Evaluation of post-application rheological changes in cosmetics using a novel measuring device: Relationship to sensory evaluation

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Synopsis

A novel measuring device was developed to study the changes in the rheological properties of cosmetics after application, and the relationship of the results to subjective sensory evaluation was examined. The device can measure the frictional force (0.001-0.1 N) between a probe and the surface of the sample stage, which moves reciprocally at a constant speed (1-8 cm/s). Two different probes (block-type and roller-type) were used under a constant load (0.02-0.3 N). The relationship between frictional force and the spreading resistance was examined using massage gels with the block-type probe. The changes in the measured force with the block-type probe correlated well with the changes in the spreading resistance experienced upon massaging the skin. This indicates that the spreading properties of cosmetics can be evaluated with this device. With respect to stickiness, no clear correlation was found between the measured frictional force and the skin **sensation of moisturizing creams and essences using the block-type probe. With the roller-type probe, however, the stickiness of cosmetics due to thickening of polymers, oils, and other ingredients after application to the skin was reflected in the friction curves. The frictional force at 10 min correlated with the post-application stickiness.**

INTRODUCTION

The sensation produced by application to the skin is one of the most important properties of cosmetics, in addition to the long-term physical and physiological effects. With the advancement of formulation technologies, an understanding of the factors underlying skin sensation has become more important than ever. In the actual development of cosmetics, sensory analysis is used to find the best formulation for a certain product from a number of candidates. However, this procedure is subjective by nature, can be timeconsuming, and sometimes does not afford clear-cut results. To overcome these prob-

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lems, Aust et al. (1) developed a more objective and controlled descriptive method. This **method involves a trained descriptive panel that is capable of identifying and defining attributes of test products through the use of reference materials and is able to measure the relative intensities of product attributes on a numerical scale. The descriptive method is able to yield consistent data compared with less objective consumer evaluation, but it still has difficulties in evaluating large numbers of test samples.**

As an alternative to sensory evaluation, objective evaluation methods by means of instrumental measurements have been studied using conventional rheometers (e.g., the cone/plate viscometer or reciprocation-type rheometer) (2-5) or measuring systems that simulate the application processes of cosmetics (6–8). The use of *in vivo* friction mea**surements to evaluate skin condition has also been studied by many investigators (9- 13). These measurements basically depend on the detection of the force conducted to the probe through a thin film of the sample, although there are differences in probe geometry.**

Despite the above research, instrumental predictions of subjective sensation on the skin are still rarely used in the actual development of cosmetics. This is partly due to the intrinsic technical difficulties of simulating human sensation processes, which are highly complex and sensitive. The reported procedures may be inadequate to obtain the necessary information, since many sensory attributes are used in the subjective evaluation process, such as spreadability, stickiness, richness, dewiness, greasiness, and so on. Moreover, most studies focused on the initial properties of cosmetics and only a few have involved successive measurements (e.g., 0, 5, 10, and 30 min after the application). **Continuous monitoring of the properties of cosmetic samples after application may be the most effective approach to instrumental prediction of subjective sensation.**

In this paper, we describe a novel rheological measuring device developed to study the rapid changes in the physical properties of cosmetics after application, and we discuss the relationship of the results to the results of sensory evaluation. The characteristics of the system are described in detail, focusing on the detection of the changes in spreadability and stickiness after application.

EXPERIMENTAL

STRUCTURE OF THE MEASURING DEVICE

Figure 1 shows the novel rheological measuring device used in this study. The device can measure frictional force (0.001-0.1 N) between a probe and the surface of the sample stage, which reciprocates horizontally at a constant speed (amplitude: 30 mm; speed: **1-8 cm/s). Two different probes (block-type and roller-type; Figure 1 C, D) were used with a constant load (0.02-0.3 N). The block-type probe has a curved contact surface** (curvature radius: 10 mm) covered with polyimide film. The roller-type probe has an aluminum roller (diameter: 10 mm) equipped with ball bearings (MF63, NSK, Tokyo). **The bearings were lubricated with low-viscosity oil (decamethylcyclopentasiloxane, Shin-Etsu Chemical, Tokyo) each time, just before the measurement, and the baseline friction (the friction measured without sample application) was less than 0.001 N at 0.05 N probe load. The surface of the sample stage was covered with polyimide film. The detected** force was conducted through a leaf spring $(10 \times 25 \times 0.2 \text{ mm})$, phosphor bronze) to a

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Figure 1. Measuring device used in this study: measuring device (A), diagram of the device (B), block-type probe (C), and roller-type probe (D). Scale bars under (C) and (D) represent 10 mm.

pair of strain gauge sensors and then converted to an electric signal (-10-10 V) with an amplifier. The electric signal was digitized through an A/D converter and sent to a computer for data processing. Ablock diagram of the device is shown in Figure 2.

MEASUREMENT PROCEDURE AND DATA PROCESSING

A sample (5-40 pl) was applied to the center of the stage, and the probe was placed on the sample, perpendicular to the surface of the stage. Then, reciprocation was applied immediately and continued for 3-15 min. To avoid uneven spreading of the sample on the stage, the probe was pulled up above the stage each time for approximately 5mm

Figure 2. Block diagram of the measuring device.

at the end of the sweep area and placed again at the start of measurement in the reverse direction [probe lifting function, as reported by Date and Inaba (14)]. During the measurements, ambient temperature and relative humidity were kept at 25^oC and 50%, **respectively. The temperature of the sample stage was kept at 32øC. For calibration, a certain force (0.01-0.1 N) was applied to the probe horizontally by a weight and a** pulley. To compensate for zero-point shifting during measurement, the measured fric**tional force in each reciprocating process was calculated with the following equation:**

$$
F = \frac{Fc}{Vc} \times (Vf - Vb) \div 2
$$

where Fc is the force applied at calibration, and Vc , Vf , and Vb are the voltages detected **at calibration, at the forward process, and at the backward process, respectively.**

SAMPLES USED FOR MEASUREMENT

Various types of commercial cosmetic emulsions were used for measurement. These include massage creams, moisturizing creams, and essences (O/W-type and W/O-type emulsions that can give various skin sensations). Standard cosmetic cream formulations that contained conventional cosmetic ingredients were also used for measurement. Viscosity standard oils were purchased from Toki Sangyo (Tokyo, Japan).

SENSORY EVALUATION OF COSMETIC SAMPLES

Skilled panels carried out evaluation of cosmetic samples to assess their sensory attributes, including spreading, stickiness, absorption, and richness. The panels graded the test samples on a scale from -3 to $+3$ against the selected references for each attribute. **Special attention was paid to the changes in spreadability and stickiness during application of the cosmetics.**

MEASUREMENT OF SPREADING VALUE

Spreading values of oils were measured according to the method developed by Zeidler (15), with slight modifications. Oil samples (5 lnl) were placed on the forearms of human volunteers. The oil that spread spontaneously on the skin was blotted on a piece of wax paper to measure the area of spread. The area at 5 min after application was used as the spreading value. During the experiments, ambient temperature and relative humidity were kept at 25øC and 50%, respectively.

RESULTS AND DISCUSSION

MEASUREMENT USING THE BLOCK-TYPE PROBE

When cosmetic emulsions are applied to skin, they usually spread evenly and form thin films on the skin. To simulate this process by rheological measurement, it is important **to spread samples evenly on the sample stage. In preliminary experiments using conventional friction analyzers that consist of reciprocating sample stages and load cell sensors, however, the probe squeezed and piled up the applied samples at either end of the sweep area during reciprocation. To avoid this phenomenon, our measuring device**

was equipped with a probe-lifting function as described in Materials and Methods. Improved spreading status of samples on the stage was obtained with this function (Figure 3).

Figure 4 shows the results of measurements with the block-type probe using O/W-type massage gels (Table I) that drastically change their spreading properties during massaging. The changes in the measured frictional force reflected the changes in spreadability experienced during massaging. Since the measured frictional force decreased at almost the same rate that the spreadability decreased on the skin, the device could reproduce and detect the same phenomenon that occurred on the skin.

To examine the relationship between the measured frictional force and the viscosity of samples, viscosity standard oils were used for measurement (Figure 5). A high correlation between the frictional force and the viscosity was found over a broad range, with only a minor dependence on the sample volume, which possibly could decrease over time due to evaporation if conventional cosmetics are measured. In addition, large differences in viscosity resulted in relatively small differences in friction (e.g., a 70-fold increase in viscosity yielded only an 8- to 9-fold increase in friction). This is due to the variation of sample thickness according to viscosity. The relation between the sample thickness and viscosity made it possible to detect the spreading changes that arise from large viscosity changes without changing the attenuation of the strain gauge amplifier. This is favorable for detection of dynamic changes occurring on the skin, since changing the attenuation of the amplifier is generally associated with zero-point shifting in measurement. Conversely, it may become more difficult to detect changes in skin sensation that arise from small viscosity changes.

With respect to another important sensory attribute, namely stickiness, no clear corre**lation was found between the results of the sensory evaluation and the frictional force of conventional moisturizing creams measured with the block-type probe (Figure 6A). The frictional force with the rich, sticky W/O-type cream was smaller than the force of the other two (light, non-sticky) creams. The frictional forces fell to almost the same level** (about 0.01 N) 10 min after the beginning of measurements, irrespective of the sticki**ness properties of the measured samples. The baseline friction might hinder the evaluation of stickiness.**

MEASUREMENT USING THE ROLLER-TYPE PROBE

To evaluate the stickiness of cosmetics, another type of probe, i.e., the roller-type probe, was developed and used for the measurement of the same moisturizing creams described

Figure 3. Improved spreading of samples on the stage owing to the probe-lifting function. Sample distribution after 1 min of reciprocation with probe lifting (A) and without lifting (B). A W/O-type foundation was used as a sample. Sample volume was 20 µl; probe load, 0.05 N; reciprocation, 2.4 cm/s. The **scale bar in (B) represents 10 mm.**

Figure 4. Measurements of massage gels using the block-type probe. Sample volume was 20 µl; probe load, **0.05 N; reciprocation, 7.2 cm/s. The open (O) and closed (** \bullet **) circles represent the frictional force of gels A and B, respectively. The formulae of gels A and B are given in Table I.**

Component	Massage gel A	Massage gel B		
Deionized water	11.32	15.75		
Liquid paraffin	25.5	21.07		
Trioctanoin	20	20		
Dimethyl polysiloxane	15	15		
Glycerol	5	5		
Sorbitol	12.6	12.6		
PEG 400	5	5		
Sodium alginate	0.03	0.03		
Dimethicone copolyol	0.5	0.5		
PEG-60 hydrogenated castor oil	0.5	0.5		
Sucrose stearate	2.4	2.4		
Poloxamer 184	2	2		
Sodium methyl cocoyl taurate	0.15	0.15		
Spreading profile on the skin	Simple decrease (about $45 s$)	Decrease (about 65 s) after a temporary increase		

Table I

above. The frictional force of the rich, sticky W/O-type cream was higher than that of the light, nonsticky W/O-type cream (Figure 6B). Similarly, the frictional force of the rich, sticky O/W-type cream was higher than that of the light, non-sticky W/O-type cream. These results are in accordance with the stickiness properties on skin.

To study the properties of this measuring system, typical ingredients that cause stickiness of cosmetics (oils, humectants, and thickening polymers) or those added in standard formulations were used as samples. Figure 7A shows the results of measurements of oils commonly used for cosmetic products. The frictional forces immediately reached con-

Figure 5. Relationship between sample viscosity and friction using the block-type probe. Viscosity standard liquids (5–40 µl) were measured under a probe load of 0.05 N and with reciprocation at 2.4 cm/s. The open circle (\circ) represents the amount of sample (5 µl) that was applied on the stage; the closed triangle (\blacktriangle), 10 pl; the open square (\square), 20 pl; the open triangle (\triangle), 40 pl. Apparent viscosity was measured at 32°C.

Figure 6. Measurements of typical. O/W-type and W/O-type moisturizing creams using the block-type type probe (A) and the roller-type probe (B) . The closed circle (\bullet) represents the rich, sticky O/W -type cream; the closed triangle (\triangle) , the rich, sticky W/O-type cream; the open circle (\bigcirc) , the light, non-sticky O/W-type cream; the open square (\square), the light, non-sticky W/O-type cream. The probe load and recip**rocation were 0.05 N and 2.4 cm/s, respectively. Sample volume was 20 pl in (A) and 10 pl in (B).**

stant values; viscous oils that give a rich skin sensation, such as pentaerythrityl tetraoctanoate, gave high friction, whereas light oils, such as dimethyl polysiloxane, gave low friction. Figure 7B shows aclear correlation between the frictional force and the spreading value, which can be defined as skin surface area covered spontaneously during a certain period of time (15). This is an interesting result, because the spreading values of oils have been reported to have a close relation to the sensation that the oils produce on the skin, and to be a useful index in selecting oils for cosmetic formulations (16).

Figure 8 shows the results of measurements of moisturizing gels containing various humectants (Table II). A large frictional force was observed during the first 1–2 min **when the roller of the probe slipped on the reciprocating stage. At that time, the probe**

Figure 7. Measurements of oil components uing the roller-type probe and its relation with spreading value. The measured friction for pentaerythrityl tetraoctanoate (\bigcirc) , trioctanoin (\blacktriangle) , and dimethyl polysiloxane (\Box) is shown in (A). The relation between the friction and spreading value of oils is shown in (B).

Figure 8. Effect of humectants on friction using the roller-type probe. Moisturizing gels (10 µl) containing **various humectants (shown in Table lI) were measured under a probe load of 0.05 N and with reciprocation** at 2.4 cm/s. Friction curves for gels containing 10% glycerol (\bullet), 10% DPG (dipropylene glycol, \triangle), and 10% BG (1,3-butylene glycol, \Box) are shown in (A). The detected friction levels at 10 min are summarized in (B).

spread the sample over the sweep area. Differences in the frictional force gradually appeared as the slipping of the roller disappeared over time. At 10 min, the frictional force with the gel containing 10% glycerol was 5-7 times higher than that with the gel containing 10% 1,3-butylene glycol or 10% dipropylene glycol. The gel containing 5%

Figure 9. Detection of stickiness phenomena of thickeners measured using the roller-type probe. Moisturizing gels (10 µl) containing various thickeners (0.5%) shown in Table III were measured under a probe **load of 0.05 N and with reciprocation at 2.4 cm/s. The closed circle (O) represents the gel containing** carbomer; the open circle (\circ) , methyl cellulose; the open triangle (\triangle) , carboxymethyl cellulose; the open square (\Box) , xanthan gum.

Figure 10. Observation of the contact region between the roller and the sample stage. Photos were taken at 2 min with a non-sticky sample (A) and a sticky sample (B). The non-sticky sample contained carbomer, while the sticky sample contained xanthan gum as a thickener (Table III). The scale bar indicates 5 mm.

To investigate the relationship between the frictional force and the stickiness score obtained by sensory evaluation, various types of commercially available moisturizing creams were used as samples. The initial friction, the maximum friction, and the average friction (0-3, 3-6, and 6-9 min) were used for statistical analysis (Table IV). Contrary to expectation, no correlation was found between stickiness and the friction at the initial stages of the measurements, including the initial value, the maximum value, and the average value over 0-3 minutes. However, a clear correlation was observed between the stickiness after application and the average friction in the mid-to-late stage of the measurements (3-6 and 6-9 min). In addition, these mean values were clearly related to other sensory attributes, including spreading or absorption during application and richness or hydration after application. Since the frictional force reached a constant value at 6 min, the friction at the steady state is considered to provide valuable information for the evaluation of cosmetics. This finding implies the importance of studying

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Figure 11. Effect of probe load on the frictional force. The moisturizing gel (10 µl) containing xanthan gum **shown in Table III was measured with the roller-type probe under reciprocation at 2.4 cm/s. The open circle** (C) represents 0.02 N; the closed circle $(•)$, 0.05 N; the open triangle $(•)$, 0.01 N.

Table IV Correlation Coefficients Between the Results of Rheological Measurement and Sensory Evaluation

Rheological measurement ^a (Friction)	Sensory evaluation						
	During application			After application (30 min)			
	Spreading	Absorption	Freshness	Stickiness	Richness	Hydration	
Initial value	0.41 ^b	-0.25	-0.32	0.44	0.51	0.41	
Maximum value	0.56	-0.26	-0.42	0.54	0.62	0.53	
Average $(0-3$ min)	0.48	-0.17	-0.35	0.50	0.57	0.52	
Average $(3-6$ min)	$0.81*$	-0.57	$-0.71*$	$0.80*$	$0.84*$	$0.74*$	
Average $(6-9$ min)	$0.78*$	$-0.66*$	$-0.70*$	$0.81*$	$0.82*$	$0.69*$	

^a Ten microliters of moisturizing cream (10 samples) was measured under the probe load of 0.05 N and with **reciprocation at 2.4 cm/s.**

b Correlation coefficients between the rheological measurement data (friction) and the results of sensory **evaluation.**

* Statistically significant correlations ($p < 0.05$).

rheology and tribology in the steady state. This should also be important in other rheological measurements of cosmetics.

With respect to the initial stage of the measurements (0–3 min), friction peaks were **commonly observed with O/W-type creams, whereas simple decreasing curves without peaks were observed with W/O-type creams. Generally, an increase in the inner-phase ratio leads to an increase in sample viscosity, since the inner phase acts as a rigid sphere and a larger force is required for the outer-phase deformation. After application to the stage, the viscosity of O/W emulsions increases due to the increased inner-phase ratio as a result of evaporation of the outer phase (the water phase), and subsequently decreases due to the decrease in the inner-phase ratio (the water phase) after the phase transition.**

In contrast, the viscosity of W/O emulsions decreases due to the decrease in the innerphase ratio. Therefore, these changes in viscosity probably lead to the characteristic friction curves of O/W and W/O creams in the early stage of measurement. In some cases, frictional curves at this stage can be directly attributed to stickiness before drying, as we have shown in measurements with model formulations containing various thickeners. However, many factors, including rotation speed of the roller and the spreading status of sample, should be considered upon the measurement of more complex formulations, since they could differ greatly among samples at the initial stage. Optical observation during the measurements should provide useful information for interpreting the frictional data.

Our results clearly demonstrate that our newly developed rheological measuring device can be used to predict the changes in sensation following the application of cosmetics, such as the spreading change of massage gels and the stickiness induced by thickening of polymers in moisturizing gels. The new rheological measuring device developed in this study has structural similarities to conventional reciprocation-type rheometers used in other reports (5,6), in that both have reciprocating sample stages and strain gauge sensors. It has, however, a unique combination of features: the sensitive sensor, the insensitivity to zero-point shifting, the probe-lifting function for better sample spreading on the stage, and the novel roller-type probe. Stickiness after application, which often determines consumer preference for cosmetic products, was well reflected in the frictional force with the roller-type probe, rather than with the block-type probe. Regarding evaluation of stickiness, Iida et al. (7) reported measurements of emulsions with a tensipresser, using a multipoint biting method. Their device continuously detects the force in the repeated compression and decompression of samples on the stage to a decreasing thickness (0.2 mm at the start and 0. ! mm at the end of measurement) with a flat plunger. This type of measurement depends on the contact of two flat surfaces. As opposed to this, our system with the roller-type probe depends on the contact between the roller and flat surface, which is more controllable. Moreover, the frictional force in each reciprocation process is calculated from a large pool of detected force data sampled during a relatively long time (about ! s), instead of at an instant in the decompression process. These factors presumably contribute to the high reproducibility of the method. The present method can be performed with a small amount of sample in a short time, and so should be suitable for routine work. In this study, polyimide was selected from among various plastic materials and used as an artificial skin, but future work will be directed at developing a stage material that more closely resembles human skin.

Although we have shown the usefulness of our novel device, it is important to emphasize that the balanced use of various evaluation methods is necessary in the actual development of cosmetic products. For example, our method would be especially suitable for the screening of prototype formulations or ingredients. Once candidates are selected, descriptive sensory evaluation would play an important role, since it brings reliable information about various sensory attributes. Consumer evaluation also provides valuable information on consumer perception, which is not readily accessible with other evaluation methods.

CONCLUSIONS

In this study, a novel rheological measuring device was developed to measure the rheological changes in cosmetics after application. The characteristics of the device were **investigated with two different probes (block-type and roller-type). With the block-type probe, the spreading change of massage gels was clearly related to the detected frictional force, which correlated with the sample viscosity. On the other hand, the stickiness properties of cosmetics were well reflected in the force detected with the roller-type probe. The frictional force at the steady state correlated with the post-application stickiness, as well as spreading or absorption during application, and richness or hydration after application. We conclude that this measuring device can provide useful information for the development of cosmetic formulations.**

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