Reflectance-Based Elastic Polymer Test: A Novel Fast-Screening Method for Predicting Skin Adhesion Performance of Liquid Foundation

Damayanti, Hilda[1]; Cita, Juang[1]; Devitama, Festy[1]; Komara, Sheilla[1]; **Cita, Juang[1*]**
[1] Research and Development, Paragon Technology and Innovation, Banten, Indonesia
*Juang Arwafa Cita, Kawasan Industri Jatake Blok AG No. 8, Banten, Indonesia, (+62) 81289768332, juang.acita@paracorpgroup.com

Background

Long lasting effect has become a basic requirement for foundation products in which consumers would like to look flawless throughout the day. To achieve that property, the products must have proper spreadability and wettability on the skin, creating high quality of skin-foundation bond that brings good skin adhesion. Therefore, it is crucial to develop reliable adhesion tests to provide faster and objectively quantified results, especially during the formulation stage.

Methods

PMMA plate and Bioskin was chosen as the candidate for foundation attachment substrate, which are compared with the water contact angle result on in-vivo volar forearm. Quantitative in-vitro foundation adhesion test was developed through an elastic polymer tape test by measuring the color intensity using a skin-colorimeter and further calculated into Tape Removal Ratio (TPR).

Results

Water contact angle in PMMA plate has a greater correlation ($\rho=0.8$, $p=0.331$) with in-vivo result, compared with Bioskin ($\rho=0.2$, $p=0.985$). These results confirmed that the PMMA plate is a more suitable substrate for in-vitro adhesive test. TPR results from in-vitro tape test on PMMA plate showed a strong positive correlation against the in-vivo adhesion test ($\rho=1.0$, $p=0.2$) and successfully differentiate foundation samples attachment quality to the substrate.

Conclusion

This study showed preliminary insights about the in-vitro foundation adhesion test using PMMA plate that can reduce observational bias by TPR calculation and quantification of foundation attachment using color intensity value.

Keywords:

Skin adhesion, skin affinity, foundation, contact angle, reflectance

Introduction

People use foundation to even their skin tone; cover the pores, blemishes, and wrinkles; and improve skin lightness and undertone. They would love to get the best performance foundation product which helps their skin look flawless throughout the day. To achieve that property, the products must have proper spreadability and wettability on the skin, creating a good skin adhesion that can stay on perfectly all day with comfortable wear. Skin adhesion is the force required for the separation of the cosmetic from the skin after a long period of contact, higher skin adhesion means that the foundation has better attachment to the skin. It is driven by physicochemical aspects of the skin surface and its interaction with the film-former materials in the foundation formula [1].

The attachment of foundation layer to the skin is influenced by its affinity with external and internal substances e.g., water or sebum and its adhesion with the surface of the skin. The degree of water affinity to foundation can be indicated by the wettability and spreadability of the water on the foundation surface [2]. One of the approaches to evaluate water wettability on foundation is by measuring water contact angle on the foundation. The higher contact angle value of water on foundation exhibits its low affinity with foundation layer, which mean the water will not significantly affect the performance of the foundation [3].

Nowadays, there are a lot of in vivo and in vitro evaluations to address skin adhesion properties of foundations. Barresi et. al. conducted the study to determine the adhesion property of the lipstick formulation using ASTM crosshatch tape onto Bioskin substrate [4]. However, the use of visual assessment at the final step of the evaluation was not well quantified (i.e., by summarizing the general assumption of the volunteers) which was subjected to the bias of observation [5].

This study modified the ASTM tape test method in which the use of quantitative measurements using colorimetry replaced the visual assessment method, thus reducing the subjective nature of the method. A fast-screening method to predict the foundation adhesivity to the skin needs a suitable attachment substrate that can represent the nature of the skin. PMMA plate was chosen as a potential substrate which can address an instant-, fast-, and affordable needs of the in-vitro adhesion test.

Method

Materials

Four commercial foundation products, two long-wear foundations (A and B) and two non-long-lasting foundations (C and D) were used in this study. PMMA plate (Helioplate HD6 molded PMMA Plate, 48 mm x 48 mm, HelioScreen, Creil, France) and Bioskin (#BSC, Beaulax Co.,Ltd, Japan) were used as substrate for the foundation to be attached. Reverse osmosis water was used as a liquid substrate in the contact angle test. The elastic polymer-based ASTM D3359 complied tape (Elcometer 99, Elcometer Inc., Michigan, USA) was used for in-vitro and in-vivo adhesion tests, which include taping procedures.

Foundation Preparation

In-vitro test for contact angle and adhesivity was conducted by creating a 30 µm thickness foundation film on the surface of the PMMA plate and Bioskin using a 30 µm drawdown bar (BYK-Gardner GmBH, Germany). The foundation samples were dried inside a 50°C oven for 10 minutes.

Contact Angle Test

50 µL water and artificial sebum was dropped on the surface of the foundations using a micropipette, after which a picture of the drop was taken 6 seconds after water and sebum was contacted with the foundation layer. The resulting water and sebum drop was photographed using a digital camera (FE 90mm F2.8 Macro G OSS, Sony Corporation, Japan) and the contact angle was measured using a digital microscope (Keyence VHX-7000, Japan).

In-vitro Adhesion Test

Skin-Colorimeter CL 400 (Courage + Khazaka Electronic GmbH, Germany) was used to measure the L* (lightness) value of foundation film on PMMA plate with black Byko-Chart Uncoated Paper (BYK-Gardner GmBH, Germany) as background color during the measurement. The tape was placed on the surface of foundation samples and rubbed firmly using a film applicator once again. After 120 seconds, the tape was removed by pulling it rapidly with 180° angle. The L value foundation film that remained on the PMMA plate was measured to calculate the reduction of L value after the tape was pulled.

Tape Removal Ratio Calculation

Foundation sample adhesivity to the PMMA plate is represented by TPR (tape removal ratio) which calculates the amount of foundation retained in the PMMA Plate after the tapping step. TPR was calculated as follows:

$$TPR = \frac{(L_{after} - L_{before})}{(L_{blank} - L_{before})} \times 100$$

$$L_{after} = L\ value\ of\ foundation\ after\ tapping$$

$$L_{before} = L\ value\ of\ foundation\ before\ tapping$$

$$L_{blank} = L\ value\ of\ PMMA\ plate\ before\ foundation\ application$$

In-vivo Adhesion Test

0.015 gram of each foundation (A, B, C, D) were applied manually on the 3cm x 2.5cm of human volar forearm. The foundation was allowed to dry until set (10-15 minutes). The tape was placed on the surface of the forearm which had already been applied by the sample's foundation and rubbed firmly. After 120 seconds, the tape was removed rapidly with 180° angle. The condition of foundation layer, before and after the tapping procedure, was photographed and analyzed using Keyence Digital Microscope image analysis (Keyence VHX-7000, Japan).

Results

Water contact angle tests were conducted to all the foundation samples to select the most suitable substrate that represents the skin.

Sample	In Vitro Test						In Vivo Test			In vitro – In Vivo Spearman's Correlation			
	PMMA Plate			BioSkin						PMMA - Forearm		Bioskin - Forearm	
	CA (°)	MEAN	STDEV	CA (°)	MEAN	STDEV	CA (°)	MEAN	STDEV	ρ	p	ρ	p
(A)	97,80	90,19	5,95	94,12	100,83	4,76	96,97	92,64	3,51	0.8	0.331	0.2	0.985
	89,25			103,18			92,15						
	83,31			101,02			93,02						
	90,43			104,99			88,42						
(B)	62,62	67,52	7,09	86,31	82,89	5,10	57,56	55,75	7,50				
	72,55			79,78			51,30						
	60,31			88,06			48,64						
	74,58			77,42			65,50						
(C)	81,55	78,76	4,25	76,24	76,61	3,03	61,11	58,45	3,74				
	80,57			73,24			52,94						
	80,49			80,60			59,37						
	72,42			76,36			60,38						
(D)	68,97	69,13	0,18	95,196	100,33	3,60	52,70	52,81	2,31				
	69,00			100,442			56,10						
	69,36			102,817			51,45						
	69,20			102,850			50,98						

Table 1. The performance of foundation's contact angle of in vitro (PMMA plate and BioSkin) against in vivo (human forearm)

The measurement of water contact angle on PMMA plate and Bioskin was analyzed using Spearman's Correlation. PMMA plate showed a stronger positive correlation with in-vivo test result (ρ=0.8, p=0.331) compared to Bioskin (ρ=0.2, p=0.985). Based on this result, PMMA plate was selected as the attachment substrate for in-vitro adhesion elastic-polymer test.

Table 2 showed that the TPR result from the tape test on the PMMA plate exhibited a strong positive correlation against the % reduction of in vivo result (ρ=1.0, p=0.2).

Sample	PMMA (In Vitro)						Forearm (In Vivo)					In Vitro – In Vivo Correlation	
	L PMMA	L BEFORE TAPE	L AFTER TAPE	% TPR	MEAN	STDEV	%BEFORE TAPE	%AFTER TAPE	AREA FOUNDATION REDUCTION	MEAN	STDEV	ρ	p
	49,57	61,31	58,22	26,32%			75,61	73,74	2,48%				
	49,57	61,34	58,2	26,68%			73,95	72,62	1,80%				
(A)	49,57	61,33	58,33	25,51%	25,09%	2,56%	76,31	75,84	0,62%	2,04%	0,92%		
	49,57	68,63	64,71	20,57%			76,99	75,30	2,20%				
	49,57	68,88	63,79	26,36%			78,97	76,53	3,10%				
	49,57	63,3	59,27	29,35%			76,32	71,87	5,83%				
	49,57	63,35	59,64	26,92%			75,83	70,39	7,17%				
(B)	49,57	63,18	59,72	25,42%	27,23%	1,98%	78,15	72,26	7,54%	6,16%	1,20%		
	49,57	62,86	59,78	23,18%			75,79	71,48	5,69%				
	49,57	62,09	56,94	41,13%			77,17	73,65	4,57%			1	0.2
	49,57	61,02	53,86	62,53%			79,27	72,99	7,92%				
	49,57	61,16	53,77	63,76%			77,92	71,54	8,18%				
(C)	49,57	61,24	53,85	63,32%	63,21%	0,62%	72,82	66,60	8,54%	8,40%	0,66%		
	49,57	60,07	54,54	52,67%			81,78	75,35	7,86%				
	49,57	58,91	53,60	56,85%			79,07	71,59	9,47%				
	49,57	64,83	58,68	40,30%			72,91	67,68	7,18%				
	49,57	64,8	58,85	39,07%			71,40	65,87	7,74%				
(D)	49,57	64,88	58,76	39,97%	47,62%	10,78%	72,96	66,04	9,49%	7,24%	1,55%		
	49,57	63,08	55,22	58,18%			73,11	68,36	6,50%				
	49,57	65,94	56,02	60,60%			73,83	69,91	5,30%				

Table 2. The foundation's tape removal ratio in PMMA plates (in vitro) against human forearm (in vivo)

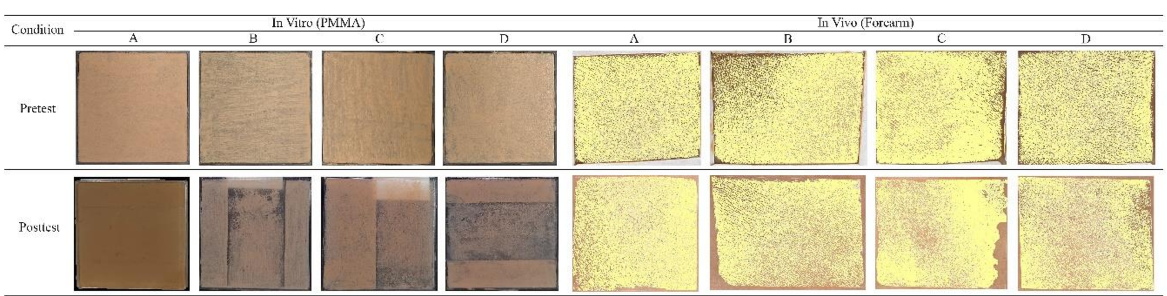

Table 3. The physical appearance of pre- and post-TPR test for in vitro and in vivo method

Discussion

The contact angles measurement of water on deposited foundation aims to provide preliminary insights into the difference in hydrophobicity of formula and the subsequent role that wettability has on skin adhesion property. The formation of water droplets on the surface of foundation was produced by the surface free energy (SFE) difference between water, foundation, and skin substrate [6]. Therefore, during the development of a reliable in-vitro test method for foundation skin attachment, the selection of foundation attachment substrate must be considered based on its correlation with the skin.

Table 1 showed that water contact angle value of foundation on the PMMA plate has a greater correlation with in-vivo result in forearm, compared to Bioskin. These results offered an insight that the PMMA plate has a closer representation as an attachment substrate for in vitro test of foundation contact angle and wettability. Therefore, further development of the foundation adhesion evaluation method was conducted using a PMMA plate.

By far the most prevalent test for evaluating "adhesion" is the tape test, which has been standardized in ASTM D3359 test method. When a flexible adhesive tape is applied to a surface of the tested layers and then removed, the removal process has been described in terms of the "peel phenomenon". Layer removal occurs when the tensile force generated along the layers-substrate interface is greater than the bond strength at the tested layers-substrate interface (or cohesive strength of the layers to the substrate). A significant compressive force arises from the response of the tape backing material to being stretched. Thus, both tensile and compressive forces are in-volved in adhesion tape testing [7].

Based on the Table 2, TPR result from the tape test on the PMMA plate exhibited a strong positive correlation against the area foundation reduction of in vivo result. It was also proven by the physical appearance at the pre- and post-test imaging by using the imaging analysis (Table 3). Sample C as non-long lasting foundation formula was the easiest foundation that can be unattached by the physical contraction while sample A (long-wear foundation formula) was the one which has the best skin adhesion property. Therefore, the in-vitro method successfully differentiated the skin adhesion performance, similar with the in-vivo result.

Visual assessment of post-tape foundation in in-vitro adhesion test which leads to subjective observational bias, is minimalized with the TPR calculation and quantification of foundation attachment value. The utilization of colorimeter as color measurement instruments provides the exact value of the amount foundation layer condition on substrate. Strong positive correlation between in-vitro and in-vivo tape tests provides a possibility in developing reliable, fast, and cost-efficient method in evaluating the skin attachment properties of foundation.

Conclusion

This study showed preliminary insights about the in-vitro foundation adhesion test using PMMA plate that can reduce observational bias by TPR calculation and quantification of foundation attachment using color intensity value. Further validation procedures should be conducted with larger samples and volunteers to elaborate the correlation factor of in-vitro adhesion tape test and in-vivo foundation skin attachment test.

Acknowledgments

The authors would like to thank Paragon Technology and Innovation for supporting this work.

Conflict of Interest Statement

All the authors confirmed that there was no conflict of interests.

References

1. Djajadisastra J (2004) Cosmetic Stability. Jakarta: Presented in Indonesian Cosmetic Association (HIKI).
2. Bui H S, Hasebe M, Ebanks J (2020) Evaluation of Sebum Resistance for Long-Wear Face Make-Up Products Using Contact Angle Measurements. Advances in Contact Angle, Wettability and Adhesion Volume 4: 193–222
3. Capra P, Musitelli G, Perugini P (2017) Wetting and adhesion evaluation of cosmetic ingredients and products: correlation of in vitro – in vivo contact angle measurements. International Journal of Cosmetics Science 39(4): 393-401
4. Barresi R, Liao I (2021) Lip Biophysical Properties and Chararacterization Methods for Long-Wear Lipsticks : 21.

5. Raheem, Zainab (2019) Standard Test Methods for Measuring Adhesion by Tape Test 1. 10.1520/D3359-09E02.
6. Richard C (2021) Lipstick Adhesion Measurement. Surface Science and Adhesion in Cosmetics: 635–662
7. ASTM International (2010) Standard Test Methods for Measuring Adhesion by Tape Test.