IFSCC 32nd Congress

A sustainable and functional alternative to microplastics in cosmetic applications

Hordy, Nathan[1]; Jacques, Cindy[1]; Trigiani, Alexandria[1]; Fabre, Clementine[1]; Fields, Howard[1]; Jones, Rhian[2]; Hollingsworth, Angela[2]; Nayyir-Mazhir, Afshan[2]; Ong, Jaemi[3]; Chavan, Bhaven[2]

[1] Anomera Inc, Quebec, Canada

[2] Croda Europe Ltd, England, United Kingdom

[3]Croda Inc, New Jersey, USA

*Nathan Hordy, 805-460 rue Sainte-Catherine Ouest, Montreal, QC, H3B 1A7, Canada, nhordy@anomera.ca, 514-845-4444

Abstract

Consumer and regulatory pressure are driving the removal of microplastics from cosmetic products around the world. For cosmetic manufacturers to make the transition to natural alternatives, performance needs to be at the same level between these eco-friendly ingredients and the microplastics they are replacing. In this study we compare the optical effects and sensory benefits of cellulose powders to Polymethyl Methacrylate and Nylon microbeads to quantitatively demonstrate that biodegradable spheroidal cellulose powders, ChromaPur CV2 and CV7, can not only perform as good as microplastics, but in some cases outperform these harmful ingredients in skin care and colour cosmetic applications.

Keywords: Cellulose; biodegradable; sustainable; soft focus; microplastics, sensorial powder.

1. Introduction

Plastic pollution is a well-known worldwide environmental issue. There have been initiatives recently from numerous countries to limit the uses of plastics and reduce plastic pollution. Legislation to ban plastic microbeads in “rinse-off” personal care products has been enacted in several jurisdictions, beginning with the United States in 2015. However, these bans didn’t include plastics regulation in “leave-on” products, such as make-up and skin care. Due to the small size of the particles, a single cosmetic product can contain over 1 billion plastic microbeads. As part of the global initiative to reduce microplastics in the environment, the European Chemical Agency (ECHa) is also restricting the use of microplastics in cosmetic products. The proposed regulation which is expected to be adopted by the end of 2022 will include, this time, a ban on plastic microbeads in leave-on products. Currently there is a proposal for a 6-year grace period between adoption and enforcement of such a ban [1].

Consumers have become more attentive to composition, sourcing and labels in the food and fashion industry. Now we see the same trend for the beauty market, where consumers are preferring eco-friendly and sustainable ingredients. Moving towards sustainable and transparent practices across the whole production cycle is now expected and customers want brands that align with their own individual morals and values.

To address this market need, ChromaPur CV2 and ChromaPur CV7 have been developed. They are two spheroidal cellulose powders offering an excellent alternative to microplastics for sensory and optical benefits in skin care and colour cosmetic applications. ChromaPur CV is 100% natural, vegan suitable, and derived from upcycled black spruce from Canada. These trees are sustainably harvested and sourced from 100% Forest Stewardship Council Certified Canadian Forests and all trees that are harvested are replaced. The ChromaPur CV product is then produced via an environmentally friendly and patented process that maintains native Cellulose, type 1, the same cellulose which occurs in nature (Figure 1).

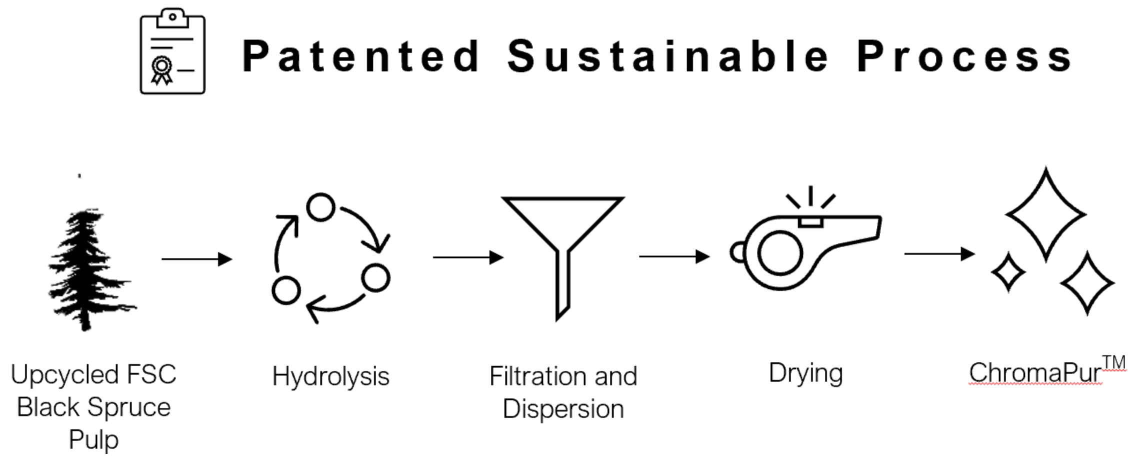

Figure 1: Process flow diagram for ChromaPur CV

ChromaPur CV2 and CV7 are produced in a sustainable manner, resulting in minimal pollution. This is achieved using solely renewable and hydroelectric power, resulting in negligible CO_2 emissions and zero solid waste. The only waste steam created is 100% aqueous containing dilute sugars and weak acids, which is treated on-site and released back into the environment as clean water. As a comparison to microplastics, ChromaPur CV is readily biodegradable according to OECD 301 F protocols.

As the cosmetic industry moves towards sustainable ingredients, the need for natural and biodegradable alternatives to microplastics is becoming critical. In the past, the natural sensorial powders available have not been able to meet the skin care and colour cosmetics market need, particularly for specialized benefits such as wrinkle and pore minimization. This study will compare optical effects and sensory benefits of ChromaPur CV2 and CV7 to three plastic microbeads commonly used in cosmetics (PMMA, Nylon-6 and Nylon-12) and Cellulose Fibers, to quantitatively demonstrate that biodegradable and eco-friendly ingredients can not only perform as good as microplastics, but in some cases outperform these harmful materials.

2. Materials and Method

2.1 Materials

Five sensorial powders were examined as part of the study. ChromaPur CV2 and CV7 (also referred to as CV2 and CV7) were produced in Témiscaming, Quebec, Canada. Both materials are produced from 100% FSC certified trees from Canadian forests and are readily biodegradable as per OECD 301-F protocols (INCI cellulose). The other cosmetic sensorial powders were obtained from commercially available sources. The tradenames of these powders will not be disclosed and will only be described in this study as PMMA, Nylon-6, Nylon-12 and Cellulose Fibers (C-Fibers). The INCI designation of the powders are Polymethyl Methacrylate, Nylon-6, Nylon-12 and Cellulose, respectively. The surface chemistry of a sensory powder is very important determining the compatibility with formulation type for emulsion and water-based applications. If a powder is not compatible with the formulation type (such as Nylon-12 in O/W or anhydrous formulations), then the dispersion in the formulation will be low leading to poor performance. Therefore, to be as aligned as possible with the industry uses of the various powders, Nylon-6 was used in the O/W formulations tested as part of this study and Nylon-12 was used in the lipstick formulations (i.e., anhydrous formulations).

2.2 Characterization

The size of the different powders was determined using a Mastersizer 3000 laser diffraction particle size analyzer (Malvern Panalytical). The morphology of the samples was observed with scanning electron microscopy (SEM) (FEI Quanta 450 SEM with a platinum coating).

The Refractive index of each powder was determined by the immersion method. The powders were dispersed in oils of known refractive index and observed under a microscope. When the dispersion appears transparent, this means refractive index of the powder matches the refractive index of the oil, thus indicating the refractive index of the powder.

Oil absorption was measured based on ISO 787-5:1980 with Ricinus Communis (Castor) Seed Oil. 1.0 g of powder material was placed on a dry and clean watch glass. The oil was added in a slow dropwise fashion. After each addition, the oil was rubbed into the powder, addition of oil was continued at this rate until a conglomerate of oil and powder was formed. Oil addition ceased once the powder formed a 'play-doh' like ball (spreads without cracking or crumbling and only just adhering to the plate). The quantity of consumed oil was recorded, and the percent oil absorption value was calculated.

Optical effect:

Soft focus effect was observed on NO.10I cheek skin model Bioskin® Plate (Beaulax, Japan) that mimics a 50-year-old skin relief. Bioskin® was treated with 2.5mg/cm² film of O/W emulsions containing 5% w/w of powder added to water phase representing in total 76% of the formula, the remaining 24% representing oil phase. Images were taken after 20 min of drying at room temperature using the Handheld Digital Microscope Pro (Celestron) with light on and set up in a light box with controlled condition, using the Software MicroCapture Pro (Celestron). All images were taken under the same condition.

Reduction of pores of the treated NO.10I cheek skin model Bioskin® Plates were analyzed using cross-polarized VISIA CR-1 in the standard 1 lighting mode (Canfield Scientific, Inc.) and analyzed through the Line & Pore Tracking Software App. A region of interest (ROI) was drawn in the center of the Bioskin® image and replicated on each image. The pores were detected and quantified in terms of density (total area taken up by the pores), perimeter (total perimeter of all the pores) and object (total number of pores).

The lipsticks were prepared with 5% w/w powder and containing 5% w/w of untreated pigments. The general formula contained approximately 15% of a blend of waxes and 75% oils selected to get the required texture. The colour intensity was evaluated by applying 3 layers of lipstick (draw down) to the clean and dry inner forearm of a volunteer. Application was conducted as a quadrant (2x2 chart) on the forearm to compare two lipsticks at a time. At the end of each quadrant color intensity test, a picture was taken of the volunteer's forearm and color intensity was evaluated visually. Lipsticks were also tested on lips by applying 3 layers and pictures were taken 5 minutes after the application. All forearm and lip images were taken under the same conditions.

Sensory:

A blind and randomized comparison test to evaluate the sensory properties of the powders in formulation was performed by a four-person internal panel to ensure an unbiased assessment. Tests were conducted with an O/W emulsion with 3% w/w of powder added to water phase representing about 86% of the total formulation. The remaining 14% representing the oil phase. The formulation was applied on clean forearm using a controlled quantity and application protocol. Two emulsions were compared side by side. For each pair, the panelist gave a mark for each attribute (1-the most, 0-the least, 0.5-no difference). Statistical analysis was conducted on the results using ANOVA at a 95% confidence level. This methodology was used in order provide the most precise and quantitative data to conduct statistical analysis using the ANOVA methodology.

3. Results

3.1 Physical characterization

Various physical characterization techniques were used to study the 5 sensorial powders under investigation. The results are summarized in Table 1. Particle size was measured using laser diffraction and morphology was confirmed using scanning electron microscopy. Refractive index and oil-up take were also measured as these are known to be key parameters that can affect the performance of a sensorial powder once formulated. Figure 2 shows the spheroidal shape of ChromaPur CV2 and CV7. The particle size distribution of the CV7 was found to be relatively broad, while the CV2 has a large proportion of particles below 1 µm. The Cellulose Fibers used in this study were shown to be long (10-70 µm) whereas the PMMA plastic microbeads have a similar size distribution to ChromaPur CV7. Nylon-12 microbeads were larger with a broader distribution than the Nylon-6 microbeads. Both Nylon powders are spheroidal in morphology. The micrographs of the other sensorial powders are not shown as to protect the commercial identities of the product used. The refractive index of the cellulose powders and Nylons were significantly higher than of the PMMA microbeads.

Table 1: Summary of the physical characterization of the sensorial powders.

	ChromaPur CV2	ChromaPur CV7	Cellulose Fibers	PMMA	Nylon-6	Nylon-12
Morphology	spheroidal	spheroidal	fibrous	spherical	spherical	spherical
Particle size D50 (µm)	2	7	30	6	12	5
Particle size D90 (µm)	5	15	72	11	20	7
Particle size D10 (µm)	0.4	4	12	4	6	3
Refractive Index	1.54	1.54	1.54	1.49	1.54	1.53
Oil up-take (mL/100g)	62	56	142	46	112	127

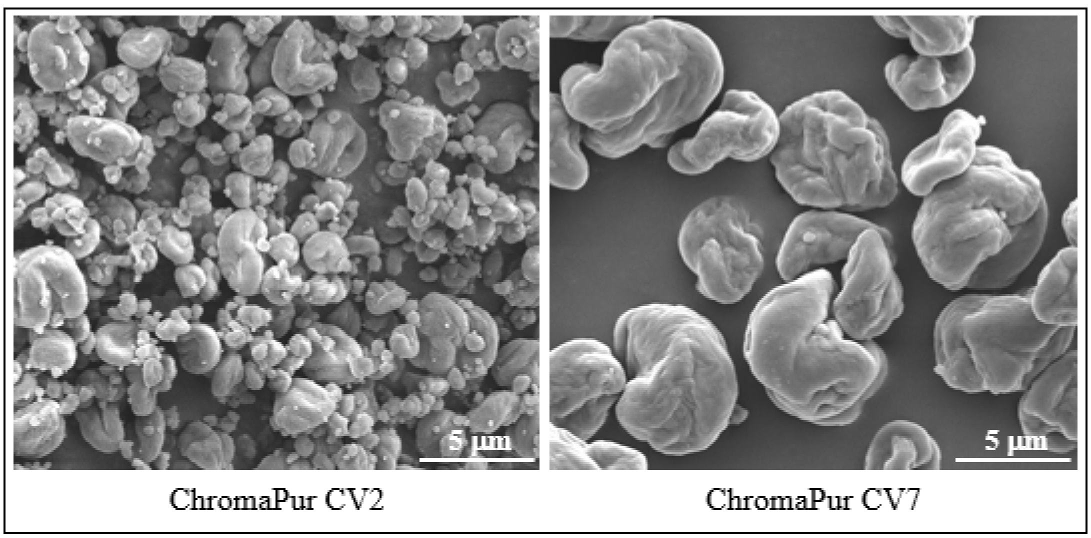

Figure 2: Scanning electron micrographs at 5,000 magnification of ChromaPur CV2 and CV7

3.2 Soft focus characterization

One of the primary uses of sensorial powders in make-up and skin care applications is to improve the optical performance of the product. The soft focus effect refers to the blurring of wrinkles and blemishes, without reducing transparency, thus making the skin look younger and more vibrant. To examine the soft focus performance of the various powders under study, six oil-in-water formulations were prepared and applied on Bioskin® Plates (50 year-old skin). A control formulation with no powder was compared to each sensorial powder at a 5% w/w inclusion level. As can be seen in Figure 3, by visual analysis, ChromaPur CV2 shows the highest level of soft focus of all the powders tested, followed by the ChromaPur CV7. The PMMA and Nylon-6, and Cellulose Fibers microbeads provide a matte effect but not significant soft focus. The control shows shininess and no soft focus.

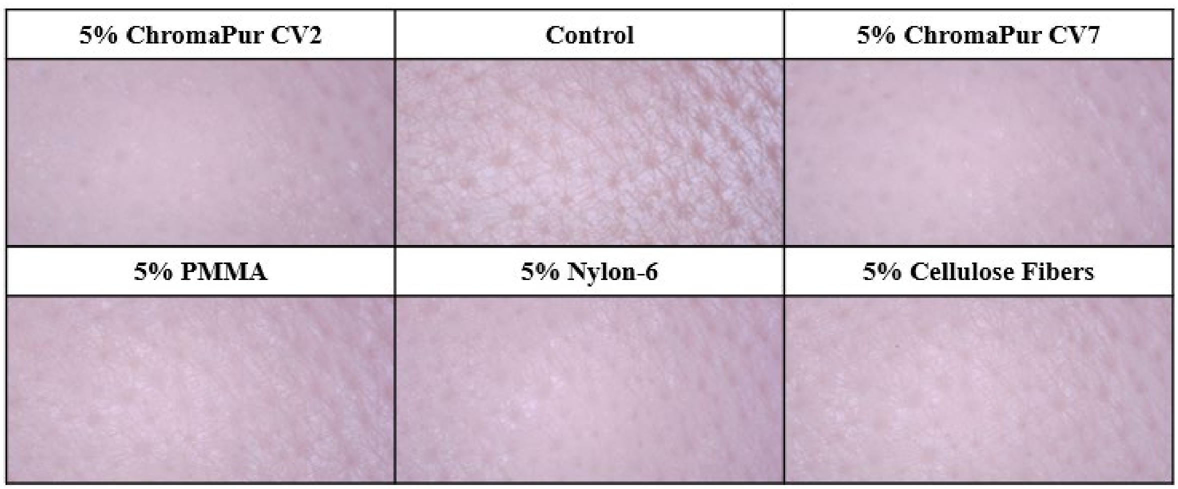

Figure 3: Optical images comparing the effect of the various powders (5% w/w) incorporated into an O/W emulsion on NO.10I cheek skin model Bioskin.

To provide a quantitative analysis, VISIA pore counting software was employed to determine the number of pores that remain visible after application. The results shown in Figure 4 confirmed the visual analysis that ChromaPur CV2 provides the highest level of soft focus, and as a result, pore reduction. In comparison to the control a significant reduction of pores (21%) is observed with ChromaPur CV2 and CV7 (11%). Whereas the PMMA, Nylon-6 and the Cellulose Fibers had no significant effect.

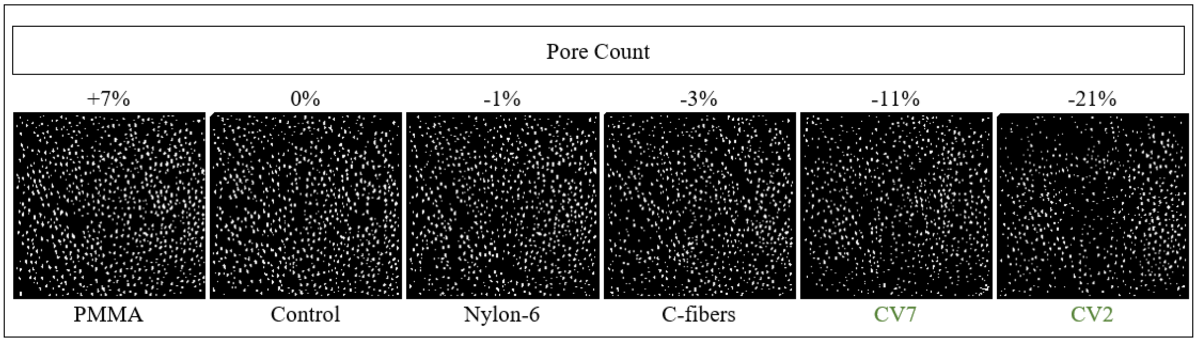

Figure 4: Pore reduction characterization using VISIA CR-1 software comparing the effect of the various powders (5% w/w) incorporated into an O/W emulsion on NO.10I cheek skin model Bioskin.

3.3 Colour intensity characterization

To study the effect of the various powders on colour intensity, the five sensorial powders were formulated into a lipstick at 5% w/w and compared to a control with no powder. Blind testing was carried out to quantify the effect on colour intensity when the lipstick is applied on a volunteer's forearm and to the lips (Figure 5 and 6). For each sensorial powder, a comparison was made between the formulation with the powder and the control. As can be seen in Figure 5, the lipsticks containing ChromaPur CV2 and CV7 show a better colour intensity compared to the control, with ChromaPur CV7 providing higher intensity than ChromaPur CV2. A similar level of enhanced colour intensity was shown with the PMMA and Nylon-12 microbeads compared to the control, however, no significant improvement was seen when the Cellulose Fibers were incorporated.

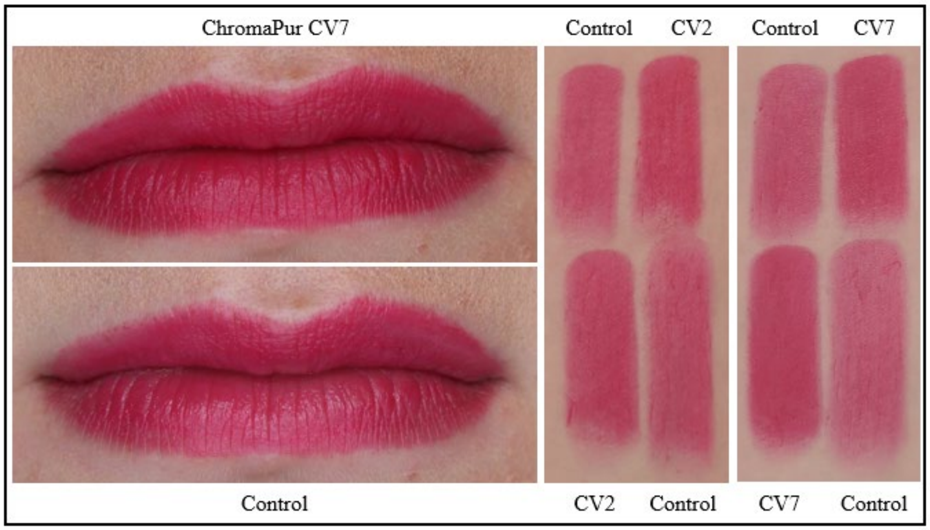

Figure 5: Colour intensity of ChromaPur CV 2 and CV7 in a lipstick with 5% powder loading compared to the control (no powder).

To better analyze the differences between powders, a direct comparison between each powder and ChromaPur CV7 was carried out (Figure 6). ChromaPur CV7 offers equivalent performance to the Nylon-12 and the PMMA. In comparison to Cellulose Fibers, the ChromaPur CV7 had significantly improved colour intensity. Similar results were seen when the lipstick was applied to the lips (not shown here).

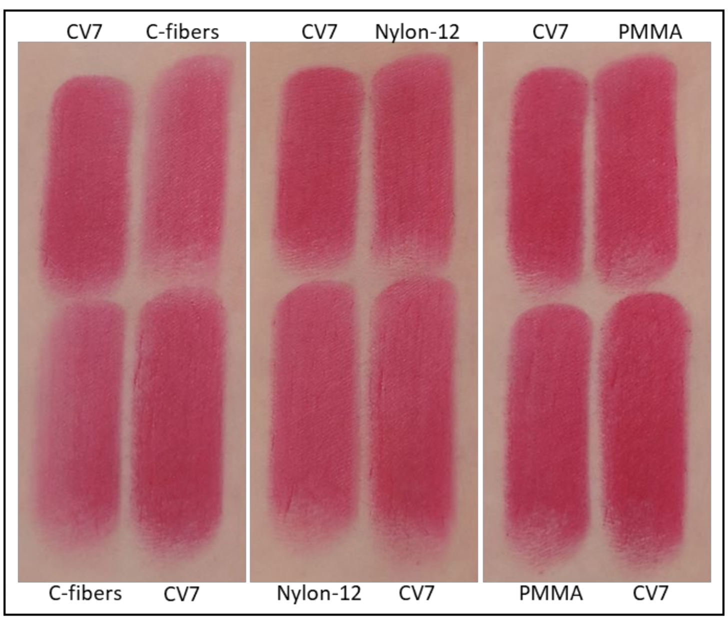

Figure 6: Colour intensity in a lipstick on the forearm with 5% powder loading of ChromaPur CV7 compared to Nylon-12, PMMA and Cellulose Fibers.

3.4 Sensory Characterization

Improved sensory performance is one of the primary reasons micron sized powders are added to cosmetic products. For this reason, this category of ingredients is often referred to as texturizing powders. To investigate the effect the powders have on sensory performance, the following five attributes were evaluated in an oil-in-water emulsion formulation for each powder at a 3% w/w inclusion level: softness, shininess, tackiness, drag and drying time. A blind paired analysis technique was employed whereby each powder was compared to the ChromaPur CV2 and CV7 directly. The testing results were analyzed through statistical analysis (ANOVA). As can been seen in Figure 7, at a 95% confidence level there are significant differences in the sensory performance of the powders, particularly between the Cellulose Fiber and the ChromaPur CV materials. Both ChromaPur CV2 and CV7 were found to reduce shine and drag compared to the Cellulose Fibers, whereas only ChromaPur CV7 was found to dry faster and only ChromaPur CV2 showed increased softness. The results between either ChromaPur CV2 or CV7 and the PMMA and Nylon-6 microbeads show no significant differences (positive or negative), aside from ChromaPur CV providing superior shine reduction in comparison with PMMA. No significant difference was found between ChromaPur CV2 and ChromaPur CV7.

ChromaPur CV2 and CV7 compared to:

	C-fibers	PMMA	Nylon-6
Dry faster	CV7 / CV2	=	=
Reduced shine	+	CV7 / CV2	=
Reduced tack	=	=	=
Reduced drag	+	=	=
Is softer	CV7 / CV2	=	=

+ Positive Significant Difference   = No Significant Difference

Figure 7: Sensory testing results characterized using ANOVA analysis at a 95% confidence level comparing Cellulose Fibers, PMMA and Nylon-6 to ChromaPur CV2 and CV7 in an O/W emulsion at a 3% powder loading.

4. Discussion

Various optical and sensory characterizations were carried out to compare and understand the benefits provided by 5 different sensorial powders. In terms of the soft focus effect, ChromaPur CV2 was found to give the highest level of blurring and pore reduction. This significant benefit can be attributed to the small particle size and refractive index of the powder. The ChromaPur CV2 has a median particle size of 2 microns, which is not only smaller than the plastic and other cellulose powders included as part of this study, but also most other sensorial powders on the market. In addition, the CV2 also has a significant portion of particles below 1 μm (D10 of 0.4 μm), which leads to increased scattering without absorption (> 2% decrease in transmittance over 1 cm pathlength). The larger CV7 powder also showed improved soft focus and pore reduction, but not to the same extent as the smaller CV2 (11% pore reduction compared to 21%). When one increases the cellulose particle size to the 30 μm level, the soft focus and pore reduction benefit decreases to a similar level of the control (no powder).

Previous studies have shown that the refractive index of the material, particularly with small particles, significantly affects the ability of the material to forward scatter [2]. To obtain significant scattering, one requires a large enough difference between the refractive index of the material and that of the surrounding media in order avoid refractive index matching. However, if the difference is too high, reflectance will increase and transmittance will decrease. Cellulose, with a refractive index of 1.54 is an ideal material to provide soft focus in cosmetic formulations as it is above the refractive index range of traditional formulations (1.40-1.5), but not too high to cause opacity (such as TiO_2). This is the reason the ChromaPur CV7 powder outperforms the PMMA microbeads, even though the PMMA microbeads are smaller in diameter.

In make-up products such as lipstick, it was shown that the larger spheroidal particles (CV7, PMMA, Nylon-12) provided better colour intensity than the smaller particles (CV2) and than the larger Cellulose Fibers. It is hypothesized that the larger spheroidal powders increase skin adherence and result in a better payoff and color intensity in lipstick than the smaller particles. Similar results were seen in pressed eyeshadows (not included here). Importantly, a natural and biodegradable material, ChromaPur CV7 is shown to perform at the same level as PMMA and Nylon-12.

From a sensory perspective in an O/W emulsion ChromaPur CV2 and CV7 have both been shown at a 95% confidence level to perform as good as the PMMA and Nylon-6 microbeads. Cellulose Fibers provide some benefits at a statistically equivalent level to the ChromaPur CV, but overall did not perform as well as the CV2 or CV7. This result can be explained by the spheroidal nature of the ChromaPur CV, which reduces shine more effectively due to superior scattering properties (discussed above) and provides a better skin feel, due to the well-known “ball-bearing effect” [3].

5. Conclusion

Over the past 10-years there has been a strong push to use eco-friendly, biodegradable ingredients in cosmetics. However, the cosmetic industry has been hesitant to switch from more traditional ingredients, such as plastic microbeads, as the natural alternatives could not meet the same performance standards. In this study we show that the spheroidal cellulose powders, ChromaPur CV2 and CV7, represent a viable alternative to microplastics in skin care and colour cosmetic applications without any loss in performance or significant reformulation required. In terms of soft focus and pore reduction, ChromaPur CV2 and CV7 outperform PMMA and Nylon-6. This is attributed to their unique optical properties stemming from their small particle size and high refractive index. ANOVA analysis quantitively showed that sensory benefits were as good or better than PMMA and Nylon-6. Cellulose Fibers, due to their size and morphology do not perform as well as the spheroidal cellulose powders or plastics in terms of optical or sensory properties.

6. Acknowledgments

7. Conflict of Interest Statement. None

8. References

1. European Commission (2022) Microplastics pollution – measures to reduce its impact on the environment. https://ec.europa.eu/info/law/better-regulation/have-your-say/initiatives/12823-Microplastics-pollution-measures-to-reduce-its-impact-on-the-environment_en

2. Han X, Shen J, Yin P, Hu S, Bi D, (2014) Influences of refractive index on forward light scattering. Optics Communications 316:198–205.

3. Moussour M, Lavarde M, Pense-Lheritier AM, Bouton F, (2016) Sensory analysis of cosmetic powders: personal care ingredients and emulsions. International Journal of Cosmetic Science 2016, 1–7