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ABSTRACT OF THE DISSERTATION

„Development of technology of obtaining pressed cosmetics of increased hardness"

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In 2014, Bell sp. z o. o. started the production of powder cosmetics using back- and side-injection technology. Both technologies are based on suspensions, made of a powder base, a binder, and a solvent. The stage of the technology is pressing under pressure with simultaneous partial removal of the solvent by the filter tape. The residual solvent is then evaporated over a specified period. Transferring a powder bulk to the form of a suspension eliminates most of the formulation limitations present in standard pressing methods under pressure. It permits obtaining multi-colored pressed cosmetics in various forms and application effects.

New technology to produce cosmetics at Bell sp. z o. o. was developed as part of the doctoral thesis. The research aimed to solve the existing production problems and eliminate isododecane. Suspensions with isododecane present several issues during the production process: difficult transport of the suspension through lines supplying to the die and its high adhesion to the filter tape, making it hard to form. Following the principles of green chemistry, isododecane used so far has been substituted with water. Replacing the flammable isododecane with water made it possible to shorten the drying process of the moulding significantly.

All work on developing the new technology has been studied using comparative tests of the commercial pressed product from Bell's portfolio. Work on new cosmetic formulations (with water as a solvent) was carried out by examining the impact of raw materials (a thickener, fillers, oils) on the application, sensory and performance properties of the product obtained by injection methods. From 50 developed formulations, the recipe was selected to optimize the drying process parameters of moldings at 110–150 ºC. It was found that changes in the amount of thickener significantly affect the rheological properties of the tested suspensions. The brittleness of cosmetics decreases with the increasing content of magnesium-aluminum silicate. Modifications of the powder base and the binder are crucial

in achieving the final product's specific application and sensory properties. The best application was obtained using fillers of various shapes (platelets talc and mica, spherical silicas and nylon, and hemispherical or horseshoe-shaped tapioca starch). Using oils with different partition coefficients (castor oil and isononyl isononanoate) improves the coverage of the cosmetic.

Optimization of the drying process of the prepared samples was carried out, which led to the development of industrial conditions for "baking" cosmetics. At this stage of the research, the drying parameters were changed: temperature and time, as well as the composition of the formulation: functional fillers were introduced, the thickener magnesium-aluminum silicate was replaced with the xanthan gum, the amount of solvent was reduced to achieve greater drying efficiency of moldings. The best results were obtained when the moldings containing the xanthan gum (0.15 % by weight) and the corn starch (8.82 % by weight) were dried at 115 °C for 45 minutes. The improvement of application properties results from greater porosity and better water binding by the mixture of the xanthan gum and the corn starch. These raw materials interact due to chemical interactions or the entangling of their polymer networks.

In the next research stage, a mixture of water and ethanol in various proportions was used in the solvent phase to accelerate the solvent evaporation process and lower its temperature. The best results were obtained using a mixture of water and ethanol in an equal ratio, related to the durability of the hydrogen bonds between these components. In addition, the greater surface roughness of the product, demonstrated by profilometry, confirmed the effect of ethanol on the deformation of starch granules, which increased the mechanical strength and the hardness of the samples by entangling ingredients during pressing.

They selected suspensions and samples after key formulation changes were compared. The developed cosmetic suspensions exhibit the characteristics of shear-thinned liquids with thixotropic properties and tend to aggregate. The suspensions containing xanthan gum are more rheologically stable. On the other hand, ethanol increases the thixotropic effect, which is important in mass storage. A method for testing the affinity of the samples to the skin using *n*-octanol has been proposed. The results were related to their application properties. The hardness and Young's modulus of the samples were tested using nanointendance. It was found that the thickener content in the formulation has the greatest impact on these parameters, affecting the plastic deformation capacity of the samples. With the increasing content of the thickener in the formulation, the structure of the samples was more compact, which was confirmed by SEM images.

The formulation developed as part of the work allowed us to obtain, as a result of drying in optimized conditions (115 °C for 45 minutes), a hard and non-brittle sample with good application and sensory

properties. A large-scale technological test production was carried out. The suspensions obtained in production conditions are more rheologically stable due to a more effective mixing process that improves the powder base's wettability. The samples from the trial production were assessed as non-brittle, so the performance properties were improved compared to the samples obtained on a laboratory scale. Using micro CT, it was found that samples obtained on a laboratory scale are characterized by greater porosity due to the difference between the applied pressing parameters. A problem concerning the unreproducible moldings obtained on a large scale was pointed out. Based on the test production, some of the samples were found to have shrinkage, which resulted in their non-adhesion to the metal plates. The heating chamber must be checked to optimize the drying process.

Rheological tests, SEM imaging, micro CT and hardness tests with surface analysis using a profilometer were performed, and the application, sensory and performance properties were assessed. The last step was to compare the suspensions and samples obtained in the test production with the initial product with isododecane. The suspension obtained from the formulation developed as part of the doctoral thesis was characterized by favorable rheological parameters in the production process, facilitating the mass flow and injection. The hardness and brittleness of the final product were improved by replacing the isododecane to the mixture of water and ethanol.

The result of the research was the development of innovative technology in the company scale of obtaining pressed cosmetics with increased hardness. The formulation with the xanthan gum and the corn starch in a mixture of water and ethanol in a 50:50 ratio allows products with increased hardness, reduced brittleness and preserved application and sensory properties compared to the products in which isododecane was used. The developed technology shortened the production process to 45 minutes.

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