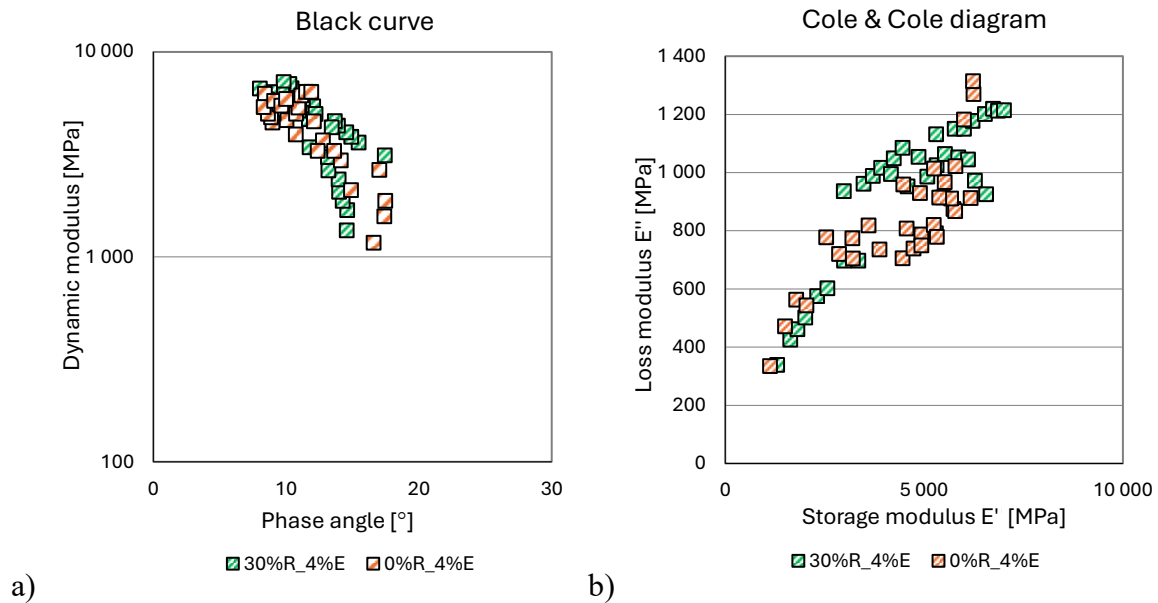


Figure 64 Viscoelasticity assessment of the 0%RAP – 30%RAP mixtures: a) Black curve, b) Cole & Cole diagram.

In summary, the analysis of the Black curve and Cole–Cole diagram for the 0%R_4%E and 30%R_4%E mixtures confirms their limited viscoelastic behaviour, which is consistent with their low total binder content. The scattered distribution of data points and the absence of a coherent curve structure point out that these mixtures are governed by a primarily elastic and granular response, rather than by a uniform viscoelastic mechanism characteristic of traditional asphalt mixtures.

6.3. Discussion on the cohesion-viscoelasticity interrelation

6.3.1. Key insights from cohesion and viscoelasticity analyses

The discussion presented in this chapter aims to address the formulated research questions and to verify the validity of the dissertation thesis statement. The realisation of the main objective of the study – namely, the assessment of cohesion development in BSM-E mixtures over time – allowed for the identification of the key factors influencing the mechanical performance of the material.

The research results show that RAP content played a decisive role in both initial and time-dependent cohesion development of BSM-Es. Low-RAP mixtures exhibited high initial resistance to permanent deformation due to the dominance of frictional forces and interparticle contact within the mineral skeleton. As RAP content increased, the mixtures became more binder-dependent, showing a shift toward viscoelastic behaviour and increased susceptibility to permanent strain accumulation. On the other hand, the amount of BE affected the balance between the internal friction and binder-related cohesion in mixtures with different RAP levels. Lower BE contents were associated with improved deformation resistance across all RAP levels, suggesting that higher amounts of the residual bitumen can reduce the mixture's stiffness and accelerate permanent deformation under loading.

The results also confirmed cohesion evolution under conditions simulating asphalt pavement structures and enabled its quantification through the methodology proposed in this thesis. The cohesion development in BSM-Es was identified as a time-dependent phenomenon, with half of the analysed mixtures exhibiting performance changes, particularly those with high RAP content. The laboratory-simulated conditions can be considered representative of real-world scenarios, following the natural curing process (i.e., evaporation of water from the base layer) and subsequent pavement service life.

Cohesion development was non-linear, with two conditioning phases identified: a short-term phase (0–28 days, including a stable period of 0–14 days) and a long-term phase (90–120 days). The short-term conditioning phase (0–28 days) revealed critical differences between mixtures in terms of the increase in cohesion, enabling the identification of materials prone to further structural evolution. Particularly in high-RAP mixtures, the 28th day marked a turning point, beyond which statistically significant improvements in performance parameters were recorded, suggesting the occurrence of long-term binder interaction processes. Long-term conditioning (beyond 28 days) resulted in a continued cohesion development in selected mixtures, followed by performance stabilisation after approximately 90 days. One of the key findings of this thesis is that, for BSM-Es, the combination of intermediate to high RAP contents (50%–90%) with moderate BE dosages (4%–6.4%) the cohesion development is progressive and improves the long-term mechanical behaviour of the material. Mixtures designed with this balance consistently showed statistically significant improvements in FN and FN index over time. These interactions likely contribute to the formation of an internal structure with a greater capacity to develop continuous bonding, and consequently, to the manifestation of viscoelastic behaviour. This phenomenon, however, was not observed in mixtures with substantially higher BE contents (7.1%–7.4%) and a low RAP share (0%–30%), which ultimately exhibited

viscoelastic behaviour. Therefore, this observation suggests a link between the cohesion evolution, understood as internal structural integrity, and the potential of RAP to consolidate within the pavement structure under long-term pressure from overlying courses and traffic loads.

This interpretation is supported by earlier studies, particularly those of Ebels [91] and Jenkins [82], which indicate that BSMs initially behave like unbound granular materials, with stiffness increasing systematically due to moisture loss and mechanical densification under traffic loading. Over time, this behaviour transitions toward a quasi-viscoelastic response, associated with partial binder activation and structural reconfiguration within the material. Similarly, based on the results of the research carried out by Twagira [90] and Nivedya et al. [20], it can be stated that RAP consolidation is a critical mechanism in the structural development of the layer. The compaction of the mineral skeleton within the pavement layer – combined with the potential for partial rebounding of binder-coated particles – contributes to cohesion development and enhanced resistance to permanent deformation. These findings underline the fact that the material consolidation plays a key role in the BSMs' long-term mechanical performance. In this context, the research conducted in this thesis confirmed previously reported mechanisms of the evolutive behaviour in BSMs and extended current knowledge by introducing a Flow Number-based method to quantify cohesion changes. The proposed procedure enabled the quantification of cohesion development over time and facilitated the identification of threshold material compositions associated with distinct viscoelastic responses.

The conducted analysis of the viscoelastic properties of the selected mixtures, based on the previously defined criteria, confirmed that the mechanical performance of the analysed BSM-Es – although exhibiting a less pronounced viscoelastic character compared to HMA – can nonetheless be effectively described using the time-temperature superposition principle. At lower temperatures, BSM-E mixtures demonstrated notably lower stiffness but similar or slightly higher viscous contributions than AC, based on phase angle values, suggesting differences in stress transfer mechanisms within this material and asphalt mixture. In the temperature range of 20°C to 40°C, all BSM-E mixtures showed a limited transition toward a viscous-dominated behaviour in comparison to the AC mixture.

A simultaneous analysis of both the cohesion and viscoelastic behaviour of the BSM-E mixtures is justified in order to address the question – formulated concurrently as the thesis of this dissertation – regarding the contents of Reclaimed Asphalt Pavement (RAP) and bitumen emulsion (BE) in BSM-Es, for which these materials exhibit linear viscoelastic properties.

In order to confirm the dissertation thesis and determine the RAP and BE contents at which cold recycling mixtures exhibit viscoelastic behaviour while simultaneously meeting the definition of Bitumen Stabilised Materials, it is necessary to consider the practical aspects of applying the analysed materials in pavement structure, as well as to take into account the ranges of RAP and binding agent contents suggested in widely recognised scientific classifications of mixtures produced within the cold recycling technology (see section 2.1.3). In both cases, it is advantageous to maximise the RAP content in the mixture due to economic and environmental reasons – reducing the consumption of virgin aggregates, increasing the utilisation of reclaimed asphalt, and enhancing the potential for in-situ recycling applications, where the share of RAP is typically high. With respect to the bitumen emulsion content, it is favourable to minimise its level in order to obtain economic benefits and ensure adequate compactability of the mixture, while at the same time maintaining a sufficient residual binder content which – as demonstrated in this thesis – has a significant impact on resistance to permanent deformation and on the variability of viscoelastic parameters depending on temperature and loading conditions at constant RAP contents. Moreover, the use of high emulsion contents would bring the cold recycling mixtures closer to cold mix asphalt (CMA) in terms of their compositional characteristics.

Considering the material-related factors and external conditions (cohesion evolution), the following restrictions on RAP and BE contents were established for cold recycling mixtures containing 1% cement. These restrictions were derived from a comprehensive analysis of the results of cohesion and viscoelasticity tests, which enabled a robust interpretation of the material behaviour.

- It can be concluded that mixtures with low RAP contents (0% and 30%) combined with a BE content of 4% should be excluded from the recommendations. These mixtures represent materials whose mechanical performance cannot be described within the framework of the time–temperature superposition principle and, additionally, do not demonstrate cohesion development over time. Furthermore, the mixtures 0%R_7.4%E and 30%R_7.1%E, although exhibiting a viscoelastic response, were also rejected due to their markedly increased emulsion content, which makes them less suitable for practical implementation.
- On the other hand, the viscoelastic response and cohesion development observed over time in mixtures with RAP and BE contents of 70–90% and 4–5.7%, as well as in the mixture containing 50% RAP and 6.4% BE, indicate that these materials may be included in the recommendations. Although initially characterised by lower resistance

to permanent deformation, these mixtures exhibited a favourable increase in FN values over time, which is indicative of the progressive development of cohesion within the material. Such observations support the formulation of material guidelines applicable to the broader group of cold-recycled mixtures, including both BSM and CMA types.

- Within the group of mixtures containing 70–90% RAP, both the compositions with 4% BE and those with an increased BE content (5.5–5.7%) demonstrated viscoelastic behaviour. This finding allows for the proposal of the following material parameter ranges for BSM-E mixtures, in line with the principle of maximising RAP content while simultaneously limiting the emulsion dosage to values classified as appropriate: $\text{RAP} \geq 70\%$ and BE 4–5%, where a 5% bitumen emulsion content corresponds to approximately 3% residual bitumen in the mixture.

However, it should be noted that the established material recommendations and thresholds are specific to the qualitative and quantitative characteristics of the materials examined in this study and require verification on locally sourced materials, with subsequent adjustment to local conditions, as they may not be universally applicable.

6.3.2. Technical and engineering recommendations

As demonstrated by the research results, the unconfined Flow Number test, not included in the design standards for cold recycling mixtures, proved effective for evaluating the time-dependent cohesion development of BSM-E mixtures. The BSM-Es' performance characterisation was possible by applying testing conditions verified in the preliminary research (test temperature of 50°C, 600 kPa repeated axial stress level). It should be also noted that during the initial conditioning period (0–14 days), no statistically significant changes in the cohesion of the analysed mixtures were observed, regardless of the RAP or BE content. This indicates that when considering the use of mixtures with higher RAP and moderate BE contents – where initial resistance to permanent deformation may be noticeably lower – it is advisable to evaluate their properties in Flow Number testing after a minimum of 28 days of conditioning at ambient temperature (20±2°C), due to the potential for cohesion development in time through material consolidation.

Considering the results of viscoelasticity testing, the combination of lower dynamic modulus and similar phase angle values observed in BSM-E mixtures at lower testing temperatures (e.g., 5°C) compared to HMA presents both advantages and limitations, depending on the intended application and performance requirements. On one hand, BSM-E mixtures at low temperatures are characterised with similar to slightly elevated phase angles compared to

AC, which indicates an increased capacity for energy dissipation and may contribute to limiting crack propagation in pavement structures under cyclic loading. On the other hand, the significantly lower modulus – up to four times lower than that of conventional asphalt concrete – may compromise the structural load-bearing capacity of BSM-E mixtures, particularly in high-traffic conditions. Moreover, any further shift toward viscous behaviour could increase the risk of permanent deformation under sustained or repeated loading. Equally noteworthy are the Flow Number test results obtained under the conditions adopted in this dissertation, which indicate that BSM-Es exhibit substantial internal friction compared with conventional asphalt concrete, as evidenced by the FN and the accumulated permanent strain values.

From a practical perspective, the findings of the experimental program show that while BSM-E mixtures do not replicate the full viscoelastic performance of hot mix asphalt, their moderate thermal response and evolving mechanical behaviour support their application in base layers of flexible asphalt pavements. The results confirm that while BSM-Es may not offer the same stiffness and load-bearing performance as conventional asphalt mixtures, they can still maintain stable viscoelastic characteristics. Therefore, they can be effectively used in pavement base layers, particularly where crack resistance and material reuse are prioritised over high modulus requirements. Moreover, properly designed BSM-E mixtures, especially those engineered in terms of RAP and BE content, may offer a balance between the structural support and reduced cracking susceptibility in the road pavement structure.

It should be noted that the performance of BSM-E mixtures in terms of resistance to permanent deformation – which was concluded to be an indicator for assessing whether a material exhibits viscoelastic behaviour – may be influenced by a wide range of material-related factors. These include not only the proportions of the mixture components but also their specific properties. In the case of RAP, important factors include the aggregate gradation, binder content, and the degree of binder ageing. For bitumen emulsion, the base binder type used in its production, the presence of modifiers, and the type of emulsifiers may all affect the potential interaction between the fresh and aged binders. In the case of the remaining components of the mixtures, the origin, gradation, and quality of the virgin aggregates, as well as the characteristics and content of hydraulic binder should be taken into account. Additionally, technological conditions during laboratory specimen preparation and curing, as well as during in-plant mixing and field compaction processes also influence the volumetric and mechanical properties of the mixtures. These include the air void content, which is critical to the development of the internal cohesion and, consequently, the characteristics of the contact zone between the bituminous binder and the mineral skeleton.

The laboratory findings of this thesis provide a robust basis for the formulation of material selection criteria and performance-based mix design methodologies tailored to BSM-E materials. As a subsequent stage of research, inter-laboratory investigations on a wider range of materials, along with full-scale validation on in-service pavement sections, are recommended to account for material variability and the conditions present within actual pavement structures.

7. Conclusions and further research directions

The findings of this dissertation demonstrate that the proposed innovative approach to the BSM-E mixtures' performance investigation, presented in the experimental part and supported by the literature review, enabled the identification of the key material variables influencing the development of the BSM-Es' cohesion – the amounts of RAP and BE. The novelty of the research approach lies in the assumption that a certain level of cohesion – defined as internal structure integrity – must be ensured in order to characterise BSM-E mixtures as viscoelastic materials.

The outcomes of the dissertation include both the development of an original decision tree method – based on a hierarchical assessment of RAP content, cohesion development over time, and BE content – supporting the estimation of the viscoelastic response of BSM-E mixtures, and the implementation of the Flow Number method for the evaluation of cohesion as a function of time and the identification of threshold material factors influencing viscoelastic properties.

The research conducted within this doctoral dissertation has led to the following final conclusions:

1. The material factors – specifically, the content of RAP and bitumen emulsion – have a significant impact on the cohesive performance of BSM-E mixtures. RAP content strongly influences both the initial mechanical behaviour and cohesion development of BSM-E mixtures. Low-RAP mixtures resist deformation mainly through frictional contact, while higher RAP levels result in an increased binder-dependence of the mixture, showing a shift toward viscoelasticity and increased strain accumulation. The amount of bitumen emulsion affects the balance between internal friction and binder-related cohesion, influencing the structural integrity of the BSM-E mixtures.
2. BSM-E mixtures with higher RAP content (50%–90%) and intermediate bitumen emulsion addition (4%–6.4%) demonstrated potential for cohesion evolution over time, despite their higher susceptibility to permanent deformation at the initial stage of testing. Changes in the BSM-Es' permanent deformation resistance followed a non-linear pattern, enabling the analysed period to be divided into short-term and long-term phases, each characterised by distinct levels of the assessment parameters. External conditions within the pavement structure may provide a favourable environment for such cohesion development to occur.

3. The unconfined Flow Number test performed under the conditions of the 600 kPa repeated axial stress level and the test temperature of 50°C was positively verified to quantify the changes in BSM-Es' cohesive performance over time. The proposed evaluation parameters based on the FN and accumulated permanent strain values allowed for the quantitative assessment of the cohesive properties of the material.
4. The FN index can serve as a parameter quantifying the rate of BSM-Es' permanent deformation accumulation, as well as a classification tool for distinguishing mixtures based on their mechanical behaviour (granular vs. viscoelastic) and as a predictor of the material's potential for cohesion development. Specifically, FN index values ≤ 0.050 [%·100] correspond to mixtures characterised by granular material behaviour, whereas values ≥ 0.090 [%·100] are indicative of viscoelastic behaviour accompanied by cohesion development. Values between these threshold limits can be considered transitional with respect to the onset of viscoelastic response and cohesion evolution. However, in these studies, the transition value range for these specific materials was found to be in the vicinity of 0.065 ± 0.005 [%·100].
5. Classification of BSM-E mixtures based on Flow Number test results – as structured in the decision tree diagram proposed in this thesis – considering the material factors (RAP and BE content) and external factors (cohesion development), enables reliable identification of the materials with viscoelastic properties.
6. The mechanical performance of BSM-Es – although exhibiting a less pronounced viscoelastic character compared to asphalt mixtures – can be effectively described using the time–temperature superposition principle.
7. Based on the combined analysis of the Flow Number and Dynamic Modulus test results, the RAP and BE content levels supporting the desired viscoelastic behaviour of BSM-E mixtures can be established. For the BSM-Es analysed in this work, containing 1% of cement, a RAP content threshold of $\geq 70\%$ and a BE content range of 4%–5% are recommended as indicative limits.

Summarising the results of the conducted research, it can be concluded that the formulated dissertation thesis has been confirmed and that the research questions posed in the study have been comprehensively addressed.

The research and analyses carried out in the dissertation have provided a detailed insight into the BSM-E mixtures' initial and long-term cohesive properties and their connection with the predicted viscoelasticity of the material. Nevertheless, the topic remains open to further exploration. As a result, several potential directions for continued research in this area have been identified. It is recommended that comprehensive interlaboratory studies be conducted in order to:

- Develop performance-based mix design methodologies specific to BSM-E materials, which incorporate both cohesion development and viscoelastic property evolution. These should take into account the interactions between aged and fresh binder, as well as the quantitative and qualitative variability of mixture components.
- Further investigate the influence of the key components' characteristics – such as the degree of RAP binder ageing, bitumen emulsion chemistry, aggregate properties, and the type and dosage of hydraulic binders, including alternative binders – on the short-term and long-term cohesive behaviour and mechanical performance of BSM-E mixtures.
- Validate the proposed Flow Number and Dynamic modulus testing conditions across an extended range of BSM-E mixtures designed according to locally applied mix design practices and produced using locally available materials. These studies should aim to assess the repeatability and reproducibility of these methods.
- Extend research to field-scale studies, including long-term conditioning monitoring in laboratory settings and observation of performance evolution in trial pavement sections (e.g., monitoring of the stiffness evolution with yearly FWD testing campaigns). A valuable extension of the research would be the field validation of mixtures with the proposed RAP and BE contents, using CCPR and CIR technologies, to assess the effect of production-stage quality control on the long-term BSM-E performance. Such investigations would allow for validating the laboratory-derived thresholds and performance predictions under realistic construction, environmental, and traffic loading conditions.

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List of Standards

In this dissertation, the titles of standards are cited in the text without detailed information regarding the year of publication, corrigenda, or annexes. These details are provided below in the complete list of standards.

- [S1] EN 13286-7:2004 – *Unbound and hydraulically bound mixtures – Part 7: Cyclic load triaxial test for unbound mixtures.*
- [S2] AASHTO MP 31-22 – *Standard Specification for Performance-Graded Asphalt Binder Using the Multiple Stress Creep Recovery (MSCR) Test.*
- [S3] AASHTO T 180-22 – *Standard Method of Test for Moisture–Density Relations of Soils Using a 4.54-kg (10-lb) Rammer and a 457-mm (18-in.) Drop.*
- [S4] EN 13286-2:2010/AC:2012 – *Unbound and hydraulically bound mixtures – Part 2: Test methods for the determination of the laboratory reference density and water content – Proctor compaction.*
- [S5] AASHTO T 342-22 – *Standard Method of Test for Determining Dynamic Modulus of Hot Mix Asphalt (HMA).*
- [S6] AASHTO TP 62-07 – *Provisional Standard Method of Test for Determining Dynamic Modulus of Hot Mix Asphalt Concrete Mixtures.*
- [S7] AASHTO T 378-22 – *Standard Method of Test for Determining the Dynamic Modulus and Flow Number for Asphalt Mixtures Using the Asphalt Mixture Performance Tester (AMPT).*
- [S8] EN 12697-26:2018 – *Bituminous mixtures – Test methods – Part 26: Stiffness.*
- [S9] ABNT NBR 16505:2016 – *Misturas asfálticas – Resistência à deformação permanente utilizando o ensaio uniaxial de carga repetida.*
- [S10] AASHTO PP 94-19 – *Standard Practice for Preparation of Asphalt Mixture Performance Tester (AMPT) Specimens by Means of the Superpave Gyratory Compactor.*
- [S11] BSI DD 226:1996 – *Method for determining resistance to permanent deformation of bituminous mixtures subject to unconfined dynamic loading.*
- [S12] PN-EN 1097-6:2022-07 – *Badania mechanicznych i fizycznych właściwości kruszyw – Część 6: Oznaczanie gęstości ziaren i nasiąkliwości (in Polish).*
- [S13] PN-EN 12697-1:2020-08 – *Mieszanki mineralno-asfaltowe – Metody badań – Część 1: Zawartość lepiszcza rozpuszczalnego (in Polish).*

- [S14] PN-EN 1426:2015-08 – *Asfalty i lepiszcza asfaltowe – Oznaczanie penetracji stożkiem* (in Polish).
- [S15] PN-EN 1427:2015-08 – *Asfalty i lepiszcza asfaltowe – Oznaczanie temperatury mięknięcia – Metoda Pierścienia i Kuli* (in Polish).
- [S16] PN-EN 13398:2017-12 – *Asfalty i lepiszcza asfaltowe – Oznaczanie odzysku sprężystego asfaltów modyfikowanych* (in Polish).
- [S17] PN-EN 12697-2+A1:2019-12 – *Mieszanki mineralno-asfaltowe – Metody badań – Część 2: Oznaczanie uziarnienia* (in Polish).
- [S18] PN-EN 933-1:2012 – *Badania geometrycznych właściwości kruszyw – Część 1: Oznaczanie składu ziarnowego – Metoda przesiewania* (in Polish).
- [S19] PN-EN 12697-1:2020-08 – *Mieszanki mineralno-asfaltowe – Metody badań – Część 1: Zawartość lepiszcza rozpuszczalnego* (in Polish).
- [S20] PN-EN 12697-30:2019-01 – *Mieszanki mineralno-asfaltowe – Metody badań – Część 30: Przygotowanie próbek za pomocą ubijaka udarowego* (in Polish).
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Appendix 1 – Results of the Modified Proctor Moisture–Density Relationship Test

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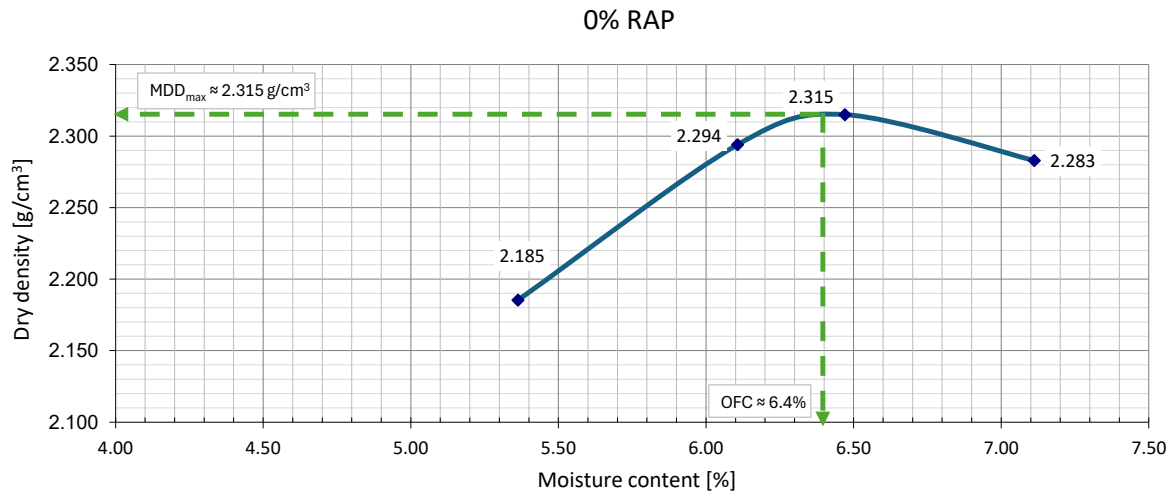


Figure 65 OFC/MDD relationship for 0% RAP mineral mixture

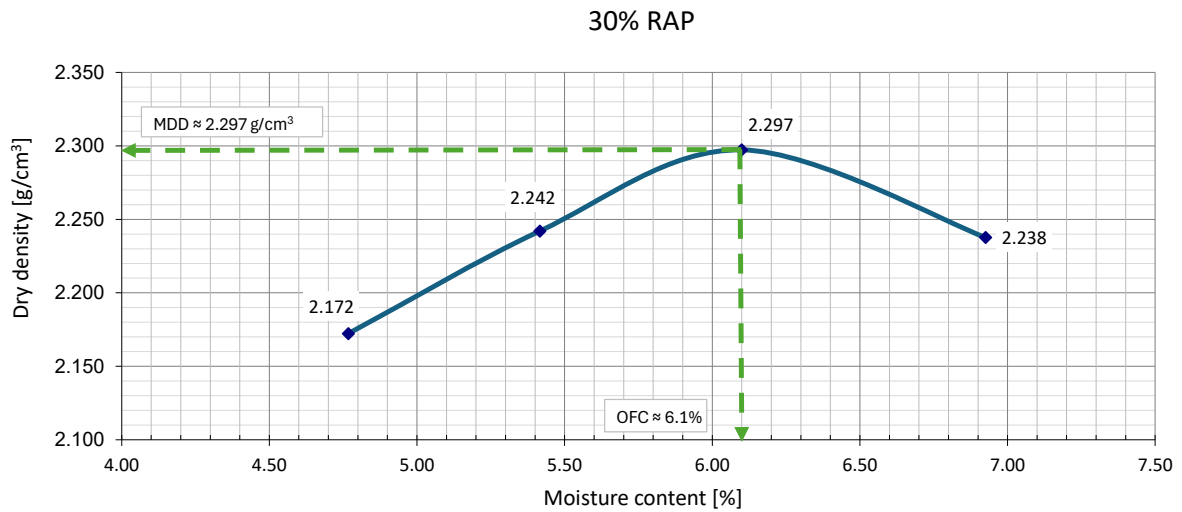


Figure 66 OFC/MDD relationship for 30% RAP mineral mixture

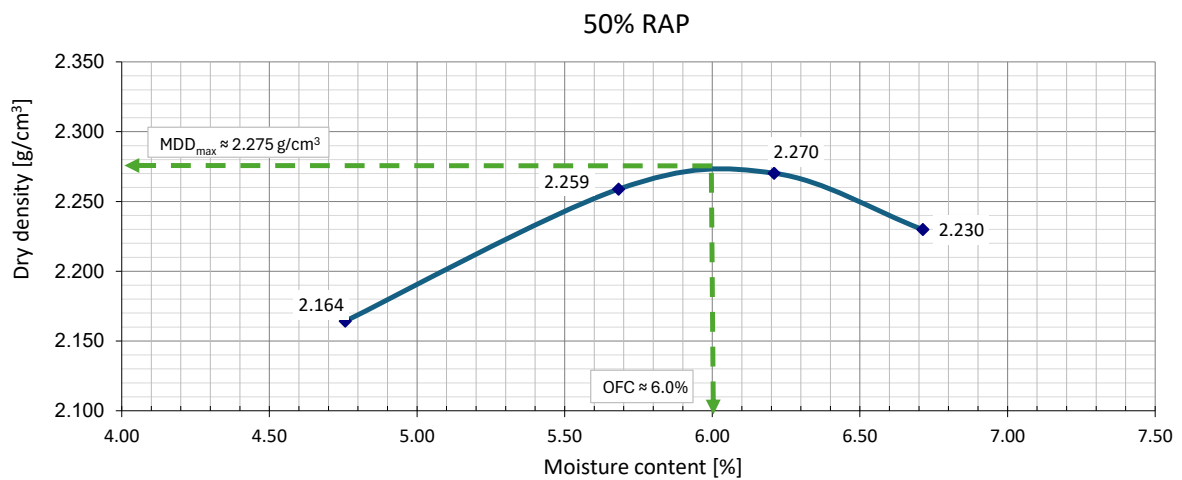


Figure 67 OFC/MDD relationship for 50% RAP mineral mixture

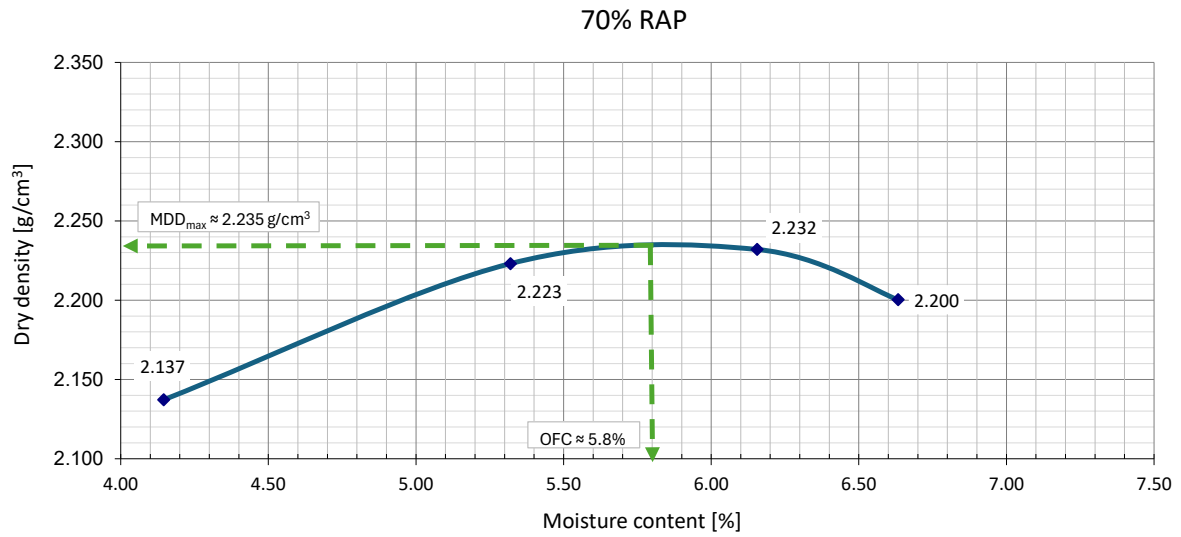


Figure 68 OFC/MDD relationship for 70% RAP mineral mixture

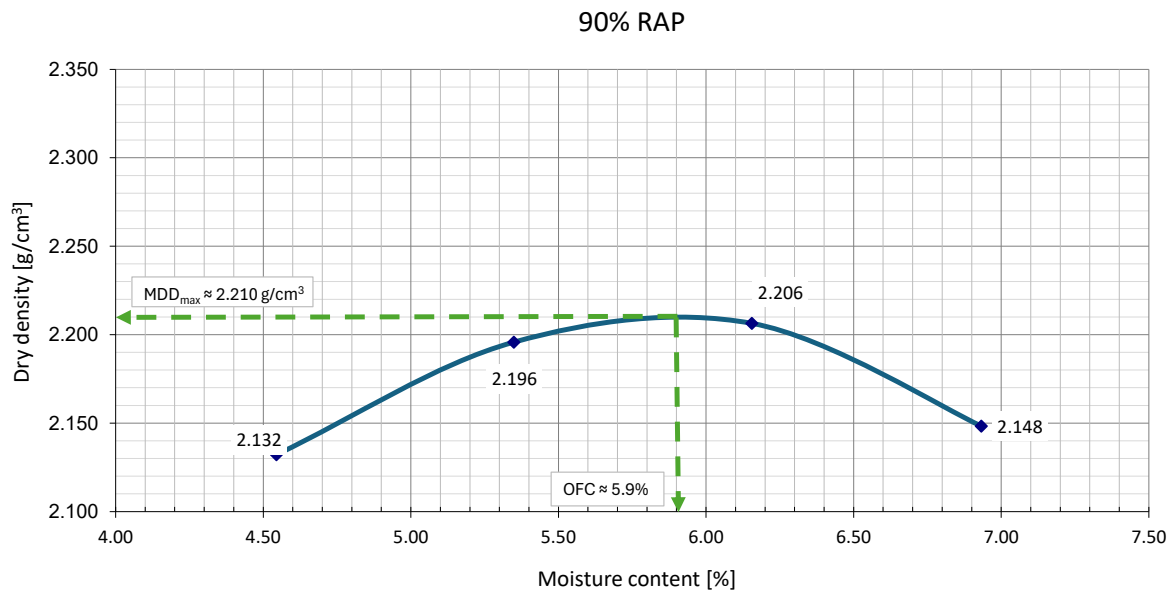


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Table 1 Flow Number test results for 0%R_4%E mixture

Mixture	0%R 4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\varepsilon(\text{FN})$ [%]	$\varepsilon(\text{FN}) - \text{avg}$ [%]	$\varepsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	2760	2907	160	1.00	0.98	0.02	0.036	0.034	0.002
2	0	2884			0.95			0.033		
3	0	3078			0.99			0.032		
4	7	2806	2830	67	0.98	0.97	0.04	0.035	0.034	0.002
5	7	2776			1.00			0.036		
6	7	2906			0.93			0.032		
7	14	2878	2914	75	0.87	0.93	0.06	0.030	0.032	0.002
8	14	2864			0.99			0.035		
9	14	3000			0.92			0.031		
10	28	2807	2863	72	1.03	0.96	0.05	0.037	0.034	0.002
11	28	2957			0.95			0.032		
12	28	2881			0.92			0.032		
13	28	2807			0.95			0.034		
14	90	2798	2947	84	0.96	0.97	0.05	0.034	0.034	0.001
15	90	2790			0.92			0.033		
16	90	2940			1.02			0.035		
17	120	2868	2967	82	1.01	0.95	0.04	0.035	0.032	0.002
18	120	3009			0.91			0.030		
19	120	3055			0.96			0.031		
20	120	2937			0.93			0.032		

Table 2 Flow Number test results for 0%R_7.4%E mixture

Mixture	0%R 7.4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1804	1713	85	1.02	1.03	0.04	0.056	0.060	0.003
2	0	1635			0.99			0.061		
3	0	1701			1.08			0.063		
4	7	1760	1725	61	1.02	1.04	0.05	0.058	0.060	0.005
5	7	1761			1.00			0.057		
6	7	1654			1.10			0.067		
7	14	1678	1770	80	1.09	1.06	0.04	0.065	0.060	0.004
8	14	1822			1.08			0.059		
9	14	1809			1.02			0.056		
10	28	1711	1764	48	1.03	1.04	0.01	0.060	0.059	0.001
11	28	1803			1.04			0.058		
12	28	1778			1.05			0.059		
13	90	1795	1790	60	1.08	1.06	0.05	0.060	0.059	0.005
14	90	1847			1.00			0.054		
15	90	1728			1.09			0.063		
16	120	1883	1802	73	1.09	1.08	0.04	0.058	0.060	0.003
17	120	1743			1.11			0.064		
18	120	1780			1.04			0.058		

Table 3 Flow Number test results for 30%R_4%E mixture

Mixture	30%R 4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	2002	2057	100	1.02	1.00	0.03	0.051	0.049	0.004
2	0	2172			0.96			0.044		
3	0	1997			1.01			0.051		
4	7	2135	2089	49	1.00	1.02	0.02	0.047	0.048	0.002
5	7	2094			1.04			0.050		
6	7	2038			1.03			0.051		
7	14	2128	2065	67	1.05	1.02	0.04	0.049	0.049	0.001
8	14	2073			1.03			0.050		
9	14	1994			0.97			0.049		
10	28	2080	2094	47	1.01	1.00	0.08	0.049	0.048	0.005
11	28	2146			0.92			0.043		
12	28	2056			1.08			0.053		
13	90	2061	2086	76	1.05	1.02	0.04	0.051	0.049	0.002
14	90	2026			0.97			0.048		
15	90	2171			1.04			0.048		
16	120	2048	2102	49	1.00	1.04	0.06	0.049	0.050	0.003
17	120	2116			1.11			0.052		
18	120	2142			1.02			0.048		

Table 4 Flow Number test results for 30%R_7.1%E mixture

Mixture	30%R 7.1%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1541	1637	93	1.12	1.06	0.06	0.073	0.065	0.008
2	0	1644			1.07			0.063		
3	0	1726			0.99			0.057		
4	7	1597	1655	85	1.12	1.12	0.03	0.070	0.068	0.005
5	7	1753			1.10			0.063		
6	7	1615			1.15			0.071		
7	14	1751	1702	82	1.15	1.08	0.07	0.066	0.063	0.002
8	14	1607			1.02			0.063		
9	14	1747			1.07			0.061		
10	28	1684	1685	47	1.10	1.12	0.03	0.065	0.067	0.002
11	28	1672			1.09			0.065		
12	28	1635			1.14			0.070		
13	28	1749			1.16			0.066		
14	90	1660	1705	50	1.06	1.08	0.02	0.064	0.064	0.001
15	90	1759			1.10			0.063		
16	90	1696			1.09			0.064		
17	120	1678	1766	88	1.15	1.12	0.04	0.069	0.063	0.005
18	120	1766			1.13			0.064		
19	120	1854			1.07			0.058		

Table 5 Flow Number test results for 50%R_4%E mixture

Mixture	50%R 4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1531	1641	99	1.10	1.11	0.03	0.072	0.068	0.004
2	0	1723			1.15			0.067		
3	0	1670			1.08			0.065		
4	7	1649	1715	75	1.10	1.10	0.06	0.067	0.064	0.005
5	7	1652			1.13			0.068		
6	7	1791			1.15			0.064		
7	7	1768			1.02			0.058		
8	14	1743	1695	70	1.11	1.12	0.03	0.064	0.066	0.002
9	14	1727			1.15			0.067		
10	14	1615			1.09			0.067		
11	28	1773	1752	73	1.15	1.12	0.02	0.065	0.064	0.003
12	28	1682			1.12			0.067		
13	28	1846			1.11			0.060		
14	28	1707			1.10			0.064		
15	90	1702	1773	63	1.10	1.13	0.03	0.065	0.064	0.001
16	90	1821			1.16			0.064		
17	90	1796			1.13			0.063		
18	120	1869	1796	85	1.22	1.16	0.06	0.065	0.065	0.004
19	120	1702			1.16			0.068		
20	120	1817			1.11			0.061		

Table 6 Flow Number test results for 50%R_6.4%E mixture

Mixture	50%R 6.4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1029	1082	76	1.25	1.24	0.04	0.122	0.114	0.011
2	0	1169			1.20			0.103		
3	0	1047			1.28			0.122		
4	7	1077	1021	49	1.25	1.21	0.03	0.116	0.119	0.002
5	7	996			1.20			0.120		
6	7	990			1.19			0.120		
7	14	1100	1067	39	1.21	1.22	0.04	0.110	0.114	0.006
8	14	1017			1.20			0.118		
9	14	1095			1.19			0.109		
10	14	1056			1.28			0.121		
11	28	1123	1100	27	1.20	1.21	0.02	0.107	0.110	0.005
12	28	1070			1.24			0.116		
13	28	1107			1.20			0.108		
14	90	1160	1207	50	1.15	1.18	0.03	0.099	0.097	0.004
15	90	1260			1.17			0.093		
16	90	1201			1.21			0.101		
17	120	1179	1219	45	1.19	1.17	0.02	0.101	0.096	0.005
18	120	1268			1.15			0.091		
19	120	1209			1.18			0.098		

Table 7 Flow Number test results for 70%R_4%E mixture

Mixture	70%R 4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1613	1739	127	1.22	1.18	0.05	0.076	0.068	0.008
2	0	1739			1.20			0.069		
3	0	1866			1.12			0.060		
4	7	1875	1810	58	1.02	1.10	0.07	0.054	0.061	0.006
5	7	1766			1.16			0.066		
6	7	1789			1.13			0.063		
7	14	1678	1765	79	1.19	1.18	0.02	0.071	0.067	0.004
8	14	1726			1.20			0.070		
9	14	1855			1.18			0.064		
10	14	1802			1.16			0.064		
11	28	1928	1852	72	1.23	1.19	0.03	0.064	0.064	0.001
12	28	1880			1.20			0.064		
13	28	1843			1.17			0.063		
14	28	1758			1.17			0.067		
15	90	2066	2014	61	1.16	1.16	0.02	0.056	0.058	0.002
16	90	2029			1.15			0.057		
17	90	1947			1.18			0.061		
18	120	2027	1992	59	1.17	1.13	0.04	0.058	0.057	0.002
19	120	2025			1.10			0.054		
20	120	1924			1.13			0.059		

Table 8 Flow Number test results for 70%R_5.7%E mixture

Mixture	70%R 5.7%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1313	1310	81	1.27	1.31	0.05	0.097	0.100	0.010
2	0	1228			1.37			0.112		
3	0	1390			1.29			0.092		
4	7	1385	1405	41	1.30	1.28	0.05	0.094	0.091	0.006
5	7	1461			1.26			0.086		
6	7	1409			1.22			0.087		
7	7	1365			1.34			0.098		
8	14	1465	1401	62	1.30	1.34	0.04	0.089	0.096	0.006
9	14	1342			1.35			0.101		
10	14	1397			1.37			0.098		
11	28	1615	1563	58	1.33	1.33	0.02	0.082	0.085	0.003
12	28	1573			1.35			0.086		
13	28	1501			1.32			0.088		
14	90	1796	1795	74	1.30	1.33	0.04	0.072	0.074	0.003
15	90	1868			1.37			0.073		
16	90	1721			1.33			0.077		
17	120	1932	1873	81	1.30	1.34	0.03	0.067	0.071	0.004
18	120	1879			1.36			0.072		
19	120	1757			1.35			0.077		
20	120	1923			1.33			0.069		

Table 9 Flow Number test results for 90%R_4%E mixture

Mixture	90%R 4%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\epsilon(\text{FN})$ [%]	$\epsilon(\text{FN}) - \text{avg}$ [%]	$\epsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	1056	1085	25	1.15	1.17	0.06	0.109	0.108	0.006
2	0	1102			1.12			0.101		
3	0	1096			1.24			0.113		
4	7	1040	1100	61	1.19	1.23	0.04	0.114	0.112	0.004
5	7	1162			1.25			0.108		
6	7	1098			1.26			0.115		
7	14	1191	1151	49	1.22	1.20	0.03	0.102	0.104	0.005
8	14	1097			1.21			0.110		
9	14	1165			1.17			0.100		
10	28	1248	1247	55	1.19	1.21	0.02	0.095	0.097	0.003
11	28	1318			1.24			0.094		
12	28	1239			1.20			0.097		
13	28	1183			1.20			0.101		
14	90	1294	1361	56	1.21	1.20	0.02	0.094	0.088	0.004
15	90	1445			1.22			0.084		
16	90	1334			1.20			0.090		
17	90	1356			1.17			0.086		
18	90	1375			1.20			0.087		
19	120	1429	1404	39	1.18	1.19	0.02	0.083	0.085	0.004
20	120	1359			1.21			0.089		
21	120	1424			1.18			0.083		

Table 10 Flow Number test results for 90%R_5.5%E mixture

Mixture	90%R 5.5%E									
Specimen No.	Conditioning time [days]	FN [cycles]	FN - avg [cycles]	FN – st.dev. [cycles]	$\varepsilon(\text{FN})$ [%]	$\varepsilon(\text{FN}) - \text{avg}$ [%]	$\varepsilon(\text{FN}) - \text{st.dev.}$ [%]	FN index [%·100]	FN index -avg [%·100]	FN index – st.dev. [%·100]
1	0	839	839	91	1.53	1.46	0.07	0.183	0.175	0.015
2	0	809			1.37			0.170		
3	0	962			1.51			0.157		
4	0	745			1.42			0.191		
5	7	780	802	75	1.40	1.44	0.04	0.179	0.181	0.014
6	7	816			1.46			0.179		
7	7	896			1.48			0.165		
8	7	716			1.43			0.200		
9	14	895	853	61	1.47	1.48	0.02	0.164	0.174	0.016
10	14	783			1.51			0.193		
11	14	881			1.47			0.167		
12	28	966	977	41	1.39	1.42	0.03	0.144	0.146	0.007
13	28	1005			1.45			0.144		
14	28	1012			1.42			0.140		
15	28	924			1.43			0.155		
16	90	1032	1115	67	1.38	1.41	0.03	0.134	0.127	0.008
17	90	1055			1.42			0.135		
18	90	1146			1.45			0.127		
19	90	1186			1.40			0.118		
20	90	1155			1.39			0.120		
21	120	1176	1137	49	1.39	1.41	0.03	0.118	0.124	0.008
22	120	1077			1.42			0.132		
23	120	1179			1.38			0.117		
24	120	1116			1.44			0.129		