

KEY POINTS

- Silicones are widely used as binders in pressed powders but growing consumer skepticism has prompted the use of alternatives.
- The aim of this study was to assess the efficacy of three plant-derived natural binders in obtaining optimal pressed powders, using dimethicone as a standard.

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> quality compact powder product should have a uniform color and be strong enough to resist breakage through everyday use, yet be soft

enough to provide sufficient payoff. Binders are liquid or solid substances that hold other materials together, which is crucial in pressed powder cosmetics. Binders work by replacing the interstitial air and reducing the surface tension, which increases intermolecular forces between particles and therefore leads to higher density and adhesion/cohesion forces.¹

Binder concentration has a direct impact on the quality of any compact, including

Replacing Dimethicone with Plant-derived Alternatives*

consumer acceptability and manufacturing processability. The concentration also dictates whether a powder is cohesive enough to form a stable, uniform compact upon compression while remaining sufficiently free-flowing to be processed in subsequent manufacturing steps where it is blended; transported through hoppers and feeders; and filled into compacts and compressed. Powders that do not have adequate flowability can cause inconsistent batches, frequent stoppages or incomplete filling—requiring additional time and resources.²

The pressure at which a powder is pressed also influences the quality of the compact, with very high pressure resulting in a compact that is too hard with poor payoff and a

A key feature for pressed powders is the resistance to breaking since they are subjected to transport and consumer use.

tendency to glaze. Very low pressure produces a soft compact that is prone to breakage and rubs off easily. Pressure usually varies from 300 psi to 2,000 psi,³ depending on factors such as product composition, binder concentration, number of press steps or press duration.

Pigments can be prone to degradation by a variety of factors such as temperature, humidity, oxidation and light, both visible and ultraviolet (UV). Color stability is essential for consumer acceptability, therefore the addition of a binder mustn't reduce a pigment's chemical stability. It is speculated that some binders could even aid in protecting the pigment from degradation by mechanisms such as hydrophobic coating and UV protection. Liquid binders in powder cosmetics wet the pigments and improve their ease of dispersion by displacing the air from the particles' surface and replacing it with a liquid, which is crucial to develop

The global pressed powder market size is forecasted to grow from \$3.10 billion in 2021 to \$3.27 billion by 2028, at a CAGR of 5.8%.



Source: DATAINTELO

Table 1. Pressed Powder Formulations

Phase	INCI	% w/w		
A	Talc	qs to 100.0		
	CI 77510	10.0		
	Methylparaben	0.2		
	Propylparaben	0.1		
Binder variables				
В	Dimethicone	3.0-6.0-9.0		
	Diisooctyl Succinate	3.0-6.0-9.0		
	Triheptanoin	3.0-6.0-9.0		
	Heptyl Undecylenate	3.0-6.0-9.0		

the full strength of the color.⁴ Consequently, the main roles of liquid binders in pressed powder cosmetics are to improve color intensity (by wetting the pigments), to maintain color stability and to increase the powder cohesiveness (and thus reducing powder flow) to the desired level.

In the past, the combinations of gums, sugars and soaps have been used as binders. Nowadays, the most common liquid binders are silicone-based or hydrocarbon-based from petrochemical sources.5 According to Mintel,6 more than 60% of color cosmetics contain silicones. Silicones are most widely used for their hydrophobicity, spreadability, water and oxygen impermeability, and the unique soft skin feel they confer. The most commonly used silicone binders in pressed powders are cyclopentasiloxane, dimethicone and dimethicone crosspolymers. Studies by Stevens7-9 into the biodegradability of silicones showed that while they are not biodegradable, non-volatile polydimethylsiloxanes are degradable in soil, and volatile silicones such as cyclopentasiloxane degrade in the air in the presence of sunlight-ultimately to silica, water and carbon dioxide.

Due to a discordant history of safety, environmental impact and their synthetic nature, consumers have a poor perception of silicones. Despite studies confirming the non-occlusive behavior of commonly used silicones.^{10, 11} consumers and mainstream media still perceive them as such.¹²⁻¹⁴ Paired with the current market trends toward natural products, environmental concerns and sustainability,15, ¹⁶ there has been an increase in consumer demand for silicone-free, palm oil-free and natural products from sustainable sources.17 The cosmetic industry has responded by including natural emollients from sustainable sources to its portfolios, including their refined versions in the form of esters, triglycerides or alcohols.

The present study aimed to evaluate a range of natural binder alternatives and compare

them with a silicone standard. Given the fact that most information on binders and their properties is provided by raw material suppliers with no comparative data, carrying out an interand intra-formulary comparison of different binders could be beneficial for future formulating strategies.

Materials and Methods

Ingredients: Talc was used as a bulking agent, with a mixture of methylparaben and propylparaben as the preservative. CI 77510, also known as ferric ferrocyanide, Prussian blue or iron blue, was used as a pigment. It is graded color-stable and enabled testing of the influence of variable binders on the pigment, independent of the light conditions.

The three selected binders were liquid emollients from sustainable, plant-based sources. These were compared with medium-weight dimethicone (DM). All were used at three concentration levels and two different press forces. DM was chosen due to its status as the most widely used silicone in powder cosmetics.

According to their suppliers, all chosen alternative binders had favorable green credentials. Diisooctyl succinate (DOS) is an emollient and slip agent with a light cushion and silky after-feel. Triheptanoin (TH) is a medium-chain, non-greasy, clear and odorless triglyceride derived from castor bean and coconut. Heptyl undecylenate (HU) is a light and dry emollient derived from castor oil, which is suitable as an alternative to synthetic fluids such as cyclomethicone and mineral oils.

Test formulas: The test formulas used in the described studies are shown in **Table 1**. Phase A was premixed manually, after which it was milled using a laboratory-scale cutter mill for 1 min. A Phase B binder was then added and the mixture milled for 1 min. This bulk was left to stand for 24 hr. Bulk powder testing was carried out, after which the powder was left to stand for another 24 hr before being pressed



into rectangular metal godets using a manual powder press^a at 1,000 psi or 2,000 psi. A benchmark formula with no binder was also made.

Aerated and tapped bulk densities: Aerated and tapped bulk density methods were adapted from the British Pharmacopeia.¹⁸ A test sample mass (m) of 100 g was poured through a funnel into a 250-mL volumetric cylinder. The volume of powder was then recorded (V_0) and the density (ρ) calculated through equation:¹

$\rho = \mathbf{m} \, / \, \mathbf{V}$

The tapped bulk density was obtained by clamping the cylinder into a holder and performing manual tapping. The tapping procedure consisted of lifting the cylinder until it reached the clamp, which was fixed at a height of 3 cm, and then dropping it. This was repeated 50×, as suggested by Lau,¹⁹ after which the volume of tapped powder was recorded (V_f). The tapped density was then calculated.¹

The ratio of bulk volume to tapped volume is known as the *Hausner ratio*, which is an indication of the flowability and compressibility of a powder; it is calculated using equation.²

$\mathbf{H} = \mathbf{V}_0 - \mathbf{V}_f$

The Hausner ratio (H) is related to the Carr index I, which is another indicator of compressibility by the following equation:^{3, 20}

H = 100 / (100 - C)

The Carr index can also be calculated directly from bulk and tapped volumes by the equation:⁴

$C = [100 (V_0 - V_f)] / V_0$

A Carr index greater than 25 is considered an indication of poor flowability/high compressibility, while a Carr index below 15 is a sign of good flowability/poor compressibility. The higher the compressibility index, the more cohesive the powder, thus the more effective the binder. **Table 2** shows the scale of powder flowability and their corresponding Carr index and Hausner ratio ranges.²¹

Angle of repose: The angle of repose was measured according to the *British Pharmacopeia*

^a Kemwall, UK

method.²¹ A glass funnel with an orifice 12 mm in diameter was held at a height of 60 mm above a flat, circular base with a fixed diameter (d) of 58 mm. The powder was poured through the funnel until a conical heap was formed on the base and overflow occurred all around. The height (h) of the powder peak was measured and the angle of repose (α) was calculated using the equation:⁵

$\alpha = \tan^{(-1)} [2h/d]$

Each sample was tested in triplicate. As shown in **Table 2**,²¹ the lower the angle of repose, the better the powder flow property.

Color measurements: Using a spectrophotometer^b, color measurements were taken in the L^*, a^*, b^* color space with the standard illuminant D65 as a reference.²² From the obtained values, ΔE was calculated for various pairs of samples according to the below equation.⁶ ΔE is the measure of change in visual perception of two given color values.

$\Delta \mathbf{E} = \sqrt{(\mathbf{L}_{1} - \mathbf{L}_{2})^{2} + (\mathbf{a}_{1} - \mathbf{a}_{2})^{2} + (\mathbf{b}_{1} - \mathbf{b}_{2})^{2}}$

The tolerance level for ΔE is 2.5,²³ so any lower values indicate acceptable differences that are not perceptible by the human eye. A ΔE value over 2.5 can be an indication of the color degradation of the sample or a noticeable difference related to the influence of binders on the color intensity of the powder between samples.

Weathering test: Using the climate test chamber^c, accelerated color stability testing was carried out according to L'Oréal's QAC-MC-151 standard.²⁴ The samples were subjected to 24 \pm 1 hr of light at an illumination of 765 W/m². Color measurements were taken before and after illumination, and Δ E was calculated in order to assess any color degradation.

Accelerated stability: Samples of all formulations were stored at 40° C for a period of 10 weeks, per IFSCC.²⁵ Color measurements were taken before and after, and ΔE was calculated to assess any color change.

Indirect sunlight: Samples of all formulations were stored on a windowsill at room temperature (RT) for 10 weeks. Color measurements were taken before and after, and ΔE was calculated in order to assess any color change.

^b CM-2600d, Konica Minolta, USA

^c SUNTEST CPS+, Atlas, Germany



The ratio of bulk to tapped volume is known as the Hausner ratio, which is an indication of the flowability and compressibility of a powder.

Drop test: In order to assess the pressed powders' integrity and resistance to breaking, a drop test was carried out. Resistance to breaking is a key feature for pressed powders since they are subject to similar forces in both transport and consumer use. The test was adapted from ASTM D5276.²⁶ A godet was dropped from a height of

30 cm onto a flat solid surface and inspected by the naked eye to assess whether any visible damage had occurred. The drop test was repeated 3× for each formulation.

Hardness: The hardness of the powder cake was assessed by texture analyzer^d with a 2 mm stainless steel needle probe and the eyeshadow test protocol. Exponent software^e

^d Ta.XTplus Texture Analyser, Stable Micro Systems, UK ^e Stable Micro Systems, UK was used to obtain the hardness value (g) as an average of 3 readings. The higher the force required to penetrate the cake, the stronger the cake and the more effective the binder.

Payoff: Using the texture analzyer^c with a lipstick break strength rig, a makeup brush was fixed vertically in the lipstick holder and

Table 2. Scale of Powder Flowability

Flow character	Angle of repose (deg)	Compressibility index (%)	Hausner ratio
Excellent	25-30	≤ 10	1.00-1.11
Good	31-35	11-15	1.12-1.18
Fair	36-40	16-20	1.19-1.25
Passable	41-45	21-25	1.26-1.34
Poor	46-55	26-31	1.35-1.45
Very poor	56-65	32-37	1.46-1.59
Very, very poor	> 66	> 38	> 1.60

a powder godet was attached to the cantilever. A custom protocol was designed to allow the cantilever to move down and up at a fixed speed and force, allowing for the godet to sweep over the makeup brush for a total of 10 passes. The

Table 3. ΔE of Pressed Powders Beforeand After Weathering Test

Pressed Powder	1,000 psi	2,000 psi	
Benchmark	1.16	0.47	
DOS 3%	1.62	0.69	
DOS 6%	0.83	0.50	
DOS 9%	1.22	1.14	
DM 3%	1.00	1.64	
DM 6%	3.62	2.05	
DM 9%	10.18	9.86	
HU 3%	2.49	1.34	
HU 6%	2.41	1.79	
HU 9%	1.62	1.30	
TH 3%	2.14	1.21	
TH 6%	0.51	1.13	
TH 9%	1.20	0.86	

godet was weighed before and after brushing to calculate the percent of mass reduction. Force measurements were recorded for each reading to ensure consistency. A small percentage of mass reduction equals a low powder payoff, indicating a strong binder.

Results

The benchmark had the lowest Hausner ratio/Carr index, as expected. The addition of any binder increased the Hausner ratio/Carr index, indicating a decrease in the powder's flowability and an increase in its compressibility—an effect desired in pressed powder cosmetics. There was also a clear increase in the Hausner ratio within all binders from 3% to 6% but not from 6% to 9%.

When comparing different binders at the same concentration, there was a clear difference between binders at 9%, with DM giving the lowest Hausner ratio/Carr index and a passable flow character, while the other binders showed poor to very poor flow characters. Thus, DM gave the lowest compressibility, showing that the natural alternatives performed better at increasing powder cohesion. The results obtained from the angle of repose were in line with the aerated/ tapped density, thus the same conclusions apply.



Figure 1. ΔE of pressed powders with binders at different concentrations and different press strengths; DOS = Diisooctyl Succinate; DM = Dimethicone; HU = Heptyl Undecylenate; and TH = Triheptanoin

As shown in **Figure 1**, an increase in color difference was observed in proportion with the increase in binder concentration. This was expected since binders are known to "develop" the color of pigments due to their wetting



Table 4. Integrity of Pressed Powders After 1, 2 and 3 Drop Tests

Pressed Powder	1,000 psi			2,000 psi		
	1st Drop	2nd Drop	3rd Drop	1st Drop	2nd Drop	3rd Drop
Benchmark	Х	Х	Х	_	—	
DOS 3%	—	_		0	—	—
DOS 6%	0	—		0	—	_
DOS 9%	0	0	_	0	0	0
DM 3%	Х	Х	Х	0	—	—
DM 6%	0	—	—	0	0	—
DM 9%	0	0	0	0	0	0
HU 3%	0	0	—	0	0	0
HU 6%	0	0		0	0	0
HU 9%	0	0	0	0	0	0
TH 3%	0	_	_	0	0	0
TH 6%	0	0	0	0	0	0
TH 9%	0	0	0	0	0	0

X = unacceptable damage; - indicates minor damage; O = no damage



action. The effect was more pronounced at a higher press strength. Only DOS at 3% and HU at 9% showed visible color differences ($\Delta E > 2.5$) between the two press strengths. Overall, DM showed the smallest color differences at all binder concentrations and press strengths. On the other hand, DOS, HU and TH showed low ΔE values between them, which suggests the natural binders used in this study have a similar capacity to wet the pigment.

As seen in **Table 3**, the L^* , a^* , b^* values of the formulations with natural binders remained similar after the weathering test, with all ΔE values below 2.5. However, the samples with DM showed a detectable color difference when DM was used at 6% at a lower press strength and a large color difference when DM was used at 9% at both press strengths.

These results were mirrored in the accelerated stability test where the only detectable differences in color were found in the DM samples. The same ΔE value (3.31) was found for the 6% sample, pressed at both 1,000 psi and 2,000 psi, and similar ones for the 9% sample (10.00 and 10.85, respectively). Consistently, the same changes were detected in the indirect sunlight test at RT (2.5 and 2.81 for the 6% sample, and 10.15 and 10.27 for the 9% sample, respectively), indicating color instability in the presence of higher concentrations of DM.

Table 4 shows the results of the drop test, where X indicates unacceptable damage, i.e., the pressed powder broke or cracked; — indicates minor damage, i.e., the pressed powder showed some chipping; and O indicates no damage. In general, all binders increased the resilience of the pressed powders proportionally with the increase in binder concentration and with the increase in press strength, as expected. However, DM at 3% did not increase the resilience of the powder pressed at 1,000 psi, with the drop test results being identical to the benchmark. The remaining samples performed acceptably, while HU performed the best overall with no damage after the second drop.





Color measurements and stability tests showed natural binders provided a more intense color profile and better stability than DM.

Figure 2 shows the powders pressed at 2,000 psi were harder than those pressed at 1,000 psi, as expected. The samples with DM presented lower hardness values compared with the samples with natural binders at all concentrations and press strengths. These results are in line with those obtained from the drop test. The hardness of the pressed powders with natural binders pressed at 2,000 psi appears to drop with increasing binder concentration, which may be due to the binders working as a lubricant to the needle penetration. Moreover, when using natural binders in powders pressed at a lower press strength, the results indicated that binder concentration does not significantly affect the cakes' hardness.

Figure 3 shows the payoff of the pressed powders, described as the percent mean weight reduction (the samples identified in the same way as in **Figure 2**). As expected, powders pressed at 1,000 psi had a better payoff; i.e., higher weight reduction than those pressed at 2,000 psi. Furthermore, an increase in binder concentration resulted in a reduction of payoff.

In line with the results of hardness and drop tests, pressed powders with DM showed a

higher payoff than those with natural binders. The pressed powders with natural binders showed similar payoff among themselves, with DOS showing a tendency for higher values, especially when used at 3%. This is an interesting observation since DOS provided the samples with a hardness similar to that of other natural binders, therefore the use of DOS could provide a good combination of stability and payoff.

As seen in **Figure 2**, the hardness of the pressed powders with natural binders pressed at 2,000 psi tended to decrease with increasing binder concentration. However, the payoff did not follow the same pattern, which shows that even if not working to increase hardness, a higher concentration of binder will still increase the cohesiveness of the cakes and reduce their payoff.

Discussion

Both bulk powder tests showed that samples with DM possessed good powder flow and low compressibility, making the natural alternatives potentially better regarding cake stability. The press strength had a direct influence on the color measurements, which could be explained by the powder particles becoming more unidirectionally oriented and tightly packed under higher press strengths. This orientation could alter the way light is reflected and thus decrease light scattering, leading to a reduction in "color noise" and increased homogenous color intensity.²⁷

Color differences between samples were more pronounced at higher binder concentrations due to the binder lubricating action on powder particles, which facilitated their unidirectional orientation under pressure. The DM samples revealed the smallest color difference both when compared with the benchmark and among different concentrations. DM may be able to wet the pigments fully even at a lower concentration due to its low surface tension and molecular flexibility, making color differences unnoticeable at higher concentrations. All-natural binders presented similar color profiles, expressed as low ΔE . This similarity in pigment wetting may be due to their similar surface tensions (32.1 ± 3.0) mN/m, 34.9 ± 3.0 mN/m and 30.5 ± 3.0 mN/m for DOS, HU and TH, respectively), as well as their similar chemical nature, as opposed to a silicone-based polymer.

All light stability testing showed that the formulations containing DM were not color stable at higher concentrations and the color after testing was similar to that of the benchmark. As ferric ferrocyanide is a color-stable pigment, it is unlikely that DM caused pigment degradation. It is, however, possible that DM itself degraded into silica, water and carbon dioxide, which either evaporated or provided no pigment wetting. DM has been shown to degrade in the soil through hydrolysis catalyzed by clay minerals.28 This process is known to be especially rapid in dry soil with 50% degradation within several days.^{29, 30} The primary hydrolysis product, dimethylsilanediol (DMSD), is then either biodegraded or evaporated into the atmosphere where it is further broken down into its end products of silica, water and carbon dioxide.31

While there are no apparent studies on the degradation of dimethicone in the presence of other minerals such as talc, it is plausible that the same degradation process occurs in a dry talc-based medium. This is supported by the fact that DM's degradation also occurred dur-

ing the accelerated stability testing conditions (40 °C), where it was not exposed to sunlight,³¹ meaning its degradation was not solely related to light/UV exposure.

The pressed powder tests also showed that DM produced the weakest cakes and displayed a high payoff. It is speculated that DM's flexible siloxane backbone, which gives it a rubberlike elasticity, makes it difficult to hold a cake together while the more rigid hydrocarbons form stronger cakes. HU gave the strongest cake in the payoff and drop testing, especially at higher concentrations. This result is in line with the findings from the bulk powder and color tests. Higher press strengths resulted in harder and more cohesive cakes, probably due to a greater reduction of interstitial air between particles and in turn increase their intermolecular forces.

Overall, the results have shown that diisooctyl succinate, triheptanoin and heptyl undecylenate are more effective binders than dimethicone in both bulk and pressed powder forms. However, these benefits often translate into lower payoff, requiring a careful balancing of the opposing binder characteristics. For efficient formulation work in this area, it would be useful to acquire the data from consumer trials, showing the level of payoff acceptable to consumers.

Conclusion

Based on bulk and pressed powder test results, the plant-derived binders assessed here provided better cohesion to the powder formulations when compared with DM. The color and stability tests also showed that natural binders provided a more intense color profile and more favorable color stability than DM. These results were unexpected since it was assumed that natural binders would provide poorer color stability due to their natural origin. This disparity probably relies on the degradation of DM under the test conditions.

Providing that the low payoff is resolved, the results obtained indicated that plantderived silicone alternatives diisooctyl succinate, heptyl undecylenate and triheptanoin could be effectively used as binders in pressed powder cosmetics with no negative implications on color stability, intensity or product functionality. **Acknowledgments:** The authors wish to thank Azelis (UK) for generously supplying the ingredients used in this study.

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