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"In vivo and in vitro study on the long-lasting makeup of biomimetic film-forming compositions in foundation"

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1. Introduction

Long-lasting wear performance is a key indicator of the core competitiveness of foundation. Poor makeup longevity and makeup wear-off are the main pain points for consumers when purchasing and using foundation. Research showed that the film-forming system in formulations directly determines the foundation's adhesion to skin surface, environmental interference resistance and dynamic stability. Film-forming systems constructed with single type film-forming agents often face limitations due to their molecular structures and characteristics, manifesting as insufficient membrane flexibility, weak environmental resistance or poor adaptability to dynamic deformation. In high temperature and high humidity environments or situations where makeup or masks are worn for a long time, such formulas containing only single film-forming agent are prone to membrane layer rupture caused by sebum secretion, mechanical friction or moisture migration, leading to lack of staying power, powder accumulation or uneven makeup appearance.

In recent years, biomimetic materials science has provided a new research approach for innovative cosmetic formulations. Bird feathers exhibit excellent mechanical properties and environmental adaptability due to their unique hierarchical structure (Figure 1). Rachis provides mechanical support as a rigid skeleton. Barbicels form cross-linked fillers which make the feathers more hydrophobic. And barbules achieve stability of the feather structure through the mechanism of interlocking and connection.

The multi-dimensional and multi-level collaborative structure of feathers inspire us to propose the design concept of feather biomimetic membrane (FBM). By simulating the multi-level structural characteristics of feathers, a gradient film-forming system is constructed to break through the functional bottleneck of traditional single film-forming agents. FBM is mainly composed of three types of film-forming components compounded together. The rigid hard membrane form a rigid three-dimensional mesh structure by simulating the structure of large feather branches, which make the foundation more stable on the skin, thereby improving its durability and color migration resistance. The flexible soft membrane is used to construct a surface functional layer, its low surface energy characteristics can simulate the hydrophobic and oleophobic ability of small feather branches, while improving the adherence of foundation. The silicone elastomer with dynamic cross-linking properties is used to fill the gaps between

the membrane, which make the combination of hard membrane and soft membrane more tightly to maintain the integrity of the film-forming system.

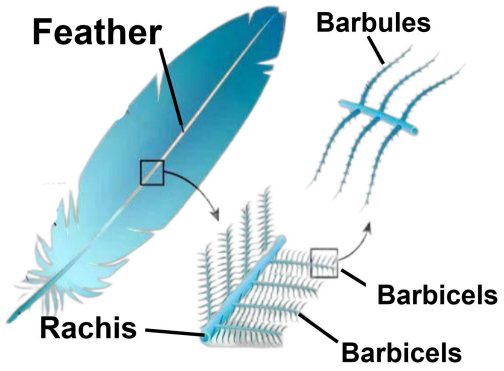


Figure 1. Schematic diagram of bird feather structure

This study combines in vitro and in vivo testing to explore the advantages of film-forming performance of FBM. In terms of adhesion and homogeneous degree, we evaluated through the change of surface roughness. In terms of film-forming stability, we evaluated the tensile resistance, sebum resistance, sweat resistance and friction resistance of FBM. And we compared the differences in these film-forming performance between FBM and single film-forming agent, combination of hard membrane and soft membrane. Secondly, through human clinical testing, we evaluated the long-lasting wear performance of water resistance, sweat resistance and friction resistance of FBM in practical formula applications. This study not only provides a scientific basis for the performance of FBM, but also offers innovative new solutions for the development of foundation with the long-lasting wear efficacy.

2. Materials and Methods

2.1 Materials

Trimethylsiloxysilicate (Wacker, Germany), Dimethicone/Vinyl Dimethicone Crosspolymer (BATAI, China), Polymethylsilsesquioxane (Wacker, Germany), Artificial sweat and artificial sebum (Shenzhen Zhongwei,China), PMMA plate (WENEOS, France).

2.2 Equipment

Scanning electron microscope (Hitachi, Japan), Skin magnifier PC35 (Courage+Khazaka, Germany), Skin color testing probe Colorimeter CL440 (Courage+Khazaka, Germany), Facial imaging system Vplus (Fuhuan, Shanghai).

2.3 Evaluation of membrane adhesion and homogeneous degree

Trimethylsiloxysilicate (TMS) was selected as hard membrane, Polymethylsilsesquioxane (PMS) as soft membrane, Dimethicone/Vinyl Dimethicone Crosspolymer (DVDC) as silicone elastomer. As shown in Table 1, these ingredients were added in different proportions to the base formulation without other film-forming agents. The base formulation consisted of 6.0% PEG/PPG-18/18 Dimethicone, 0.5% Polyglyceryl-3 Diisostearate, 10% Toner (contains CI 77891, CI 77492, CI 77491, CI 77499), 30% Cyclopentasiloxane, 0.5% Tribehenin, 0.5% Disteardimonium Hectorite, 1.0% Magnesium Sulfate and the remainder were made up of water.

PMMA plate was considered as artificial skin and used its rough texture to simulate the surface texture of the skin, each group of samples (according to Table 1) was tested in parallel in three areas. Samples from different groups were applied to PMMA plate and form a

film after standing for 20mins. Before and after application of samples, texture maps of the surface of plate were captured using skin magnifier PC35. The linear light intensity was analyzed using Image Pro Plus[1]. This value indicates the roughness of test surface. If the roughness after the application of samples is smaller, it indicates that the surface of PMMA plates is more homogeneous and smooth, and if the difference in roughness before and after the application of samples is close, it means that the adherence of samples are more better.

Table 1. Mass percent of film-forming agent added to the base formulation

Component	Base	Gourp1	Gourp2	Gourp3	Gourp4	Gourp5	FBM
TMS	0.0%	10.0%	0.0%	0.0%	4.0%	4.8%	4.0%
PMS	0.0%	0.0%	10.0%	0.0%	6.0%	7.2%	6.0%
DVDC	0.0%	0.0%	0.0%	2.0%	0.0%	0.0%	2.0%

2.4 Evaluation of membrane stability: sweat resistance, sebum resistance and friction resistance

Samples from different groups (according to Table 1) were applied to PMMA plate at the dosage of 2.0mg/cm^2, and let it stand for 20mins to wait for membrane formation. After collected the L* value of the surface of PMMA plate with Colorimeter CL440, PMMA plates were treated by three ways. Each groups were tested for three areas in parallel.

1.Artificial sebum were applied to the surface of test sample at a dosage of 0.27mg/cm^2 (this dosage is the amount of sebum produced in 8h which based on the average sebum production rate of adults being 1mg*10 cm^{-2} *3h^{-1}[2]) to interfere with the stability of samples, then tested the L* value after waited for 20mins.

2.Artificial sweat was added for 1ml to the surface of samples and infiltrate samples for 1 min, then tested the L* value after waited for 20mins.

3.L* value was tested after rubbing the sample surface with the same strength by using dry tissue paper.

Some studies evaluated the film-forming performance of sunscreen by using PMMA plate[3], and the changes in L* value were also commonly used for evaluating the long-lasting wear efficacy[4]. Similarly, in this experiment, the L* value of PMMA plates coated with different samples before and after different treatments were analyzed to evaluate the long-lasting wear of samples against interference from sebum, sweat and friction. The smaller the change in L * value before and after interference, the better the long-lasting wear of samples.

2.5 Evaluation of membrane stability: tensile resistance test

TMS (hard membrane), PMS (soft membrane) and DVDC (silicone elastomer) was dissolved in Cyclopentasiloxane according to different ratios in Table 2 to prepare different samples.

Table 2. Mass percent of film-forming agent dissolved in Cyclopentasiloxane

Component	Gourp1	Gourp2	Gourp3	Gourp4	Gourp5	FBM
TMS	20.0%	0.0%	0.0%	8.0%	9.6	8.0%
PMS	0.0%	20.0%	0.0%	12.0%	14.4	12.0%
DVDC	0.0%	0.0%	4.0%	0.0%	0.0%	4.0%

Samples from different groups were taken in 200μL and applied them onto a 10cm conductive adhesive, and placed the conductive adhesive in oven with temperature set to 60°C to dry. Then the dried conductive adhesive were stretched by 1mm with the same force and placed the conductive adhesive under scanning electron microscope to capture the

surface morphology of samples which tend to observe the integrity of the membrane formation.

2.6 Human clinical testing

Thirty subjects aged 24-38years were screened and the test meets the requirements of the Declaration of Helsinki. The test sample was foundation containing only FBM as the film-forming agent. The test environment was a constant temperature of 21±1°C and constant humidity of 50±10%RH.

The test consisted of two parts.In the first part, two test areas were marked within 3cm×3cm area on the inner side of the forearm, and foundation was applied to test areas at a dosage of 2.0±0.1mg/cm², waited for forming a film for 20mins, and L*, a* and b* value were collected of test areas. Then the test areas were rinsed at a fixed flow rate for 15s by using distilled water preheated to 36-38°C and artificial sweat. L*, a* and b* value of test areas were tested after the interference of water and sweat, then △E value was calculated.

In the second part, makeup tool was used to apply makeup on subject's face, and then wore the mask and repeatedly took it off 10 times within 1h to simulate the friction between the mask and face in daily life. Subsequently, Vplus was used to capture the facial images after applying the makeup and after wearing the mask for 1h. Images were analyzed using software to obtain indicators of the facial pore area, skin texture and skin homogeneity[5].

2.7 Data analysis

The results were expressed as Mean±Stdev. Difference was calculated based on the formula X_{after}-X_{before}. Change rate was calculated based on the formula (X_{after}-X_{before})*100%/X_{before}. Comparisons between groups were performed using two-tailed t-test and Wilcoxon signed-rank test, and $p<0.05$ was considered as a significant difference.

The formula for calculating △E value in vivo test is as follows, with reference to the group standard “Evaluation of Makeup Holding Effect of Base Makeup”, and the average value of △E < 2 indicates that the test samples have a better makeup-holding performance[6,7].

$$\triangle E = \sqrt{\left(L^*_{Tn} - L^*_{Ti}\right)^2 + \left(a^*_{Tn} - a^*_{Ti}\right)^2 + \left(b^*_{Tn} - b^*_{Ti}\right)^2}$$

Ti represents the skin parameter measured immediately after using foundation in test area and Tn represents the skin parameter measured after water and sweat interference in test area.

3. Results

3.1 Evaluation of membrane adhesion and homogeneous degree

Results (Table 3) showed that the surface roughness of FBM group were lower than base group and single film-forming agent group after coating the test samples, which indicated that the samples coated with FBM have better homogeneous degree on PMMA plates. It can also be seen from the thermal map of PMMA plates that in FBM group, there were relatively few red and yellow areas that represent significant differences compared to the original state (Figure 2).

In addition, the surface roughness increased significantly compared with that before the sample was applied, but difference of surface roughness of each test group is smaller than base group, which indicated that film-forming agent in sample helps foundation better fit the surface of PMMA plate. The difference and change rate of surface roughness in FBM group were the smallest, which indicated that the change of micro-texture of PMMA plate surface before and after application of FBM was the smallest, and the sample was more conforming.

Table 3. Results of surface roughness of different groups

Group	Surface roughness/a.u.			Difference of surface roughness/a.u.	Change rate of surface roughness/ %
	Before sample applied	After sample applied	P value		
Base	23.87±4.43	44.89±2.4	0.029*	21.01±6.33ns	88.06
10%TMS	22.22±3.13	40.45±2.1	0.025*	18.23±5.08ns	82.04
10%PMS	24.14±0.81	42.6±5.08	0.018*	18.46±4.34ns	76.47
2%DVDC	24.4±2.76	43.27±3.43	0.017*	18.87±4.32ns	77.34
4%TMS+6%PMS	21.2±1.79	38.47±2.01	0.002*	17.27±1.32ns	81.46
4.8%TMS+7.2%PMS	23.86±2.11	41.44±2.69	0.007*	17.57±2.51ns	73.68
FBM	24.29±2.93	38.55±4.65	0.026*	14.26±4.08	58.71

* means that there is a significantly difference in surface roughness before and after sample applied in this group.
“ns” means the difference of surface roughness between this group and FBM group have no significantly difference.

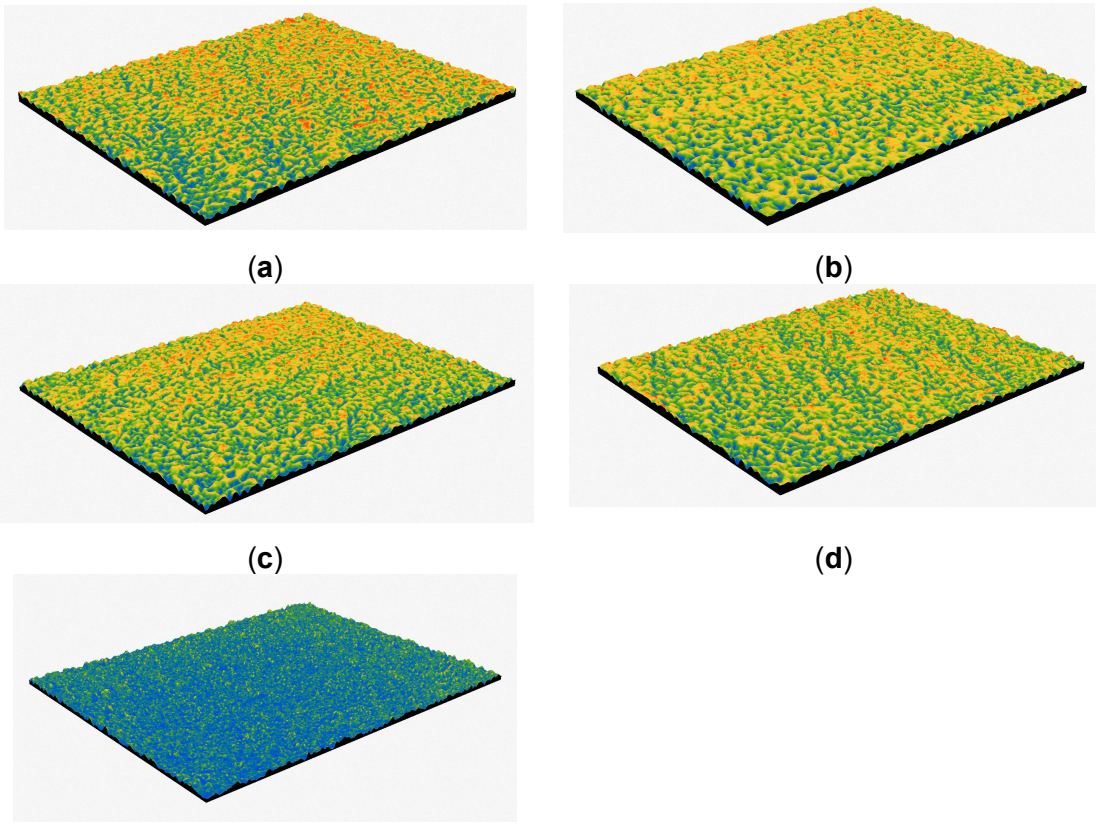


Figure 2. Surface roughness of PMMA plates from different groups. a:10%TMS, b:10%PMS, c:4.8%TMS+7.2%PMS, d:FBM, e:PMMA plates without samples. The more red and yellow areas there are, the more uneven the product is.

3.2 Evaluation of membrane stability: sebum resistance, sweat resistance and friction resistance

Under the influence of sebum, results (Table 4) indicated that L* value of base group and single film-forming agent group showed a significant decrease trend ($p<0.05$), while L* value of TMS and PMS combination group and FBM group did not show significant changes. Although TMS and PMS combination can to some extent inhibit the effect of sebum, its effect was not as good as that of FBM group. Difference of L* value before and after used the sample in FBM group were significantly higher than those in TMS and PMS combination group, single film-forming agent group and base group ($p<0.05$), which indicated that FBM has the strongest ability to resist sebum.

Table 4. Results of different groups under the influence of sebum

Group	L*/a.u.		P value	Difference of L*/a.u.	Change rate of L*/%
	After sample applied	After Interference			
Base	59.98±0.13	57.10±0.30	0.006	-2.87±0.38*	-4.80
10%TMS	67.85±0.06	66.30±0.22	0.008	-1.55±0.24*	-2.28
10%PMS	65.16±0.30	63.47±0.09	0.006	-1.69±0.22*	-2.59
2%DVDC	63.61±0.24	61.73±0.07	0.003	-1.88±0.19*	-2.96
4%TMS+6%PMS	69.79±0.06	68.86±0.56	0.118ns	-0.93±0.61*	-1.33
4.8%TMS+7.2%PMS	66.64±0.02	66.28±0.18	0.071ns	-0.36±0.18*	-0.54
FBM	63.52±0.38	63.64±0.17	0.430ns	0.12±0.21	0.19

“ns” means that there is no difference in L* value before and after interference in this group.
* means the difference of L * value in this group is significantly lower than the FBM group.

Under the interference of sweat and external friction, only L* value of FBM group did not change significantly (Table 5,6). L* value of other test groups decreased significantly ($p<0.05$), which indicated that the stability of samples in other test groups was weak, and reduction of L* value in TMS and PMS combination group was smaller than that in single film-forming agent group, which indicated that combination of TMS and PMS optimized the stability of the film-forming system.

Table 5. Results of different groups under the influence of sweat

Group	L*/a.u.		P value	Difference of L*/a.u.	Change rate of L*/%
	After sample applied	After Interference			
Base	60.47±0.25	56.11±0.41	0.007	-4.36±0.66*	-7.21
10%TMS	68.93±0.20	65.74±0.21	0.005	-3.20±0.40*	-4.63
10%PMS	64.02±0.33	60.28±0.06	0.003	-3.74±0.34*	-5.84
2%DVDC	64.55±0.12	60.38±1.07	0.025	-4.17±1.15*	-6.46
4%TMS+6%PMS	68.84±0.53	67.57±0.06	0.044	-1.27±0.48*	-1.84
4.8%TMS+7.2%PMS	66.65±0.13	65.82±0.15	0.030	-0.83±0.26*	-1.25
FBM	63.55±0.24	63.52±0.38	0.921ns	-0.03±0.47	-0.05

“ns” means that there is no difference in L* value before and after interference in this group.
* means the difference of L* value in this group is significantly lower than the FBM group.

In addition, the difference of L* value in FBM group was significantly higher than other groups ($p<0.05$). These results proved that FBM can effectively make up for the deficiencies of other groups in the resistance to sweat and mechanical friction, thus FBM significantly improved the film-forming stability of foundation.

Table 6. Results of different groups under the influence of external friction

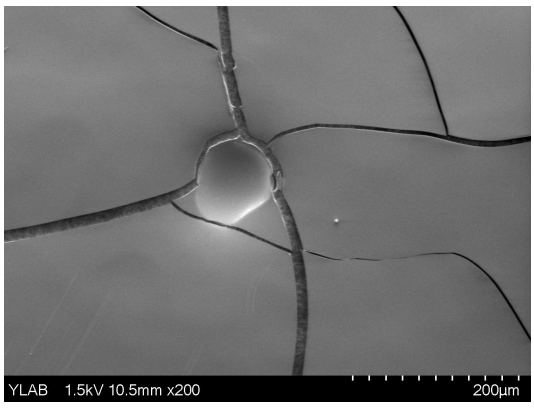
Group	L*/a.u.		P value	Difference of L*/a.u.	Change rate of L*/%
	After sample applied	After Interference			
Base	59.16±0.18	53.70±0.33	0.000	-5.46±0.15*	-9.23
10%TMS	69.53±0.34	65.39±0.18	0.005	-4.14±0.52*	-5.95
10%PMS	64.39±0.17	59.93±0.03	0.001	-4.46±0.20*	-6.93
2%DVDC	63.08±0.20	58.22±0.21	0.002	-4.85±0.36*	-7.70
4%TMS+6%PMS	67.42±0.19	65.30±0.26	0.013	-2.12±0.42*	-3.14
4.8%TMS+7.2%PMS	66.68±0.07	64.74±0.07	0.001	-1.93±0.12*	-2.91
FBM	63.68±0.19	63.65±0.18	0.868ns	-0.03±0.28	-0.05

"ns" means that there is no difference in L* value before and after interference in this group.
* means the difference of L* value in this group is significantly lower than the FBM group.

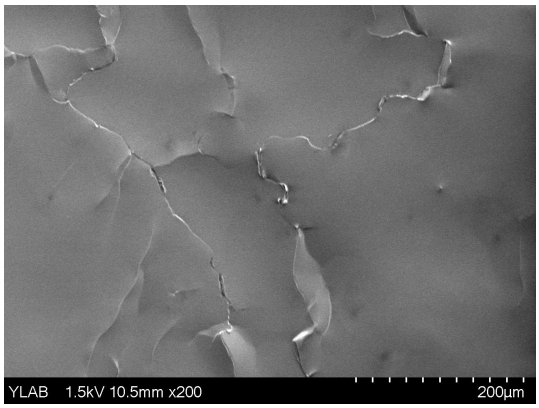
3.3 Evaluation of membrane stability: tensile resistance test

Scanning electron microscopy results showed (Figure 3) that the hard membrane (TMS) was composed of a smooth membrane, but after stretching, there were obvious cracks (Figure 2a), which indicated that although it has better stiffness, it was not enough to cope with the flexibility required during stretching. The soft membrane (PMS) did not show obvious cracks. However, there were noticeable wrinkles (Figure 2b), which indicated that it was easy to deform. The silicone elastomer itself has poor film-forming ability (Figure 2c), with a rough surface and no smooth membrane formed.

After the combination of soft membrane and hard membrane (Figure 2d), there were still obvious cracks and slight wrinkles on the membrane surface. Even with an increase in the ratio of soft and hard membrane (Figure 2e), the integrity of membrane was improved to some extent, but the cracks were still clearly visible and surface wrinkles still existed. After adding silicon elastomer to form FBM, the membrane was smooth and complete, with no obvious cracks or folds seen after stretching, and it exhibited very good toughness (Figure 2f).



(**a**)



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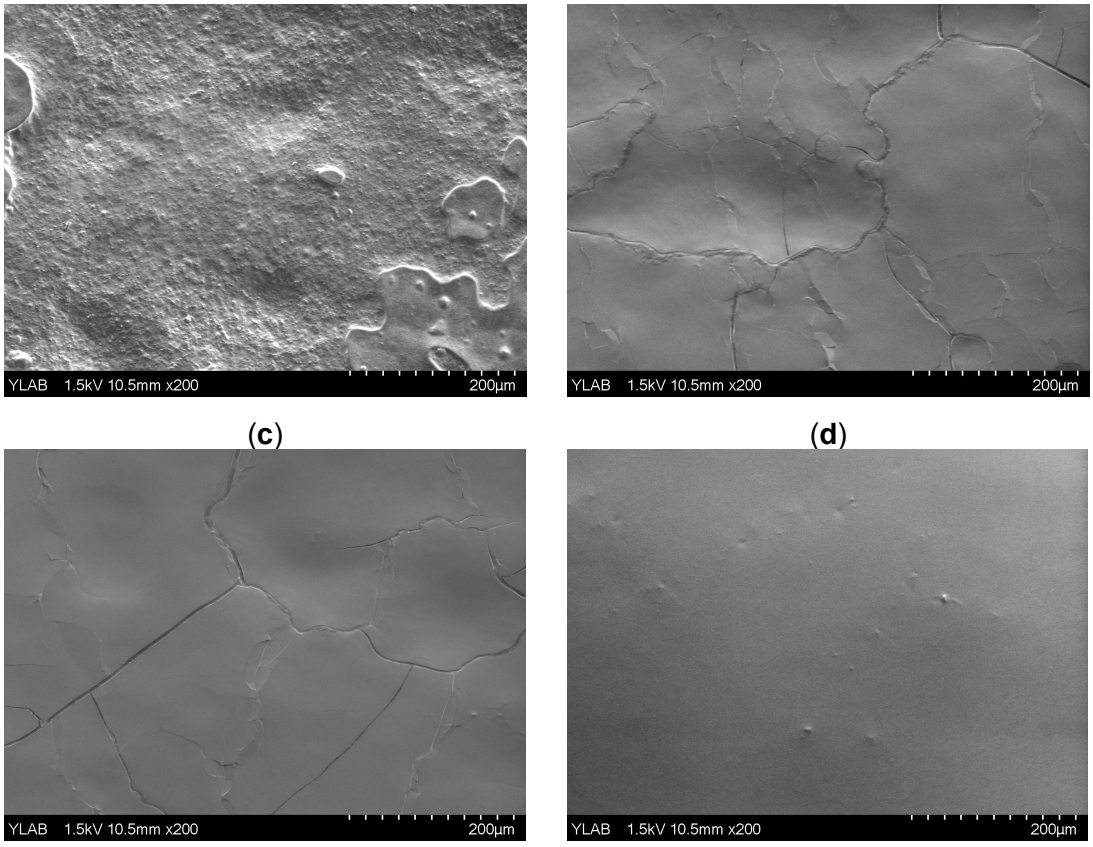

Figure 3. Comparison of surface morphology of different groups. a:20%TMS, b:20%PMS, c:4%DVDC, d:8%TMS+12%PMS, e:9.6%TMS+14.4%PMS, f:FBM.

3.4 Human clinical testing

Results in human clinical testing showed that under the interference of water and artificial sweat, the mean value of ΔE in the inner forearm test area were 1.48 and 1.14, which were both less than 2.

Under the interference of mask friction, facial skin parameters such as pore area, skin texture value and skin uniformity did not change significantly ($p>0.05$) (Table 7).

Table 7. The results of different skin parameters under the interference of mask friction

Testing Parameters	Before	After friction interference with masks
Pore area/mm²	67.71±19.37	67.94±20.88ns
Surface roughness/a.u.	79.80±5.65	78.80±6.56ns
Skin uniformity/a.u.	86.72±3.92	85.92±5.14ns

"ns" means that there is no significant change in skin parameters before and after mask friction.

Results of human clinical testing comprehensively showed that under the influence of different external disturbing factors, the foundation containing FBM maintained a better makeup holding performance.

4. Discussion

This study verified the advantages of FBM in terms of long-lasting makeup through multidimensional experiments, and the improvement in its comprehensive performance could

be attributed to the synergistic optimization of cosmetic formula science and skin compatibility.

In terms of adhesion and homogeneous degree, compared to other test groups, the FBM group showed no significant difference in surface roughness (Table 3). The adhesion and homogeneous degree of product is related to various factors, such as the binding of polar or hydrophobic groups in film-forming agent to skin components, the filling of skin texture gaps with low molecular weight agent (TMS) and the continuous soft membrane (PMS). The combination of the two film-forming agent makes the membrane layer adhere to skin, and the addition of silicone elastomer (DVDC) further enhances the toughness and elasticity of the membrane, making the adhesion of membrane more stable. However, this change might not be significant on the improvement of adhesion and homogeneous degree.

In summary, FBM has the least impact on the changes in surface roughness, reflecting that FBM can better adapt to skin micro-textures compared to other groups. This indicates that FBM fills the texture gaps on skin surface during membrane formation, forming a homogeneous interface.

In terms of membrane stability, compared to single film-forming agent, the combination of hard membrane and soft membrane could better resist external interference such as sebum, sweat and friction. This is due to the high stiffness and friction resistance of hard membrane, which can form a dense hydrophobic network through the siloxane bond (Si-O-Si) in the molecule, increase the contact angle and effectively block these interference. And the conformational changes of flexible long chain in soft membrane reduce the interference of sweat and sebum. However, the data showed that the membrane stability after the combination of these two film-forming agents was not good enough, factors from external environment could also affect its film-forming morphology.

Under the influence of external interference factors, L* value of FBM group had almost no change (nearly zero) compared with that before the interference. Its stability of L* value indicated that FBM has excellent hydrophobic, oil repellent and friction resistant properties, which may be due to the silicon elastomer filling the gap between hard membrane and soft membrane, and combining them by chemical bonding, which helps to form a more stable, soft and hard compact membrane, further enhancing the toughness of the membrane and its ability to resist migration to the environment.

Furthermore,In terms of mechanical performance, in tensile resistance test, the integrity of the combination of rigid hard membrane (TMS) and flexible soft membrane (PMS) was better than that of single film-forming agent. This may be due to the fact that the combined membrane has a dense structure with high mechanical strength in hard membrane and compensates for the brittle fracture of the hard membrane through the soft membrane with good elasticity. However, the overall deformation caused by membrane stretching was still not well improved.

FBM group added silicon elastomer (DVDC) increased the extensibility of the membrane and showed better integrity and stability in the surface morphology of the membrane. This characteristic, when applied in products, can effectively resist membrane rupture caused by changes in facial expressions, thereby prolonging the durability of the makeup effect, which was also confirmed in our human clinical test.

At last, human clinical data further confirm the practical application value of FBM in daily life scenarios such as waterproofing, anti sweating and anti mask friction.

5. Conclusion

Results indicated that FBM has excellent toughness, homogeneous degree and skin adhesion, demonstrated outstanding film-forming stability in complex environments such as

the interference of artificial sebum, artificial sweat and friction. Human clinical test further indicated that FBM effectively maintain the integrity of foundation in practical scenarios such as mask friction and sweat flushing. This study achieved a breakthrough in multiple properties through the triple film-forming system, breaking through the technical bottleneck of relying on multiple functional additives in traditional formulas. These findings provide key theoretical support and technical solutions for the development of simulated film-forming theory and application of FBM in long-lasting makeup product.

In the future, further studies can be conducted on the application of other hard and soft membrane in this film-forming system to evaluate the universality of FBM. We can also evaluate the safety and comfort of FBM, such as non-comedogenic and breathable, to ensure that FBM meet the needs of skin health while enhancing the effectiveness of makeup.

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