The Effect of Thickeners and Performance-Enhancing Powders on the Application Characteristics of Mascara

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Introduction

Mascara is a relatively simple cosmetic product, upon which consumers and formulators place a set of very complex requirements. Different formulation approaches are used in order to achieve a combination of ease of application, longevity, comfort, increased eyelash volume and perceivable eyelash curl, expected from a good product [1,2].

Mascara formulas must be thixotropic with rapid recovery to prevent dripping until the film dries after application. In addition to providing suitable rheology, thickeners improve product homogeneity, enabling the deposition of a uniform film onto the lashes.

Performance enhancing powders are added to the classic mascara formulation to help product deposition, increase lash diameter and prevent 'flaking' [3]. They may also aid the application of second and subsequent layers of product.

There are very few publications on mascara analysis [e.g. 4,5], indicating that cosmetic houses have their own testing methods. It was of interest to establish suitable protocols for the testing of a range of mascara properties, including deposition, tack, curl and compression. Image analysis was also used in this study in order to evaluate the effects of mascara products on the length, diameter and volume of the eyelashes.

Aim

The aim of this study was twofold: to establish a set of relevant application tests and to systematically assess the changes in the application of mascara due to the variations in formulation parameters.

Materials and Methods

Materials

Mascara formulation chosen for this study, based on *in situ* formed triethanolamine-stearate, is shown in Table I. Thickeners and powders were added at the expense of water. A commercial product from the French market was used as a benchmark.

INCI	**%w/w**
Beeswax	6.00
Candellila wax	5.00
Ozokerite wax	3.00
Cetyl alcohol	3.00
Stearic acid	5.00
Propylparaben sodium	0.10
Aqua	61.70
Propylene glycol	2.00
Propylene glycol (and) diazolidinyl urea (and) methylparaben (and) propylparaben	0.20
CI 33600	10.00
Triethanolamine (99% pure)	3.00

Table I. Composition of the basic formulation

Six different hydrophilic thickeners were used: xanthan gum, microcrystalline cellulose (and) cellulose gum, pectin, hydroxypropyl guar, gellan gum and acrylates/C10-30 alkyl acrylates crosspolymer. In addition, this study has looked into the performance enhancing effects of the following powders: polyamide-5, nylon-12, methyl methacrylate crosspolymer, talc and dimethicone/vinyl dimethicone crosspolymer (and) silica.

Methods

Test 1: Deposition of product onto lashes

Identical eyelashes were laboratory-made, using human hair of the same type and length. The weight of every individual eyelash was recorded and, using tweezers, it was attached onto a rim of a small beaker with scotch tape, giving it a defined curvature. The test product was applied on the eyelash in a reproducible manner; the eyelash was weighed and left to dry. This was done in triplicate for each test sample.

Test 2: Tack test – Analysis of the drying time of the product

A small amount of product to be tested was placed on a glass plate and levelled using a 200μm draw down bar. The set area was tacked every 10 seconds, using index finger. The point of tack was randomised and the finger cleaned between tack tests. Once there is no product removed by the finger, the product was considered dry and the time was recorded.

Test 3: Measurement of eyelash curl

Adhesive double-sided tape was applied to one edge of a glass plate. Three eyelashes treated with the same formulation were placed on the adhesive in a row. The plate is then placed in front of the camera, and the image of each eyelash was taken. Once printed, the angles in the photograph were measured using a protractor.

Test 4: Compression test - Analysis of the resistance to external pressure

This test was performed using the instrument Testometric M350-20CT (UK). A standard eyelash with a known amount of product was mounted on a vertical holder in a way that it just touches a horizontal glass surface. The holder was set to move for 5mm, applying a constant force, while the force of resistance exerted by the test sample was measured.

Test 5: Microscopic image analysis

Each eyelash was viewed under the microscope Leica DM E (Germany). The whole of the eyelash width and length was viewed, a representative photograph was taken and then analysed. It was possible to measure the difference between the eyelash diameter before and after product application.

Results and Discussion

The results for deposition, tack, curl and compression are presented in Table II. One-way ANOVA analysis has confirmed the existence of significant differences among samples in all tested categories. Detailed statistical analyses revealed that the thickener concentration played a major part in exerting the differences between samples. However, the highest concentration was almost never the optimal one. On the basis of similarities with the benchmark product, the formulation with 0.2% xanthan gum (shown in bold) was chosen for further study. In principle, the addition of a thickener to the basic mascara formulation aided in the application of the product and increased the amount of mascara deposited onto the lashes in a single application.

Table III presents the same set of application parameters as Table II, this time obtained from samples containing 0.2% xanthan gum and a range of powders known to enhance the performance of mascara products.

Formulation	Deposition (gram)		Tack Test (second)		Curl (angle)		Compression (newton)	
	mean	SD	mean	SD	mean	SD	mean	SD
Benchmark	0.031	0.005	-	-	14.3	2.0	0.372	0.151
Basic	0.029	0.004	170.00	0.02	7.0	2.0	0.366	0.046
Xanthan 0.2%	**0.030**	**0.003**	**173.33**	**0.02**	**11.3**	**5.5**	**0.357**	**0.175**
Xanthan 0.4%	0.025	0.004	116.67	0.02	7.0	3.0	0.510	0.227
Xanthan 0.6%	0.027	0.002	90.00	0.02	6.3	2.5	0.680	0.153
Acrylates 0.2%	0.023	0.003	170.00	0.01	9.6	1.1	0.343	0.083
Acrylates 0.4%	0.020	0.001	80.00	0.01	8.3	1.1	0.440	0.282
Acrylates 0.6%	0.013	0.004	56.67	0.01	4.6	0.5	0.179	0.118
Gellan gum 0.2%	0.028	0.004	143.33	0.02	11.0	6.0	0.487	0.224
Gellan gum 0.4%	0.033	0.003	130.00	0.02	5.3	1.1	0.509	0.298
Gellan gum 0.6%	0.026	0.005	150.00	0.01	5.3	2.5	0.684	0.156
Guar 0.2%	0.024	0.003	156.67	0.01	6.0	1.0	0.313	0.091
Guar 0.4%	0.020	0.003	156.67	0.01	5.3	1.1	0.407	0.186
Cellul.gum 0.2%	0.033	0.006	73.33	0.02	4.3	1.5	0.801	0.406
Cellul.gum 0.4%	0.031	0.005	63.33	0.02	11.6	2.8	0.454	0.146
Cellul.gum 0.6%	0.026	0.003	73.33	0.02	9.0	4.3	0.538	0.101
Pectin 0.2%	0.032	0.010	73.33	0.02	5.3	1.1	0.443	0.158
Pectin 0.4%	0.030	0.007	106.67	0.02	4.6	1.5	0.309	0.031
Pectin 0.6%	0.031	0.005	110.00	0.02	3.6	2.0	0.383	0.221

Table II. Effects of different thickeners on the application properties of mascara

Figure 1 shows variations in the measured individual hair diameter after the application of mascara formulations containing 0.2% xanthan gum and a range of powders, used in two concentrations. For comparison purposes, the diameter of bare hair, and after the application of basic formulation and the benchmark were also shown. The highest increase in hair diameter was achieved by 1.5% talcum powder, but microscopic analyses have shown that the layer of product on the hair was not uniformly distributed.

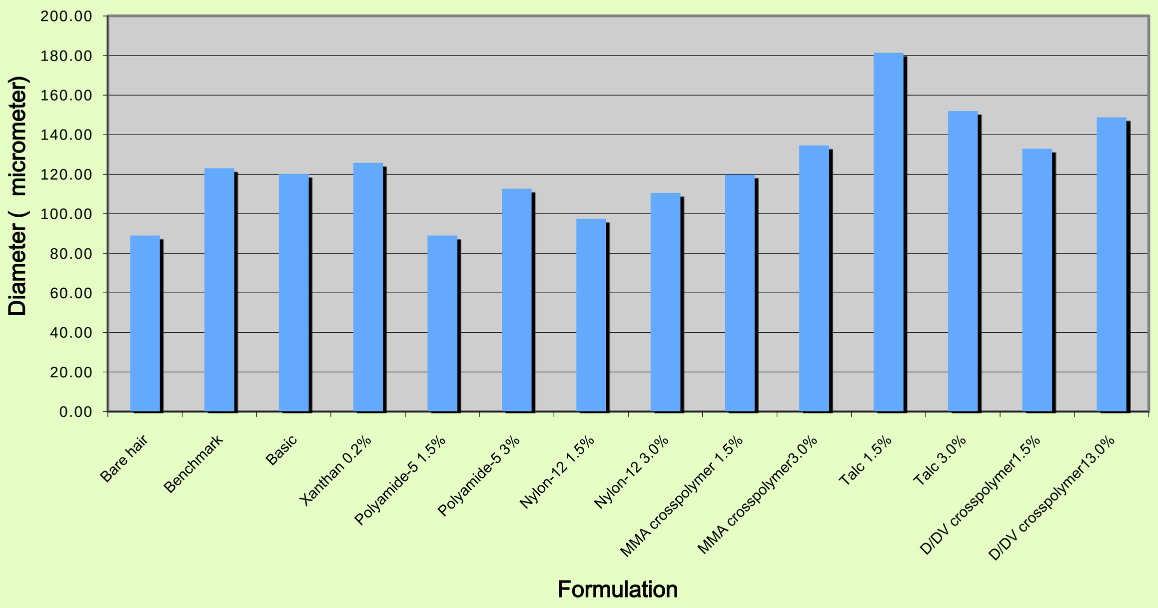

Figure 1. Average diameter of eyelashes after treatment with different formulations

On the basis of the results shown in Table III, the best performance was achieved by polyamide-5, followed by nylon-12. Polyamide-5 has produced the highest curl angle and a much shorter drying time than the basic formulation. Interestingly, its deposition is higher when added in lower concentration. However, its firmness during compression is much higher than the benchmark and this property clearly has to be optimised. In terms of the choice between the two concentrations, the microscopic analysis has shown that a 3% sample produces a more uniform deposition of product onto the lashes.

Formulation	Deposition (gram)		Tack Test (second)		Curl (angle)		Compression (newton)	
	mean	SD	mean	SD	mean	SD	mean	SD
Benchmark	0.031	0.005	-	-	14.3	2.0	0.372	0.151
Basic	0.029	0.004	170.00	0.02	7.0	2.0	0.366	0.046
Polyamide-5 1.5%	**0.078**	**0.004**	**76.67**	**0.02**	**8.3**	**5.1**	**0.840**	**0.276**
Polyamide-5 3.0%	**0.032**	**0.004**	**73.33**	**0.01**	**7.6**	**3.1**	**0.873**	**0.193**
Nylon-12 1.5%	0.031	0.005	80.00	0.01	5.7	1.8	0.619	0.345
Nylon-12 3.0%	**0.022**	**0.008**	**66.67**	**0.01**	**6.7**	**4.1**	**0.442**	**0.102**
MMA 1.5%	0.016	0.002	90.00	0.01	3.7	1.1	0.332	0.161
MMA 3.0%	0.021	0.009	130.00	0.01	4.3	1.2	0.384	0.276
Talc 1.5%	0.021	0.003	123.33	0.01	3.3	3.0	0.640	0.287
Talc 3.0%	0.020	0.002	153.33	0.02	2.3	3.1	0.567	0.068
D/VD 1.5%	0.019	0.002	76.67	0.02	4.3	3.1	0.143	0.035
D/VD 3.0%	0.025	0.003	66.67	0.02	3.7	1.6	0.311	0.019

Table III. Effect of different powders on the application properties of mascara containing 0.2% xanthan gum; MMA - methyl methacrylate crosspolymer; D/DV -dimethicone/vinyl dimethicone crosspolymer (and) silica.

a) b)

Figure 2. Examples of microscopic images: a) basic formulation; b) formulation with 2% xanthan gum and 3% polyamide-5

It appears that the uniformity of shape and size of synthetic polymers offers a more superior performance. The difference in curling ability between polyamide-5 and nylon-12 could be due to different weight of particles, since it is known that nylon-12 is a denser product.

Generally, the addition of powder to the mascara formulation has resulted in easier initial application, as well as a more successful re-application of the product. Powders reduce the appearance of clumps and the sticking of eyelashes caused by mascara.

Figure 2 provides examples of images used for microscopic analysis (performed in triplicate for each sample), showing basic formulation and the one containing 3% polyamide.

Conclusion

Five *in vitro* testing protocols for the evaluation of mascara products have been developed, i.e. deposition, tack, curl and compression tests and microscopic image analysis, and applied to two sets of formulations The results obtained were mostly highly reproducible and of practical value in the formulation work.

Out of the six rheological modifiers tested (five natural derivatives and one synthetic), xanthan gum in the concentration of 2% was shown to be the most suitable for the TEA-stearate based mascara product. It has produced the formulation that largely matched the properties of the commercial benchmark product. Out of the five powders tested (four synthetic polymers and talc), polyamide-5 has shown the best compatibly with the chosen thickener and the best overall performance, closely followed by nylon-12.

Further work in this area should involve *in vivo* evaluation, as an ultimate test of the validity of *in vitro* data.

References

[1] Barel.A., Paye.M., Maibach.H., Handbook of Cosmetic Science and Technology, Marcel Dekker, New York, USA, 2001

[2] Rieger, M., Harry's Cosmeticology. Chemical Publishing Co., New York, USA, 2000

[3] New Developments in Mascara, Make-up, Skin Dyeing and Lipsticks, SOFW Journal, Vol. 131, No 11, 2005, p.10-13.

[4] Loginova, Y., Macchio, R., Structural Analysis of Mascara Formulations Under Shear, 24th IFSCC Congress, October 2006, Osaka, Japan, Proceedings

[5] Knox, A., Loichie, J.W., Lockwood, K.P., Shaffer, M.J., An accurate, non-invasive new method for assessing volumizing, thickening and lengthening efficacy of mascara products, www.crl-inc.com/pdf/lashposter.pdf