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## ***Silicone-Free W/O Foundation Based on Poly-Ion Complex Technology for Long-Lasting Performance***

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### **1. Introduction**

Foundations are used to minimize color irregularities (such as spots and dullness), smooth skin imperfections (including fine lines, textures, and pores), and enhance overall skin tone to create a uniform makeup finish with a beautiful aesthetic appearance. From this perspective, makeup products should maintain these effects over time, commonly referred to as long-lasting effect. However, in practical use, the appearance of makeup may deteriorate over time due factors such as sweating or sebum secretion [1]. This can result in increased visibility of spots and pores, along with changes in color tone and surface unevenness [2]. Therefore, achieving long-lasting performance remains an essential requirement for makeup products.

Currently, the long-lasting performance of cosmetics relies mainly on silicone-based film-forming technologies, and one of the most widely used is trimethylsiloxysilicate (TMS), a highly cross-linked silicone resin that is resistant to sweat and sebum. However, growing environmental concerns have made the continued use of silicone-based solutions in industry increasingly problematic. Furthermore, replacing TMS with naturally derived film formers does not provide comparable long-lasting effects, largely due to their inherently hydrophilic nature.

In this context, we explore the field of poly-ion complex (PIC) technology, involving oppositely charged components that can self-assemble through electrostatic interactions to form an insoluble three-dimensional cross-linked structure, often gel- or film-like [3-7].

In this report, electrostatic interaction between chitosan lactate (CL), a water-soluble derivative of chitosan, a natural polysaccharide, which is prepared by reacting chitosan with lactic acid and amino acid surface-treated pigments was considered. Zeta potential measurements [8] were performed to verify the cationic nature of CL, to determine the ionic nature of various pigments and to select the most relevant ones. Subsequently, a water-in-oil (W/O) fluid foundation incorporating the CL dissolved in the aqueous phase and the selected hydrophobic surface-treated pigments dispersed in the oil phase was prepared. The properties of the dry thin films of this W/O foundation were investigated by static contact angle measurements, squalane drop test, Fourier Transform Infrared Spectroscopy (FT-IR), and surface observations. Clinical studies were also conducted to evaluate the long-lasting performance of this resulting fluid foundation.

## 2. Materials and Methods

### 2.1. Selection of pigments to form PIC with CL

#### 2.1.1. Zeta potential measurement

To evaluate the ionic nature of the pigments used in foundations (red, yellow, black, and white), including those treated with amino acid-based hydrophobic agents as well as to verify the cationic nature of CL, zeta potential measurements were conducted using a Litesizer 500 instrument (Anton Paar). Table 1 lists the pigments evaluated, all of which are commercially available grades.

First, each pigment was dispersed using a vortex mixer at a concentration of 20 mg/mL in an aqueous solution containing 0.5 wt% of non-ionic surfactant 2-[4-(2,4,4-trimethylpentan-2-yl)phenoxy]ethan-1-ol (Triton X-100, Thermo Scientific Chemicals). Then these pigment dispersions were subsequently diluted with the same aqueous solution containing 0.5 wt% of non-ionic surfactant to achieve a measured transmittance of 70% or higher. For CL, more simply, an aqueous solution containing 0.25 wt% of CL was prepared for the measurement.

Before starting the zeta potential measurement, the pH of each solution obtained (with pigment or CL) was first adjusted to 3 using 0.05 mol/L HCl. The zeta potential measurement was then performed by progressively increasing the pH to the desired value by gradually adding NaOH at 0.05 mol/L.

Table 1. Pigments used for zeta potential measurements

| Pigments           | Color  | INCI  |
|--------------------|--------|---|
| Untreated pigments | Red    | CI 77491 (Iron oxide)   |
|                    | Yellow | CI 77492 (Iron oxide)   |
|                    | Black  | CI 77499 (Iron oxide)   |
|                    | White  | CI 77891 (Titanium dioxide), Aluminum hydroxide   |
| SLG pigments       | Red    | CI 77491 (Iron oxide), Sodium lauroyl glutamate, Lysine, Magnesium chloride                           |
|                    | Yellow | CI 77492 (Iron oxide), Sodium lauroyl glutamate, Lysine, Magnesium chloride                           |
|                    | Black  | CI 77499 (Iron oxide), Sodium lauroyl glutamate, Lysine, Magnesium chloride                           |
|                    | White  | CI 77891 (Titanium dioxide), Aluminum hydroxide, Sodium lauroyl glutamate, Lysine, Magnesium chloride |
| DSG pigments       | Red    | CI 77491 (Iron oxide), Disodium stearyl glutamate, Aluminum hydroxide                                 |
|                    | Yellow | CI 77492 (Iron oxide), Disodium stearyl glutamate, Aluminum hydroxide                                 |
|                    | Black  | CI 77499 (Iron oxide), Disodium stearyl glutamate, Aluminum hydroxide                                 |
|                    | White  | CI 77891 (Titanium dioxide), Disodium stearyl glutamate, Aluminum hydroxide                           |

#### 2.1.2. Isoelectric point of each pigment and CL

The isoelectric point is the pH of the aqueous solution in which the compound is present at neutral charge. In our study, this value is extrapolated by drawing a line connecting two points close to neutral electrical potential and extracting the pH value at 0 mV.

#### 2.1.3. Isoelectric point of systems integrating pigments and CL

To assess whether there is an interaction between pigments and CL, the isoelectric point of a mixture solution containing pigments and CL was measured. This study was done only for SLG pigments. The pigments were dispersed at a concentration of 20 mg/mL in an aqueous solution

containing 0.15 wt% CL using a vortex mixer. Centrifugation was then applied to separate the densest solid part from lightest liquid part of the aqueous solution (1), and the upper liquid part was removed (2). The remaining precipitated part was then diluted with distilled water and mixed again using a vortex mixer (3). The centrifugation, separation, and dilution steps (1) to (3) were repeated a total of five times.

The zeta potential measurements were performed using the same method as described in 2.1.1. The isoelectric point was extrapolated using the same method as in 2.1.2.

## 2.2. Foundation test

### 2.2.1. Fluid foundation formulation (monochromatic bases)

Monochromatic W/O fluid foundations were prepared by independently dispersing red, yellow, black, and white SLG pigments in the oil phase, while CL was dissolved in the aqueous phase, following the composition detailed in Table 2. Reference formulas without CL were also prepared in the same way by adjusting the water content to compensate for the absence of CL. After preparation, the formulation stability condition was confirmed.

Table 2. Composition of each monochromatic W/O fluid foundation

| Components  | Red<br>(wt%) | Yellow<br>(wt%) | Black<br>(wt%) | White<br>(wt%) |
|---|--------------|-----------------|----------------|----------------|
| Polyglyceryl-2 isostearate, polyglyceryl-6 polyricinoleate  | 4.00         | 4.00            | 4.00           | 4.00           |
| Isododecane, disteardimonium hectorite, propylene carbonate | 12.00        | 12.00           | 12.00          | 12.00          |
| Dextrin palmitate   | 0.80         | 0.80            | 0.80           | 0.80           |
| SLG pigment (Red)   | 13.00        | -               | -              | -              |
| SLG pigment (Yellow)  | -            | 13.00           | -              | -              |
| SLG pigment (Black)   | -            | -               | 13.00          | -              |
| SLG pigment (White)   | -            | -               | -              | 13.00          |
| Undecane, tridecane   | 20.38        | 20.38           | 20.38          | 20.38          |
| Water   | 41.30        | 41.30           | 41.30          | 41.30          |
| Glycerin, pentylene glycol                                  | 7.00         | 7.00            | 7.00           | 7.00           |
| CL  | 1.00         | 1.00            | 1.00           | 1.00           |
| Chlorphenesin, capryl glycol                                | 0.52         | 0.52            | 0.52           | 0.52           |
| Total   | 100.00       | 100.00          | 100.00         | 100.00         |

### 2.2.2. Fluid foundation formulation (beige-shade)

Three W/O beige-shade fluid foundation formulas were prepared using SLG pigments: the one with CL named PIC (Foundation A), another one without CL (Foundation B), and the last one with TMS (Foundation C). The compositions of the respective formulas are described in Table 3. After preparation, the formulation stability condition was confirmed.

### 2.2.3. Foundation thin film contact angle measurement

Foundation thin films were prepared by spreading a 90  $\mu\text{m}$ -thick liquid film of foundation onto a polyethylene terephthalate (PET) substrate (Byco-chart, 100  $\mu\text{m}$  thick, BYK) using a manual film applicator (T.P. Giken). Subsequently, the thin films were dried in an oven at 35°C for 4 hours. Surface wettability tests were conducted on the day after sample preparation using the

sessile drop method with a contact angle meter (Dme-210, Kyowa). A 2.5  $\mu\text{L}$  droplet of distilled water is deposited onto the foundation film surface, and the reported value is the average contact angle measured 2 minutes after droplet deposition, based on six measurements taken at different locations on the film surface and under the conditions of 23°C / 40% RH.

#### 2.2.4. Wicking & stamping on the foundation films

A wicking test was conducted by observing the evolution of a 2.5  $\mu\text{L}$  squalane droplet shape over time. Photographs were taken immediately after the drop was deposited onto the dry thin film of foundation and after 5 hours of contact with the film surface. Subsequently, at the 5-hour mark, a stamping test was performed, and the squalane droplet was removed using a small square (1.5 x 1.5  $\text{cm}^2$ ) of absorbing fabric under a pressure of 11  $\text{g}/\text{cm}^2$  for approximately 3 seconds. A picture of the residue transferred onto the fabric was taken.

Table 3. Formulation composition of beige-shade fluid foundation

| Components  | Foundation A<br>(wt%) | Foundation B<br>(wt%) | Foundation C<br>(wt%) |
|---|-----------------------|-----------------------|-----------------------|
| Polyglyceryl-2 isostearate, polyglyceryl-6 polyricinoleate  | 4.00                  | 4.00                  | 4.00                  |
| Isododecane, disteardimonium hectorite, propylene carbonate | 12.00                 | 12.00                 | 12.00                 |
| Dextrin palmitate   | 0.80                  | 0.80                  | 0.80                  |
| SLG pigment (Red)   | 1.11                  | 1.11                  | 1.11                  |
| SLG pigment (Yellow)  | 3.75                  | 3.75                  | 3.75                  |
| SLG pigment (Black)   | 0.68                  | 0.68                  | 0.68                  |
| SLG pigment (White)   | 7.46                  | 7.46                  | 7.46                  |
| Undecane, tridecane   | 20.38                 | 20.38                 | 17.38                 |
| TMS   | -                     | -                     | 3.00                  |
| Water   | 41.30                 | 42.30                 | 42.30                 |
| Glycerin, pentylene glycol                                  | 7.00                  | 7.00                  | 7.00                  |
| CL  | 1.00                  | -                     | -                     |
| Chlorphenesin, capryl glycol                                | 0.52                  | 0.52                  | 0.52                  |
| Total   | 100.00                | 100.00                | 100.00                |

#### 2.3. Optical microscope observations

Low-viscosity simplex W/O formulations containing only the SLG white pigment with and without CL were prepared with the composition described in Table 4 and named Trial 1 and Trial 2, respectively. A 60  $\mu\text{L}$  drop of each formulation was spread on a glass plate at 37°C and observed during drying using an optical microscope (BX51, Olympus). Surface observations for both trials were reported after 5, 20, and 90 minutes of drying.

#### 2.4. FT-IR measurements

A mass of 4 g of each foundation A and B was spread in a 7 cm diameter glass petri dish and dried in an oven at 35°C for 12 hours. The CL alone was measured in its solid state by preparing the sample from CL powder. Each sample was measured using an FT-IR spectrometer (Alpha II Compact FT-IR spectrometer, Bruker) under the following conditions: resolution of 2  $\text{cm}^{-1}$ ; number of scans: 32. The results were then compared.

Table 4. Composition of formulas for microscope observation

| Components   | Trial 1 | Trial 2 |
|--|---------|---------|
|  | (wt%)   | (wt%)   |
| Polyglyceryl-2 isostearate, polyglyceryl-6 polyricinoleate | 3.92    | 3.92    |
| Dextrin palmitate  | 0.78    | 0.78    |
| SLG pigment (White)  | 1.46    | 1.46    |
| Undecane, tridecane  | 45.00   | 45.00   |
| Water  | 40.49   | 41.47   |
| Glycerin, pentyleneglycol                                  | 6.86    | 6.86    |
| CL   | 0.98    | -       |
| Chlorphenesin, capryl glycol                               | 0.51    | 0.51    |
| Total  | 100.00  | 100.00  |

## 2.5. Clinical study for long wear performance

To evaluate the wear performance of the foundation containing SLG pigments and CL (PIC foundation), two commercially available long-lasting foundation, one water-in-silicone (W/Si) containing TMS (Foundation 1) and one W/O (Foundation 2) were used as references. They were tested over a 24-hour period following a single application by Chinese female consumers (n=10-12), aged 20 to 50 years with Fitzpatrick skin type II-III (light to moderate skin tone), all of whom were regular foundation users. Clinical scoring of make-up wearing performance including “Lasting” (defined as Presence of Product + Homogeneity of Complexion), “Surface Disorder Appearance”, and “Shade Stability” was evaluated on the whole face using a standardized 0-9 scale by a validated expert. Evaluations were performed at Baseline (T0), 10 minutes after make-up application (Timm), and then at 8, 12, 16, and 24 hours after application (T8h, T12h, T16h and T24h) in a standard environment with uniform lighting (Cool Daylight, 6500K). Each subject was assessed by the same validated expert grader from baseline to study completion. All measurements were carried out in a climate-controlled room, with temperatures maintained between 20-22°C, and relative humidity ranging from 41.4% to 59.7%.

## 3. Results

### 3.1. Selection of pigments to form PIC with CL

#### 3.1.1. Zeta potential measurement

The zeta potential results of each pigment, along with CL were shown in Figure 1. The untreated pigments are cationic under acidic conditions and become anionic when the pH is above 6. On the other hand, the SLG pigments exhibit a negative charge over a wider range from about pH 3. Among the DSG pigments, the red, yellow, and black pigments show anionic properties from about pH 4, while the white pigment is rather cationic up to pH 7 and then become anionic. CL is cationic over the pH range from 3 to 9.

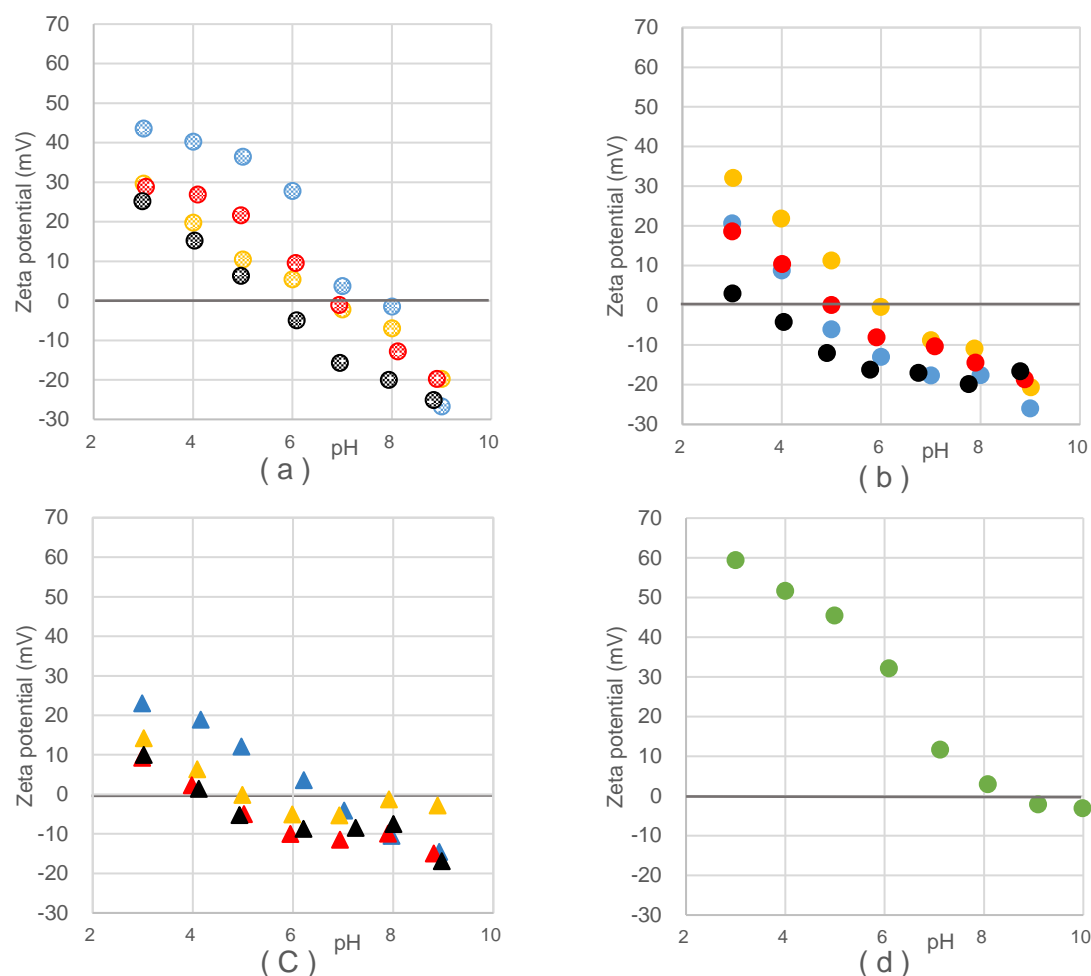


Figure 1. Zeta potential measurements: (a) Untreated pigments, (b) SLG pigments, (c) DSG pigments, (d) CL. (Red: Red, Yellow: Yellow, Black: Black, White: White, CL: CL)  $n=3$

### 3.1.2. Isoelectric point of each pigment and CL

The values of the isoelectric point of each pigment were extrapolated from Figure 1 and presented in Table 5. The black SLG pigment has the lowest isoelectric point among of all tested pigments. On average, SLG pigments have the lowest isoelectric points and are considered to have anionic behavior under our study conditions. The isoelectric point of CL is at pH 8.7.

Table 5. Isoelectric point of the pigments (pH value)

|                   | Red | Yellow | Black | White |
|-------------------|-----|--------|-------|-------|
| Untreated pigment | 6.9 | 6.7    | 5.6   | 7.7   |
| SLG pigment       | 5.0 | 6.0    | 3.4   | 4.6   |
| DSG pigment       | 4.3 | 5.0    | 4.3   | 6.6   |

### 3.1.3. Isoelectric point of systems integrating pigments and CL

The isoelectric point values of the systems mixing SGL pigments and CL are shown in Table 6. The obtained values which fall between the isoelectric point value for CL alone and that of

the pigments alone indicate that a PIC was formed by electrostatic interaction between the anionic SLG pigments and the cationic CL.

Table 6. Isoelectric point of the systems mixing SGL pigments and CL (pH value)

| SLG pigment            | Red+CL | Yellow+CL | Black+CL | White+CL |
|------------------------|--------|-----------|----------|----------|
| Isoelectric point (pH) | 5.7    | 6.5       | 6.4      | 6.2      |

### 3.2. Foundation test

#### 3.2.1. Fluid foundation formulation (monochromatic bases)

The dry thin films obtained for each monochromatic foundation are uniform. The water static contact angle measurements for all films were shown in Figure 2. All foundation films containing CL are hydrophobic with water contact angles ranging from 89.4° to 103.9°. The highest values are for black pigments then red followed by white then slightly lower values for yellow. When the films do not contain CL, all films are hydrophilic with water contact angles ranging from 30.5° to 55.4°.

#### 3.2.2. Fluid foundation formulation (beige-shade)

The dry thin films obtained for all beige shade foundations were homogeneous with a uniform color. The static contact angle of water for Foundation A, containing all SGL colored pigments and CL, indicates that the surface is hydrophobic (Figure 3). While films of Foundations B and C are hydrophilic as the water contact angles are 40.1° and 43.9°, respectively.

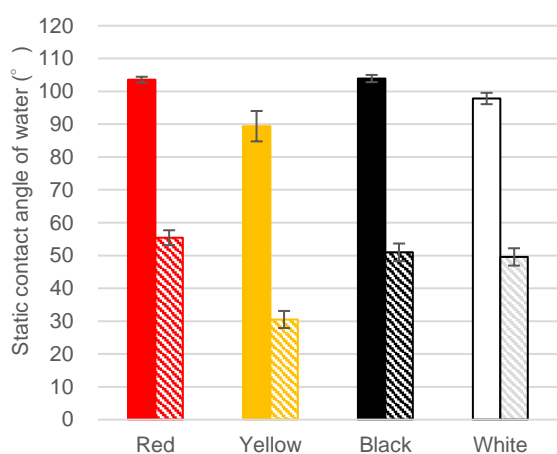


Figure 2. Contact angle of monochromatic foundation (n=6)

■ ■ ■ ■ : with CL  
▨ ▨ ▨ ▨ : without CL

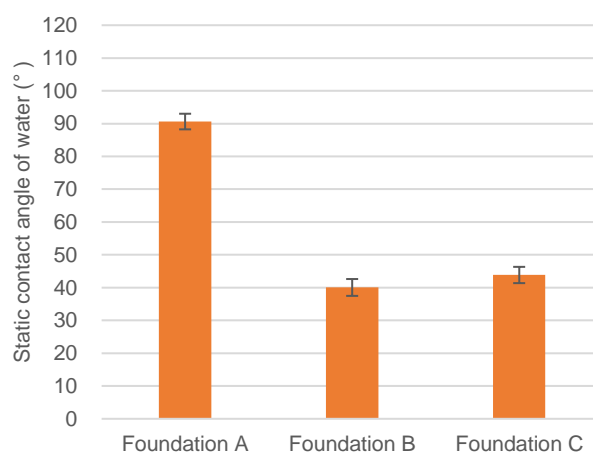
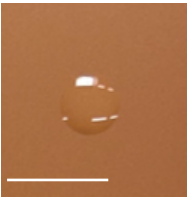
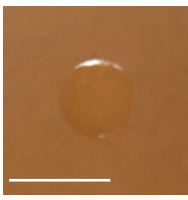
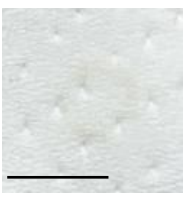

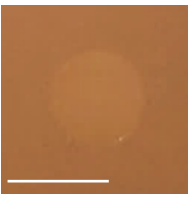
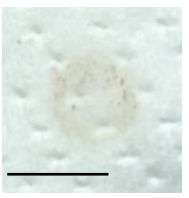
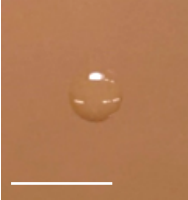
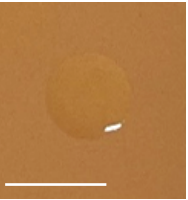
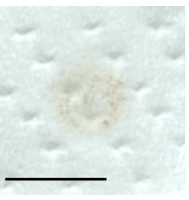


Figure 3. Contact angle of fluid foundation (n=6)

#### 3.2.3. Wicking & stamping on the foundation films

The squalane drop spread slightly after 5 hours on the thin film of foundation A containing CL, but almost no transfer to the fabric was observed when the squalane drop was removed by stamping (Table 7). In contrast, the squalane drop spread significantly on the thin film of foundation B without CL, and transfer residues on the fabric were observed. For reference, slight spreading was observed on the thin films of foundation C containing TMS, and slight transfer to the fabric was observed.

Table 7. Wicking &amp; stamping test for squalane drop on three foundation thin films

| W/O foundation | Wicking Timm*   | Wicking T5h*  | Stamping T5h*  |
|----------------|---|---|--|
| Foundation A   |  |  |  |
| Foundation B   |  |  |  |
| Foundation C   |  |  |  |

\* 10 minutes after dropping squalane (Timm) and after 5 hours (T5h). Scale bar 5mm

### 3.3. Optical microscope observations

In Trial 1, containing CL, the pigments tend to aggregate strongly at the emulsion interface during the drying process leading to a large-scale patterning, whereas in Trial 2, which does not contain CL, the pigments also aggregate but on a much smaller scale that does not appear to follow the moving of the contour of the water-oil interface (Figure 4).

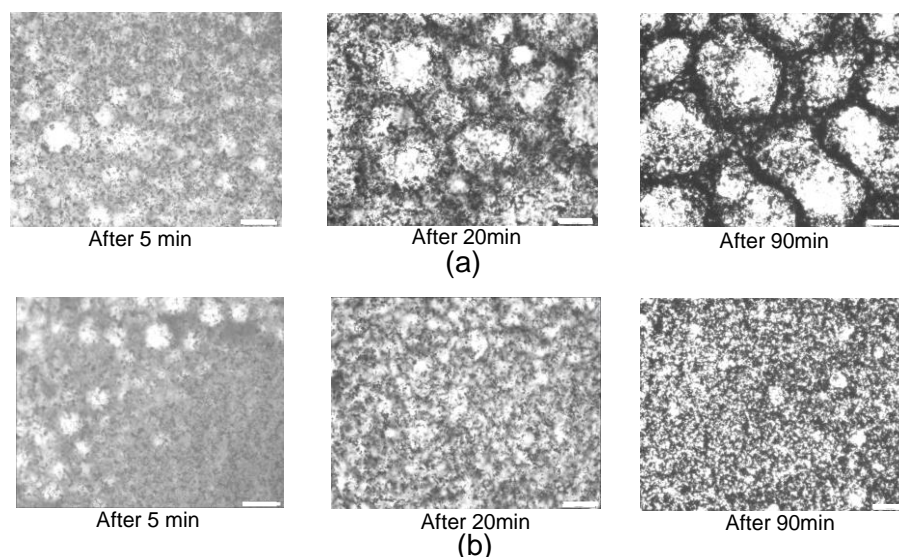


Figure 4. Photographs of optical microscope observations: (a) Trial 1, (b) Trial 2 (Scale bar: 100  $\mu\text{m}$ )

### 3.4. FT-IR measurements

The FT-IR results were shown in Figure 5. For Foundation A and for CL alone, a peak attributed to  $\text{NH}_2$  bending vibration ( $1640\text{--}1560\text{ cm}^{-1}$ ) [9] was observed. Since this peak was



absent in Foundation B, it is presumed to originate from chitosan. In addition, a slight peak shift between Foundation A and CL was noted, suggesting a change in the chitosan state.

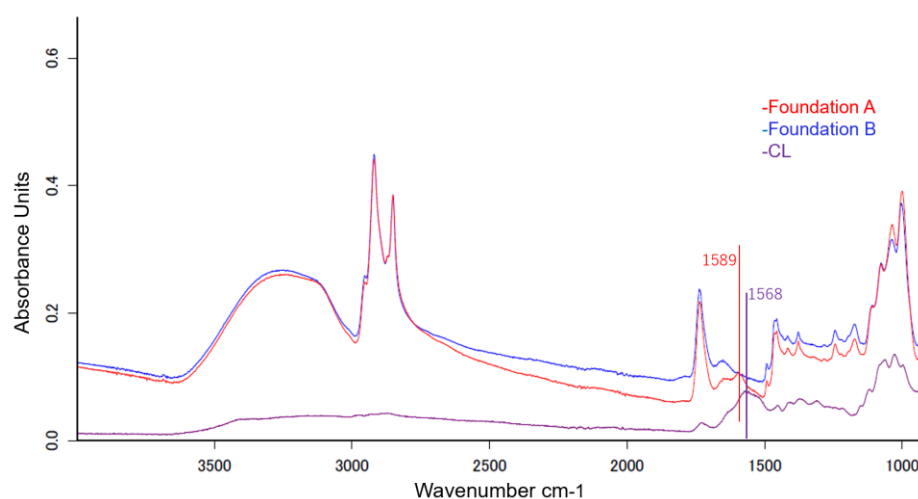


Figure 5. FT-IR measurement of Foundation A, B, and CL

### 3.5. Clinical study for long wear performance

The results of the clinical grading were shown in Table 8. The PIC foundation demonstrated long wear up to 24 hours after a single application ( $p < 0.05$  at a 95% Confidence Interval (CI)), across all three clinical attributes: Lasting, Surface Disorder Appearance, and Shade Stability. Compared to 10-minutes (Timm) evaluation, the change over time for PIC foundation was smaller (indicating better long wear) than Foundation 2, and its performance was comparable to Foundation 1 showing similar wear over 24 hours.

Table 8. The results of clinical grading for long wear performance

| Prototypes                             | Clinical Attributes         | Time comparison results (versus Timm) <sup>*1</sup> |             |             |             |
|--|-----------------------------|---|-------------|-------------|-------------|
|  |                             | T8h   | T12h        | T16h        | T24h        |
| PIC foundation<br>(n = 12)             | Lasting                     | <b>-4%</b>  | <b>-4%</b>  | <b>-7%</b>  | <b>-13%</b> |
|  | Surface Disorder Appearance | <b>-9%</b>  | <b>-10%</b> | <b>-11%</b> | <b>-24%</b> |
|  | Shade Stability             | <b>-10%</b>   | <b>-12%</b> | <b>-13%</b> | <b>-16%</b> |
| Commercially available                 | Lasting                     | NS  | NS          | <b>-4%</b>  | <b>-14%</b> |
| Foundation 1 <sup>*2</sup><br>(n = 10) | Surface Disorder Appearance | NS  | NS          | NS          | <b>-22%</b> |
|  | Shade Stability             | NS  | <b>-8%</b>  | <b>-8%</b>  | <b>-14%</b> |
| Commercially available                 | Lasting                     | <b>-15%</b>   | <b>-20%</b> | <b>-22%</b> | <b>-33%</b> |
| Foundation 2 <sup>*3</sup><br>(n = 10) | Surface Disorder Appearance | <b>-14%</b>   | <b>-17%</b> | <b>-14%</b> | <b>-31%</b> |
|  | Shade Stability             | <b>-9%</b>  | <b>-10%</b> | <b>-13%</b> | <b>-17%</b> |

<sup>\*1</sup> Variation Percentage (%) about the Timm values,

<sup>\*2</sup> W/Si with TMS long-lasting foundation

<sup>\*3</sup> W/O foundation,

**Bold:** the variation with statistically significant ( $p < 0.05$  at 95% CI using Wilcoxon Signed Rank test)

NS: the actual p-value is greater than or equal to 0.05 at 95% CI using Wilcoxon Signed Rank test

## 4. Discussion

The selection of surface-treated pigments plays a key role in the PIC technology applied to formulation. In this study, we identified SLG pigments as the most suitable candidates to form

a PIC by electrostatic interaction with CL due to its rather anionic nature, under studied usage conditions, revealed by zeta potential measurements. Furthermore, we observed a correlation between the value of the isoelectric point for each individual pigment and the water static contact angle value for each monochromatic foundation thin films containing CL. Specifically pigments with higher anionic character i.e. lower isoelectric point values tend to exhibit higher water contact angle. In contrast, thin films of foundation formulated without CL showed consistently low contact angle and hydrophilicity, regardless of pigment type.

For wicking and stamping tests using squalane drops and for water contact angle measurement, Foundation A, which incorporates PIC technology, demonstrated the highest performance. These findings suggest that the PIC formed by interaction between cationic CL and anionic SGL pigments is insoluble in both water and oil, contributing to long-lasting properties of the resulting foundation. Further evidence of the PIC formation was obtained through isoelectric point analysis, FT-IR spectroscopy, and microscopic observation of the system mixing SGL pigments and CL. These results were subsequently validated by a clinical study.

A stable W/O emulsion can be obtained by dispersing SLG pigments in the oil phase as an anionic component and by dissolving CL in the water phase as a cationic component. Upon application, the emulsion breaking and evaporation of volatile components from both phases causes interaction between CL and amino acid treated SGL pigments, leading to the formation of an insoluble film-like PIC complex, thus improving the long-lasting performance of the foundation thin film.

## 5. Conclusion

We have developed an innovative PIC technology for cosmetic formulations that achieves long-lasting efficacy without the use of silicone derivatives. Our findings indicate that even commonly used pigments exhibit significantly enhanced long-lasting performance when combined with CL, provided they are treated with an anionic surface coating. In our study, the interaction between CL and SLG-treated pigments results in excellent hydrophobicity, oil resistance and superior long-lasting effect compared to conventional W/O foundations. Furthermore, we have demonstrated that the performance of this foundation containing PIC technology is comparable to that of commercially available long-wear W/Si foundations containing TMS film former. PIC technology shows promise not only for foundations but also for a wide range of cosmetic products, including eyeshadows and sunscreens.

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