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## ***“La beauté ne transpire pas: Microstructural and sensory analysis of lipstick under sweating conditions”***

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### **1. Introduction**

The global lipstick market was valued at approximately USD 10.65 billion in 2023 and is expected to reach USD 15.49 billion by 2031, growing at a 5.6% CAGR from 2024 to 2031<sup>1</sup>. Accordingly, consumer expectations for lipstick formulations have evolved beyond basic attributes such as spreadability and moisturization to include enhanced physicochemical stability under both high and low temperature conditions.

Lipsticks are typically formulated as oil–wax gel systems, composed primarily of waxes and oils. Upon application to the skin, the wax matrix undergoes mechanical disruption, which critically influences the sensory experience. The microstructure and mechanical properties of the formulation are closely tied to user perception, with the composition and physical characteristics of the waxes and oils playing a pivotal role<sup>2,3</sup>. Waxes are essential for maintaining the structural integrity of lipsticks and are generally classified into hard and soft waxes according to their melting points. Hard waxes, with high melting points, enhance shape retention and mechanical strength, while soft waxes, characterized by lower melting points, contribute to spreadability and a smooth application texture<sup>4</sup>. Oils are equally important for defining sensory attributes, such as glossiness, moisturization, and ease of application. Their physicochemical properties, particularly polarity and viscosity, significantly impact microstructure formation and mechanical behavior. Highly polar oils interact strongly with the wax matrix, often resulting in microstructural heterogeneity that compromises uniform application. Additionally, oils with high viscosity may increase stickiness, whereas low-viscosity oils promote smooth spreading and uniform film formation<sup>5</sup>.

Therefore, the physicochemical nature of waxes and oils not only governs sensory characteristics but also plays a key role in determining the microstructure and chemical stability of lipstick formulations. Accordingly, the compositions of the wax and oil are critical to achieving optimal product performance. However, prior research has predominantly focused on sensory evaluation, with relatively few studies exploring the internal microstructure of lipsticks or correlating it with thermal and mechanical stability. This gap highlights the need for a

visualization-based analytical approach which enables examining the relationships between formulation components, microstructure, and material properties. Furthermore, a frequently reported issue in lipsticks is the phenomenon of "sweating", wherein oils migrate to the surface under fluctuating environmental or storage conditions. This phenomenon adversely affects both product stability and user satisfaction<sup>4</sup>. Despite its practical significance, the underlying physicochemical mechanisms and contributing conditions remain poorly understood.

In the present study, a freeze-substitution technique was optimized to enable direct visualization of the internal microstructure of lipstick formulations. Moreover, a cryogenic scanning electron microscopy (Cryo-SEM) was utilized to validate the freeze-substitution method for observing the oil–wax gel microstructure and image the oil-wax gel matrix at high resolution. While previous work lacked validation of freeze-substitution methods for cosmetic applications, this study employed Cryo-SEM to successfully visualize the internal microstructures in real lipstick samples, thereby establishing the analytical reliability and precision of the microstructure observation method. In addition, comparative imaging across formulations and spatial regions, including edge and middle region, revealed that wax melting point directly influences size of the wax crystals and strut morphology within the gel matrix, depending on the region of the lipstick. Furthermore, to investigate microstructural changes induced by sweating, lipstick samples stored at elevated temperatures that simulate storage-related stress were evaluated. Microstructural variations across different spatial regions of the lipstick were analyzed with thermal analysis, including thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), as well as mechanical characterization using a rheometer. The results revealed that sweating led to the collapse of the wax matrix, accompanied by shifts in thermal decomposition and melting/crystallization temperature, which is related to the crystallinity of the wax<sup>6,7</sup>. These findings provide critical insights for the design of lipsticks with enhanced thermal stability and sensory performance, and are expected to guide the development of high-performance formulations that ensure superior user satisfaction and robust physicochemical stability under diverse environmental conditions.

## 2. Materials and Methods

### 2.1. Lipstick fabrication

To prepare lipsticks with various formulations, the following waxes and oils were used. The waxes included ethylene/propylene copolymer wax, candelilla wax, sunflower seed wax, and synthetic wax. The oils used were octyldodecanol, caprylic/capric triglyceride, diisostearyl malate, and castor seed oil. Waxes and oils were mixed at a predetermined ratio and completely melted using a water bath maintained at 80 °C. The molten mixture was poured into lipstick-shaped metal molds and cooled at –20 °C for 20 minutes. The solidified lipsticks were stored at room temperature for 24 hours prior to the characterizations.

### 2.2. Microstructure observation

#### 2.2.1. Freeze-substitution method

A freeze-substitution technique was optimized to observe the microstructure of the oil–wax gel in lipstick formulations. Lipstick samples were sectioned according to spatial regions (edge and middle), rapidly frozen by immersion in liquid nitrogen for 1 minute, and fractured to observe the microstructure without deformation. The fractured samples were immersed in isopropyl alcohol (IPA, Sigma-Aldrich, USA) at room temperature for 1 hour to extract the oil phase. Subsequently, the samples were air-dried at room temperature and microstructures of the lipstick were analyzed using scanning electron microscopy (SEM, S-4800, HITACHI, Japan) after platinum (Pt) coating to prevent charging during imaging.

### 2.2.2. Cryo-SEM

To validate the reliability of the freeze-substitution method, lipstick samples were rapidly frozen in liquid nitrogen and fractured under cryogenic conditions. The internal microstructures were directly visualized using cryogenic field emission scanning electron microscope (Cryo-FESEM, Crossbeam 550, Carl Zeiss, Germany).

### 2.3. Preparation of lipstick with sweating

To investigate the effects of sweating lipsticks composed of octyldodecanol and ethylene/propylene copolymer wax containing 10 wt% wax was incubated at 45 °C for 7 days to induce oil exudation.

### 2.4 Thermal Analysis

Thermogravimetric analysis (TGA, TGA2, Mettler-toledo, Switzerland) and differential scanning calorimetry (DSC, Discovery DSC 2500, TA Instrument, USA) were conducted to evaluate the thermal behavior of lipsticks across different regions of the lipstick and under varying storage temperature conditions. TGA was performed to evaluate thermal stability of the oil-wax gel. Approximately 15 mg of each sample was placed in platinum crucibles and heated at 10 °C/min from ambient temperature to 600 °C under nitrogen atmosphere to provide the inert environment. DSC was used to evaluate the crystallization and melting behaviors of oil-wax gel. Approximately 15 mg the samples were heated from 10 to 100°C at a rate of 10 °C/min. After holding at 100°C for 5min, the samples were cooled at 10 °C/min from 100°C to 10°C.

## 3. Results

To evaluate the effect of wax and oil composition on the microstructure, thermal behavior, and mechanical properties of oil-wax gels, various combinations of waxes and oils were employed. Ethylene/propylene copolymer wax, candelilla wax, sunflower seed wax, and synthetic wax were used as the wax component and octyldodecanol, caprylic/capric triglyceride, diisostearyl malate, and castor seed oil were used as the oil component. **Table 1** presents the melting points of the waxes, which decrease in the order of ethylene/propylene copolymer wax, polyethylene wax, sunflower seed wax and candelilla wax. **Table 2** summarizes the viscosities of the oils, which decreases in the order of diisostearyl malate, castor seed oil, octyldodecanol, and caprylic/capric triglyceride.

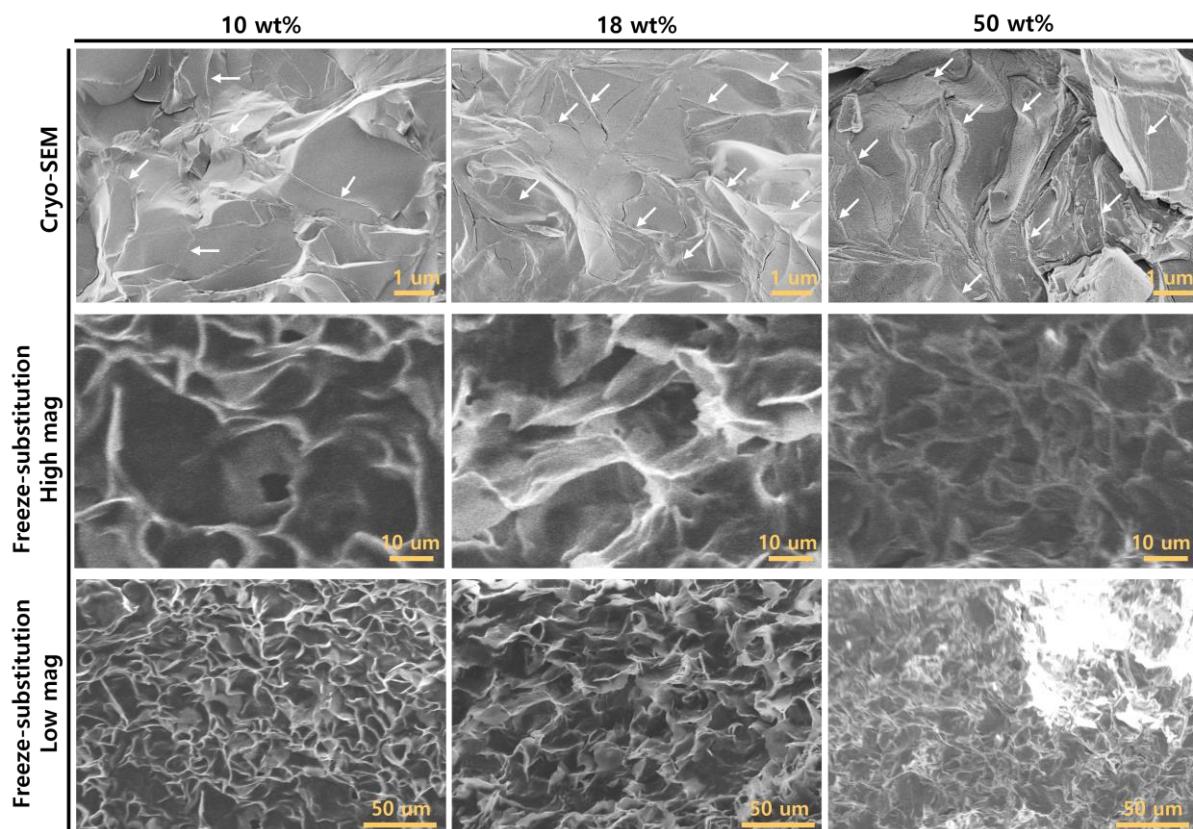
**Table 1.** Melting temperatures of the waxes

Wax	Candelilla	Sunflower seed	Polyethylene	Ethylene/propylene copolymer
Melting point (°C)	68-75	74-77	82-93	92

**Table 2.** Viscosities of the oils

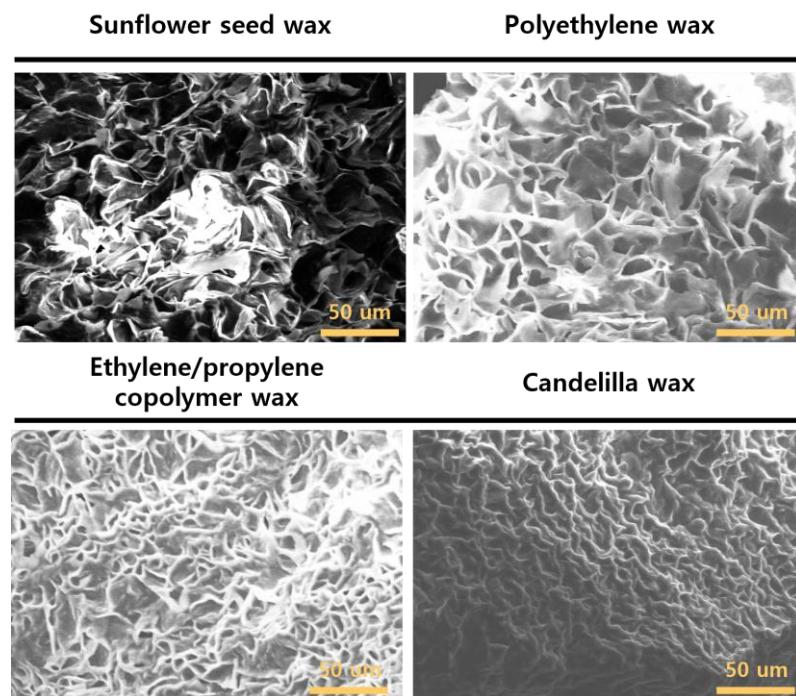
Oil	Caprylic/capric triglyceride	Octyldodecanol	Castor seed	Diisostearyl malate
Viscosity (mPa·s)	25-33	58-64	935-1100	1600-2200

To observe the microstructure of the oil–wax gels, oil need to be selectively removed while preserving the gel structure prior to SEM imaging. Therefore, a freeze-substitution method was optimized, and structural features across different regions of the lipstick such as middle and edge regions were visualized. To confirm accuracy of the optimized freeze-substitution method, Cryo-SEM was utilized to image the fracture surface and compare it with the freeze-substitution SEM images. **Figure 1** shows the microstructures of oil–wax gels with varying wax contents (10 wt%, 18 wt%, and 50 wt%), visualized via Cryo-SEM and freeze-substitution SEM. Cryo-SEM revealed a grain-like structure with interfacial boundaries (indicated by white arrows), whose density increased and sized decreased as the wax content increased. Freeze-substitution SEM showed the formation of a card-house-like wax network. Moreover, as the wax content increased, the size of voids within the card-house structure decreased. These observations were consistent with the images obtained from Cryo-SEM, confirming that the optimized freeze-substitution method is an appropriate technique for observing the microstructure of oil-wax gel systems.



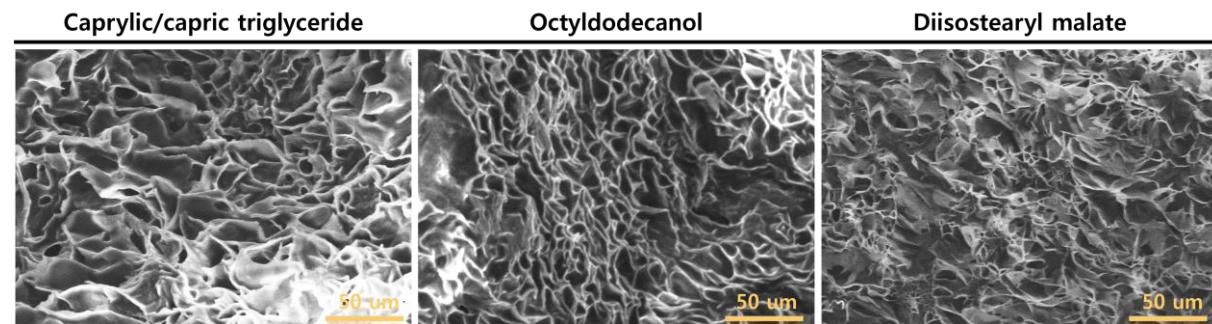
**Figure 1.** Cryo-SEM and freeze-substitution SEM images of oil–wax gels composed of octyldodecanol and ethylene/propylene copolymer wax at varying wax concentrations (10 wt%, 18 wt%, and 50 wt%).

The microstructures of oil–wax gels with different wax compositions were observed using the optimized freeze-substitution method. **Figure 2** presents the microstructures of gels formulated with octyldodecanol with different waxes. While the card-house structure was not observed in the gel containing candelilla wax, the card-house structure was clearly formed in gels with sunflower seed wax, synthetic wax, and ethylene/propylene copolymer wax. Moreover, the pore size and wax crystal dimensions of the card-house structure decreased in the order of sunflower seed wax, polyethylene wax, ethylene/propylene copolymer wax, which inversely correlated with the melting points of the waxes.



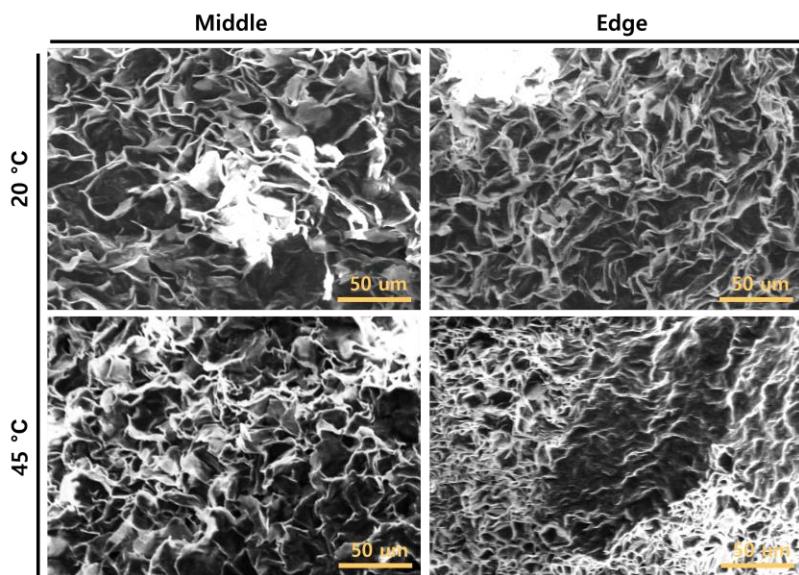
**Figure 2.** Freeze-substitution SEM images of oil–wax gels composed of octyldodecanol and different types of waxes (sunflower seed wax, synthetic wax, ethylene/propylene copolymer wax, candelilla wax).

**Figure 3** shows the microstructures of gels made from ethylene/propylene copolymer wax with various oils. The formulation with castor seed oil was too soft to analyze under the given conditions. In contrast, gels prepared with caprylic/capric triglyceride, octyldodecanol, and diisostearyl malate formed stable structures with well-defined card-house networks. Wax crystal size decreased in the order of caprylic/capric triglyceride, octyldodecanol, diisostearyl malate, inversely correlating with oil viscosity.



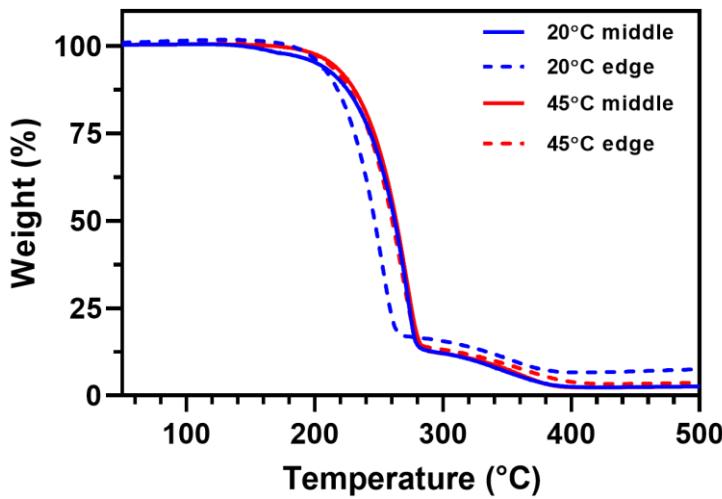
**Figure 3.** Freeze-substitution SEM images of oil–wax gels composed of ethylene/propylene copolymer wax and various oils (caprylic/capric triglyceride, octyldodecanol, diisostearyl malate). Formulation with castor seed oil did not form a stable gel structure.

The structural changes in oil–wax gels stored at elevated temperatures were also investigated across different regions of the lipstick, as shown in **Figure 4**. In the lipstick stored at 20 °C, both middle and edge regions maintained card-house structures, although crystals in the edge region were slightly smaller. In sample stored at 45 °C, the middle region showed smaller crystals compared to the middle region of the sample stored at 20 °C, and partial collapse of the card-house structure was observed in the edge region.



**Figure 4.** Freeze-substitution SEM images of different regions (middle and edge) of oil–wax gels composed of octyldodecanol and ethylene/propylene copolymer wax, stored at different temperatures (20 °C and 45 °C)

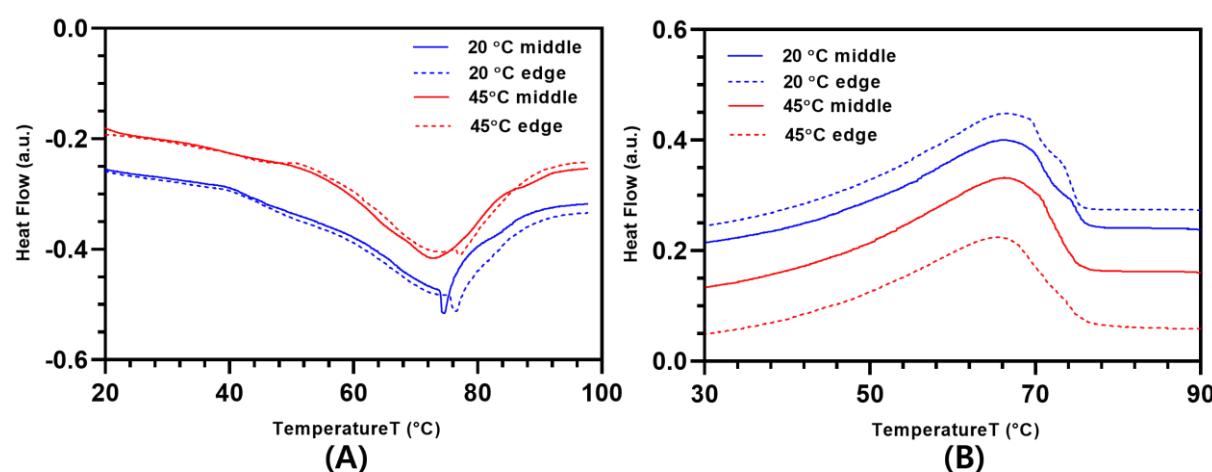
**Figure 5** displays the TGA profiles of oil–wax gels stored at different temperatures and sampled from the middle and edge regions. All samples exhibited a major weight loss between 200 °C and 300 °C, corresponding to the thermal decomposition of organic components.



**Figure 5.** TGA profiles of different regions (middle and edge) of oil–wax gels composed of octyldodecanol and ethylene/propylene copolymer wax, stored at different temperatures (20 °C and 45 °C)

**Figure 6(A)** shows the melting profiles of the oil–wax gels under different storage conditions and at distinct spatial regions (middle and edge). The edge region of the lipstick stored at 20 °C exhibited a higher enthalpy and melting peak temperature of 25.98 J/g and 76.34 °C, respectively, compared to the middle region of 24.37 J/g and 74.46 °C. In contrast, the samples stored at 45 °C exhibited relatively uniform enthalpy values between the middle and

edge regions with 25.63 J/g and 25.81 J/g, respectively. Moreover, both regions showed slightly higher enthalpy values compared to those stored at 20 °C, suggesting that high-temperature storage induced partial recrystallization, leading to an overall enhancement in crystallinity. Furthermore, the edge region of the 45 °C sample showed an higher peak temperature of 77.17 °C. **Figure 6(B)** presents the crystallization enthalpy and peak temperatures of oil–wax gels during cooling. The crystallization shows similar tendency with the melting process. Oil-wax gel stored at 20 °C exhibited a higher crystallization enthalpy of 25.90 J/g and a slightly higher peak temperature of 66.36 °C at the edge region compared to the middle region of 24.15 J/g and 65.76 °C respectively. In contrast, samples stored at 45 °C exhibited relatively uniform crystallization enthalpy between the middle and edge regions as of 25.39 J/g and 25.66 J/g. The results of melting and crystallization enthalpy, and peak temperatures of the oil–wax gels from different regions (middle and edge) stored at 20 °C and 45 °C, respectively, are summarized at **Table 3**.



**Figure 6(A)** shows the DSC profiles of different regions (middle and edge) of oil–wax gels stored at 20 °C and 45 °C, respectively.

**Table 3.** Melting and crystallization enthalpy, and peak temperature for different regions (middle and edge) of oil–wax gels stored at 20 °C and 45 °C, respectively.

	20 °C middle	20 °C edge	45 °C middle	45 °C edge
Melting enthalpy (J/g)	24.37	25.98	25.63	25.81
Crystallization enthalpy (J/g)	24.15	25.90	25.39	25.66
Melting peak temperature (°C)	74.46	76.34	72.72	77.17
Crystallization peak temperature (°C)	65.76	66.36	66.22	65.38

#### 4. Discussion

As shown in **Figure 1**, the optimized freeze-substitution method enabled reliable observation of the oil–wax gel microstructure without structural distortion, which was confirmed by

Cryo-SEM observation technique. Previous study has not validated the accuracy of freeze-substitution by using high-resolution Cryo-SEM, making this a novel methodological contribution. In addition, observations using Cryo-SEM and freeze-substitution SEM demonstrated that increasing wax concentration led to smaller sizes of wax crystal. An increase in wax concentration accelerates nucleation rate during the cooling process, resulting in the formation of higher number of wax nuclei and limiting crystal growth due to reduced molecular availability and spatial restriction<sup>8</sup>.

Microstructural analysis depending on the wax type revealed that card-house structures was not formed with the mixture of candelilla wax and octyldodecanol by the relatively low melting point of the candelilla wax (68–75 °C). Waxes with low melting points crystallize more slowly, form smaller crystals, and exhibit weaker intermolecular interactions, which hinder the formation of stable gel structures<sup>9-11</sup>. In contrast, sunflower seed wax, synthetic wax, and ethylene/propylene copolymer wax formed well-defined card-house structures as shown in **Figure 2**. The inverse relationship between melting point and crystal size suggests that waxes with lower melting point allow for slower cooling, enabling longer molecular rearrangement and thus the formation of larger wax crystals<sup>6</sup>.

As shown in **Figure 3**, castor seed oil failed to form a stable structure with ethylene/propylene copolymer wax by the high viscosity of castor seed oil, which weakens the wax crystal interactions. Moreover, solubility parameter of castor seed oil mismatches with the ethylene/propylene copolymer wax, which prevented the formation of a uniform phase, resulting in a soft oil-wax gel<sup>12</sup>. In contrast, gels formulated with caprylic/capric triglyceride, octyldodecanol, and diisostearyl malate maintained card-house structures with the decrease of wax crystal size as the oil viscosity increased. Oils with high viscosity restrict molecular mobility, thereby slowing crystal growth and producing smaller crystals. Additionally, high viscosity oils may reduce heat transfer during cooling, promoting supercooling and accelerating nucleation, which leads the formation of numerous crystals with small sizes<sup>10,13</sup>.

**Figure 4** illustrates the effect of temperature during storage on regional microstructure. The oil-wax gel stored at 20 °C exhibited card-house structures in both the middle and edge regions, while the size of wax crystals was smaller at the edge of the lipstick. The smaller crystal size at the edge was attributed to the faster cooling rate at the mold-wall interface during the molding process, which induced supercooling and increased the nucleation rate, thereby limiting crystal growth<sup>14</sup>. The oil-wax gel stored at 45 °C, the middle region showed smaller crystals than those at 20 °C, suggesting partial melting or reorganization of the wax crystal. The edge region showed structural collapse, which was attributed to thermal degradation of wax crystals at elevated temperatures<sup>15,16</sup>.

The DSC results during heating and cooling demonstrates the effect of storage temperature and spatial position on the crystallinity of oil-wax gels as shown in **Figure 6**. Oil-wax gel stored at 20 °C had higher enthalpy and peak temperature in the edge region compared to the middle which was attributed to faster cooling at the mold–wall interface during lipstick fabrication. The rapid cooling promoted supercooling and increasing the nucleation rate, thereby enhancing crystallinity at the edge<sup>17</sup>. Consistent with this, **Figure 4** shows that the wax crystals in the edge region of the 20 °C stored sample were smaller, indicating a higher nucleation density. Meanwhile, storage at 45 °C led to a slight increase in enthalpy across both regions, accompanied by homogenization of the crystallinity between the middle and edge. Especially edge region exhibited a notable increase in melting peak temperature. This behavior can be explained by partial recrystallization during high-temperature storage, where

increased molecular mobility facilitated structural reorganization and crystal growth, resulting in a more thermally stable and uniform microstructure<sup>18</sup>.

## 5. Conclusion

This study demonstrated the influence of wax and oil composition, spatial region, and storage temperature on the microstructure, thermal behavior, and crystallinity of oil–wax gel-based lipstick formulations. By optimizing a freeze-substitution technique and validating it with Cryo-SEM, we successfully visualized the internal microstructures without distortion. Increased wax concentration led to smaller wax crystals due to higher nucleation rates and limited crystal growth. The type and melting point of waxes significantly affected the formation of card-house structures, with lower melting point waxes allowing the formation of larger crystals, while highly viscous oils limited crystal growth and phase uniformity. Regional differences in structure were also observed, which edge regions cooled more rapidly during fabrication, leading to enhanced nucleation and crystallinity compared to middle regions. Thermal analysis further confirmed these trends, with the edge region of the 20 °C sample showing higher enthalpy and peak temperature, while high-temperature storage of 45 °C promoted partial recrystallization and structural homogenization. Consequently, these findings provide critical insights for tailoring lipstick formulations to achieve improved thermal stability, microstructural integrity, and overall user satisfaction under various environmental conditions.

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