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"Effect of formulation ingredients on compacted powder properties"

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1. Introduction

The make-up segment is one of the most dynamic cosmetics industries. In this category, which includes waxy, powdery and emulsified products, compact powders like eyeshadows, blushes or complexion powders are growth drivers. Compact powders are packaged in boxes containing a single shade (solo), two shades (duo), or a palette of diversified shades. They are widely used because they are nomadic products that are easy to carry and use. They are applied on the face with the help of a powder puff [1]. In foundation or eye makeup, these colored powders are increasingly sophisticated formulations that are difficult to master: their adherence to the skin promises long-lasting wear. They can provide a matte finish (foundation) or shine (eye shadow), coverage and glide, and cosmetic efficiency thanks to active substances in the formulation. The compaction process involves several stages to obtain compacts with an perfect visual appearance, meaning that the surface of the compacts must be smooth and free from delamination, uniform in color and free from residual grain. The process must also result in compacts strong enough to resist to transport and falls, while keeping their end-use properties.

The formulations contain several ingredients : (i) thinners (mica, talc, synthetic fluorphlogopite, kaolin, zea mays starch, cellulose…);(ii) texturising agents (silica, nylon, lauroyl lysine, boron nitrite, alumina, magnesium myristate…); (iii) binders (dimethicone, octyldodecyl stearoyl stearate, glycerin, phenyl trimethicone, diisostearyl malate, caprylic/capric triglyceride, ethylhexyl palmitate, squalane, octyldodecanol…), including other emollients, such as oils and vegetable butters specific to certain brands; (iv) actives (hyaluronic acid, tocopheryl acetate,

vegetable extracts, …) and pigments (titanium dioxide, iron oxides,…). In general, thinners account for the majority of the formulation (70-80%), followed by texturising agents (2-8%), binders (2-4%), pigments (3-5%), active ingredients (1-2%) and preservatives (<1%) [1]. The composition and ratios of these ingredients in the formulation vary according to the desired claims for the product, such as providing glow, light, or illumination, offering softness, comfort, and long-lasting effects. This may be related to the inherent properties of the raw materials which exert an influence on the final product [2]. Although this process is widely used in the cosmetics industry, the development and production conditions are typically based on a very empirical approach, without an in-depth study on the effect of ingredients on the final product [3,4]. The impact of powder characteristics on the sensory profile of emulsions [5] and, on the feeling after the application on the skin of a suspension containing several cosmetic powders have been studied [6]. The effect of different emollients on sensory, physicochemical, and biometrological properties has already been demonstrated in the case of emulsions [7]. However, only two studies have demonstrated the influence of raw material flow properties, such as flow energy and compressibility, on the characteristics of cosmetic powder formulations [8,9]. This project aims investigating the influence of the physical properties of talcs, with and without surface treatment, on compact performance.

2. Materials and Methods

2.1 Materials

Twenty-two commercial cosmetic talc samples were selected : ten samples correspond to talc with surface non-treated (Talc 1 to Talc 10), eleven samples with surface-treated to achieve a better long-wearing make-up (Talc 11 to Talc 21) and one sample with a surface treated with a sun filter (Talc 22). Silica, boron nitride and bis-ethylhexyl hydroxymethoxy benzylmalonate were obtained from Merck. Red, yellow and brown iron oxide were supplied by Sensient. Magnesium stearate was purchased from Acros and octyldodecanol from BASF.

2.2 Formulation and compaction process

The formulation with the different talcs was prepared in order to study the impact of talc on the properties of the compacts (Table 1). The powders were pre-homogenized in a mortar and subsequently transferred to a blade mixer, where they were vigorously mixed for 10 seconds, three times. Then, the binder and antioxidant was added to the powder mixture and blended for 20 seconds after the addition of each ingredient. Finally, the complete formulation was mixed twice for 20 seconds. The compacted powders were obtained by compacting 5g of powders contained in buckets using a manual compacting machine. In order to select the compaction pressure, preliminary tests were carried out with formulations containing Talc 1 using different compaction pressures: 8, 17, 25, 33, 42 and 50 bar.

Table 1. Compact formulation to study the impact of the talcs.

INCI name	% (w/w)
Talc (1 to 22)	70.0
Silica	15.0
Boron Nitride	10.0
Magnesium stearate	1.0
Iron oxide mixture	1.6
Octyldodecanol	1.4
Bis-ethylhexyl hydroxymetoxy benzylmalonate	1.0

2.2 Physical characterization of talcs

2.2.1 Particle size distribution

Particle size analysis were carried out using a system for particle size and size distribution by laser diffraction. A sufficient amount of powders was introduced to obtain an obscuration signal between 1 and 30 %. Each analysis was performed twice with an Aero S accessory in order to check the reproducibility of measurements. The D50 is the mean diameter that indicates the size below which 50% of total particles volume was found.

2.2.2 Specific surface

Specific surface measures of talcs were determined by gas adsorption using the Brunau-er-Emmett-Teller (B.E.T.) equation. These measurements were carried out using a surface and porosity analyser. Before testing, the powder samples were degassed for 3 h.

2.2.3 Flow properties

The flow properties of talcs were evaluated by compressibility and degree of flowability measurements using a powder analyzer based on methods developed by Ralph L. Carr (Carr Indices)[10]. The degree of flowability is defined as the movement of a powder from a stationary to a moving state characterizes the fluidity of the powder.The description used depends on the score obtained with the various parameters (compressibility, angle of repose, angle of spatula, uniformity and cohesion). The compressibility is determined by the relative measurement of loose and packed bulk density.

2.3 Compacts characterization

2.3.1 Colour

The color parameters of the compacts was carried out using a chromameter which gives the values of the L*, a* and b* coordinates of a colour. The colour difference between two products was then assessed by calculating the ΔE* using the equation below. All color analyses were carried out in a booth with a D65 light source.

$$\Delta E^* = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$$

Where : L* represents the lightness (0 black →100 reference white), a* represents the value on a green → red axis and b* represents the value on a blue → yellow axis.

2.3.2 Hardness

The hardness was determined with a texturometer analyzer. For this measurement a conical probe with a 90° angle was used to penetrate 1 mm into the compact at a speed of 0.5 mm.s-

1. A graph showing the force measured as a function of the distance travelled is then obtained. This gives the maximum force required for the probe to penetrate the compact, and therefore the hardness of the compact.

2.3.3 Cake strength

The shock resistance performance was adapted from the method designed by Ogami et al [9]. The pressed powder was dropped from a controlled height of 30 cm, then the damage to the pressed powder was assessed by measuring the loss in mass.

3. Results

The results of talc and compact characterization are shown in Figures 1, 2 and 3. The results were combined to find correlations between finished product properties and physical characteristics of talc. Figure 1 shows the hardness and mass loss of compacts produced with Talc 1 (CPTalc1) at different compaction pressures. As expected , it can be observed that the hardness increases linearly with the compaction pressure over the range of applied pressures. Conversely, the mass loss decreases considerably with increasing compaction pressure from 8 to 17 bar, and then remains practically unchanged for pressures ranging between 17 and 50 bar. Based on these results, compaction pressure at 17 bar was selected to study the effect of talc on the properties of the compacts.

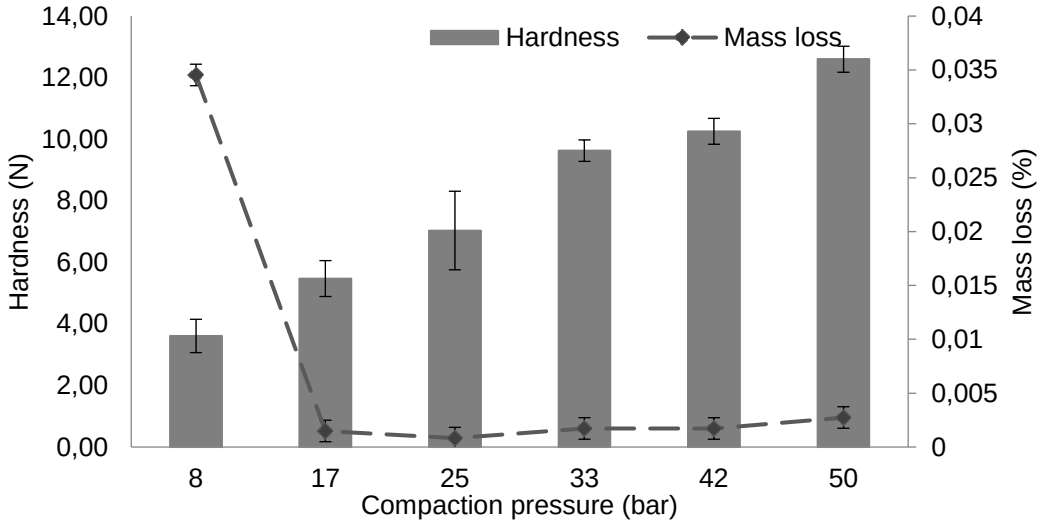

Figure 1. Evolution of hardness and mass loss (obtained after cake strength test) as a function of the compaction pressure applied to compacts made with Talc 1 (CPTalc 1).

Figure 2 illustrates the compaction hardness results obtained with non-treated surface and surface-treated talcs at a compaction pressure of 17 bar. The most stricking result is that he hardness of the compact varies according to the talc used, particularly for compacts obtained with surface-treated talcs (11, 15 and 22) comparatively to the reference compact (CPTalc1). Compressibilities values of talcs ranged from 34% to 53%, but without disclosing any definite impact on compact hardness (Figure 2). These results, combined with other flow parameters, allowed us classifying talcs as having "not good" or "bad" degree of flowability (results not shown). Regarding the results of the cake strength test, all talcs exhibited mass loss values very similar to those obtained with CPTalc 1 at a compaction pressure at 17 bar.

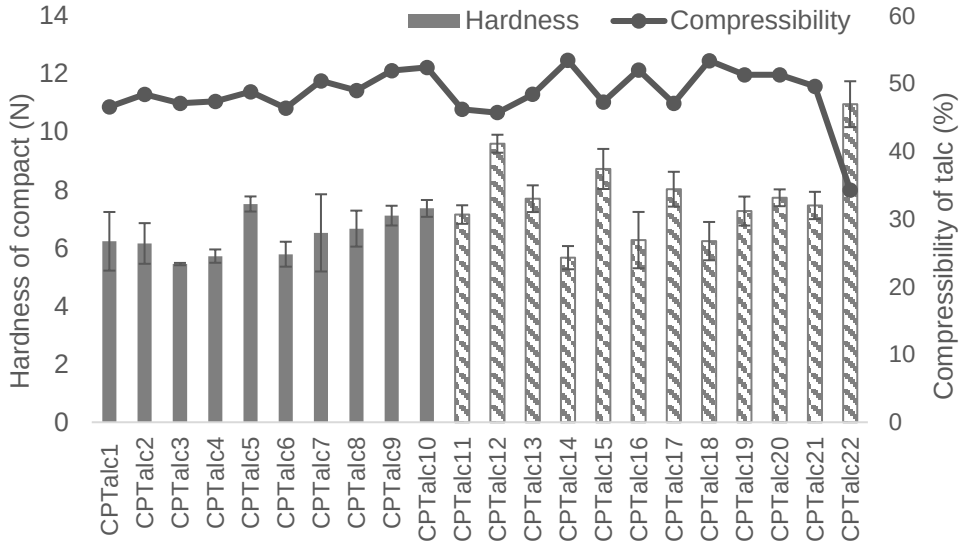

Figure 2. Hardness of compacts produced at 17 bar with surface non-treated (solid filled) and surface-treated (hatched filled) talcs as a function of their respective talc compressibility.

ΔE values obtained from the L*, a* and b* measurements of the different compacts, comparatively to the reference (CPTalc 1, reference), are illustrated in Figure 3 and visualy on the pictures on figure 4. According to these analysis, the ΔE deviations are linked to the differences in lightness given by L* values. For surface-treated talcs, a clear trend was observed: L* values decreased as D50 increased, and a* and b* values increased (resultats not shown), thus increasing ΔE, except for CPTalc 22. However, this behavior was not so clear for compacts obtained with untreated talcs, even though the results of CPTalc 9 and 10 appear

to follow it. Talc specific surface were measured and varied between 2 and 18 $m^2.g^{-1}$. No definite impact of the talc porosity was observed on the properties of the compacts (hardness, colour).

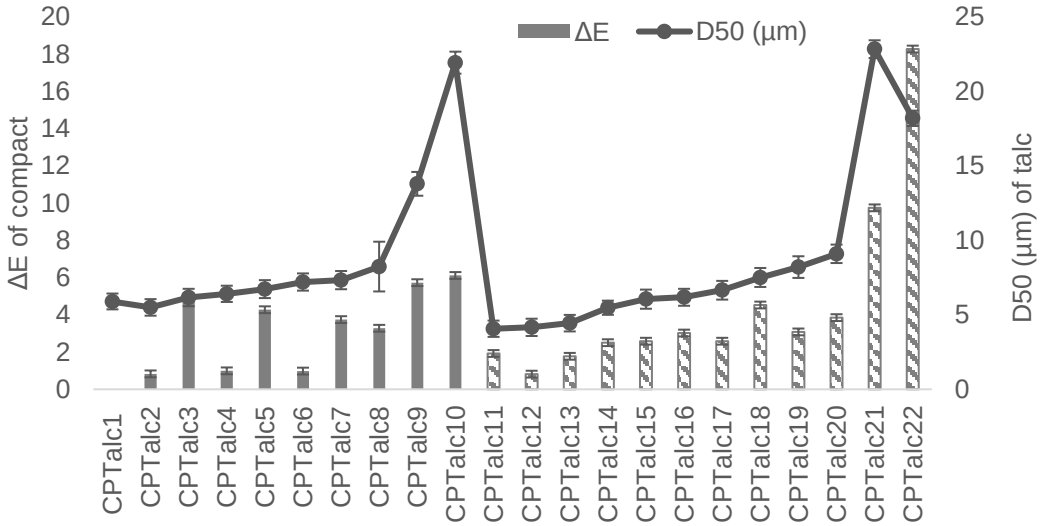

Figure 3. ΔE of compacts produced with surface non-treated (solid filled) and surface-treated (hatched filled) talcs as a function of their respective talc mean diameter. ΔE calculed using CPTalc 1 as reference. Compaction pressure: 17 bar.

Figure 4. Images of compacts produced with talcs 1, 9 and 22.

4. Discussion

The results presented, in this study, highlight the importance of selecting the appropriate ingredients and process parameters. The choice of compaction pressure can have an impact on hardness and compact resistance (Figure 1). This is in accordance with existing pharmaceutical literature, which shows that tablet hardness increases with compaction force, while friability decreases. Furthermore, for the same compaction pressure, differences in particle size, flow properties and surface treatement of talcs may also modify the technical performance of compacts, in particular color and hardness (Figures 2, 3 and 4), and therefore

impact their sensory profiles. In general, the smaller the particle size of the talc, the smoother the size and the higher the coverage [12].

Powder compaction is a complex process involving several consecutive stages: particle rearrangement, elastic then plastic deformation, and finally increased adhesion between particles. The progressive increase in applied pressure plays a crucial role in the evolution of compact density. Initially, pressure favors particle reorganization to fill empty spaces; at higher pressures, it causes plastic deformation of the particles, increasing contact areas and improving the mechanical strength of the final compact [13]. The differences in compaction hardness values may be explained by a better understanding of the behavior of treated and untreated talc and formulation during these stages of the compaction process.

A direct correlation of powder flow properties on the performance characteristics of foundation powders such as cake strength and pay-off was demonstrated [8]. These authors established that higher compressibility has the better cake strength, and lower total energy has better pay-off. In their study, talc had the highest compressibility and lowest flow energy, compared with others ingredients, and formulation with highest concentration of talc presented the best technical perfomance. The same research group investigated the flowability and compressibility of powder formulations containing talc, fumed silica or corn starch, and their impact on the colour of the final formulation [9]. The results showed that the concentration of these ingredients influences the technical performance of the formulations. Physical observation of colour showed a variation between samples containing talc or corn starch (similar color) compared to those containing fumed silica (very white). This was justified by the extremely low density of fumed silica and a significantly lower pigment volume ratio than formulations containing talc or corn starch.

In this present study, it was possible to highlight some trends regarding the physical properties of talcs and compacts performances. However, this work demands more investigations to establish a direct correlation between these parameters.

5. Conclusion

This study underscores the critical importance of ingredient selection and process parameter optimization in the successful manufacturing of high-quality powder compacts. Although trends were observed, no direct correlation could be firmly established between the physical properties of talc—such as particle size, flowability, and surface treatment—and the final technical performance of the compacts. These findings highlight the complexity of powder behavior during compaction and the need for further investigation to better understand the relationships between physicochemical properties of raw materials and the final characteristics of the compacts.

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