Sebum Absorption Characteristics of Polymer Microgel-containing Face Powder

Young Keun Lee, Fan-Long Jin,[†] and Soo-Jin Park[‡,*]

Industry-Academy Cooperation Foundation, RIC, Natural Cosmetics and Perfumery Institute, Soon Chun Hyang University, Asan, Chungnam 336-745, Korea

[†]Department of Materials Engineering, Jilin Institute of Chemical Technology, Jilin 132022, P.R. China

[‡]Department of Chemistry, Inha University, Incheon 402-751, Korea. [*]E-mail: sjpark@inha.ac.kr

Received March 16, 2007

In this study, poly(*iso*-butyl methacrylate-co-ethylene glycol dimethacrylate) [poly(*iso*-BMA-co-EGDMA)] microgel was prepared and used as a face powder additive. The spreading, adhesiveness, and skin reactivity of poly(*iso*-BMA-co-EGDMA) microgel-containing face powder II were investigated and compared with the same properties of commercially available Silica bead 700-containing face powder I. In the results, the particle size of the poly(*iso*-BMA-co-EGDMA) microgel was significantly swelled as a result of sebum absorption. Face powder II showed a lower primary irritation index and a higher adhesiveness than did face powder I. Face powder I showed a low sebum absorption ratio and a relatively high rate of sebum absorption, whereas face powder II, contrastingly, exhibited a high sebum absorption ratio and a low rate of sebum absorption, which properties would reduce the phenomena of facial strain and sliminess. These results indicate that poly(*iso*-BMA-co-EGDMA) microgel has outstanding sebum absorption characteristic and adhesiveness, and thus that it is a good candidate for use as a face powder additive.

Key Words : Microgels, Sebum absorption ratio, Adhesiveness, Smoothness, Skin reaction

Introduction

Polymer microgels have good dispersion stability, good processing characteristics, as well as various porosities and particle compositions. Thus, they are widely used in chromatographic media, surface coatings for metal panels, and catalysts. Recently, too, they have been used as cosmetic additives, drug-delivery systems, and in digital photography toner.[1-4] The methods of polymer microgel preparation include seed-swelling polymerization, emulsion polymerization, precipitation polymerization, and dispersion polymerization.[5-8]

Commonly, women use makeup both to conceal skin faults and to enhance positive attributes. Makeup use entails drawbacks, however, such as the phenomena of facial strain and sliminess caused by sebum secretion. Powder products applied to the face produce an amount of film that will vary according to the amount of sebum secreted. That is, sebum secreted by the skin will agglomerate powders, thus causing facial strain. The sliminess phenomenon caused by excess secreted sebum is another common source of dissatisfaction for women who use powder products, especially two-way cake or face powder. Powder products can be classified, according to the composition ratio of powder and oil, as face powder, compact, two-way cake, powder foundation, and others. Among these, face powder contains either no or little oil, and is the powder product therefore that consists most predominantly of powder materials.[9-11]

In a previous paper, we reported the synthesis, by fractional factorial experimentation and response surface design, of poly(*iso*-butyl methacrylate-co-ethylene glycol dimethacrylate [poly(*iso*-BMA-co-EGDMA)] microgel for use as a cosmetic additive. Fortunately, we were able to obtain the optimal conditions for synthesis of the mocrogel.[11] In the present study, then, we prepared a face powder using microgel prepared according to those optimal conditions, and investigated the spreading, adhesiveness, and skin protectiveness of the face powder compared with the corresponding properties of a commercially available face powder. The sebum absorption ratio of the polymer microgel and of the commercially available face powder, respectively as a function of time, was investigated.

Experimental

Materials. The monomer used in this study was *iso*-butyl methacrylate (*iso*-BMA), purchased from Junsei Chem. The monomer was washed with 10% NaOH solution and evaluated under a nitrogen atmosphere before use. Ethylene glycol dimethacrylate (EGDMA, Wako Pure Chem.), 2,2'-Azobis (isobutylronitrile) (AIBN, Junsei Chem.), hydroxypropylmethylcellulose (HPMC, Sumsung Pure Chem.), and acacia gum (Shinyo Pure Chem.) were used as a cross-linking agent, an initiator, a dispersive stabilizer, and a co-stabilizer, respectively. Solvents were used as received.

Synthesis of polymer microgel. The determined amounts of HPMC (0.7 wt%) and acacia gum (0.5 wt%) were dissolved in 80 ^{o}C distilled water, and then IPA (5 wt%), *iso*-BMA (20 wt%), EGDMA (2.0 mol%), and AIBN (1.0 mol%) were added to the mixture. The mixture was placed under a nitrogen atmosphere to eliminate oxygen. Then, dispersion polymerization was carried out at 80 ^{o}C for 90 min. The resulting products were put in acetone, and the deposit was washed with acetone several times. Finally, the microgels were dried at 100 °C for 4 h.

Particle form measurement. The microgel powder was dissolved in methanol after which the solution was evenly dispersed on a glass surface. The powder particle size and morphology were observed using a scanning electronic microscope (SEM, Hitachi S570).

Sebum absorption ratio measurement. The polymer microgel or face powder (0.3 g) was placed in a Millicell for 10 h in order to absorb the sebum. Then the polymer microgel or face powder was placed on tissue to eliminate the over-absorbed sebum. The sebum absorption ratio was calculated from the following equation:

$$\text{Sebum absorption ratio} = \frac{W_f - W_i}{W_i} \times 100 \qquad (1)$$

where W_i is the initial weight of polymer microgels, and W_f is the weight of polymer microgels after absorbption. The ratio was calculated by a new evaluation method, as follows:

Sebum absorption ratio (New method)
= −0.0037 + 1.18 × sebum absorption ratio (JIS method)

Animal irritation test. The animal irritation caused by the microgel and the microgel-containing face powder was studied in a 7-day experiment using guinea pigs (Hartley breed, male, 350-450 g) under a temperature of 20 ± 3 °C, a relative humidity of 50 ± 5%, a circulation of 10-15 h, an illumination time of 14 h/d (07:00-19:00), and a luminous intensity of 150-300 Lux. After completing the experiment, the seven guinea pigs exhibiting healthy and natural skin were chosen for a stimulation experiment conducted according to the National Institute of Safety Research guide book.

Spreading-quality evaluation. To evaluate the spreading quality of the twin cake and the face powder, twenty women (in their twenties and thirties) were selected to use microgel-containing face powder and normal cake. The evaluation scale included five grades (5: excellent, 4: good, 3: average, 2: somewhat, 1: bad), and the results were obtained by averaging the scores of the twenty subjects.

Adhesiveness evaluation. To evaluate the adhesiveness of the twin cake and the face powder, another twenty women (also in their twenties and thirties) were chosen to use microgel-containing face powder and normal cake. The evaluation scale used was the same, and the results were obtained also in the same way (*i.e.*, by averaging the score of the twenty subjects).

Results and Discussion

SEM micrographs. According to our previous, above-mentioned study, the optimal synthesis conditions of poly(*iso*-BMA-co-EGDMA) microgel are 1.1 wt% calcium phosphate, 0.9 wt% acacia gum, 1.3 wt% HPMC, 1.0 mole% AIBN, 2.5 mole% EGDMA, 1000 rpm stirring speed, and 14 wt% acetone/water. Under these conditions, the microgel has a particle size of 4.76 μm and a sebum absorption ratio of 434.8%. Table 1 shows recipes and formulas for face powder containing Silica bead 700 or polymer microgel.[12]

Table 1. Formulas of face powder

	Ingredient	Concentration (%)	
		Face powder I	Face powder II
A	Kaolin	5.00	5.00
	Mica	6.00	6.00
	Talc	to 100.00	to 100.00
	Zinc stearate	7.00	7.00
	Iron oxide (red)	0.33	0.33
	Iron oxide (black)	0.13	0.13
	Iron oxide (yellow)	0.57	0.57
	Methyl paraben	0.15	0.15
	Propyl paraben	0.05	0.05
	MG-45	–	20.00
	Silica bead 700	20.00	–
B	Squalane	2.50	2.50

Sebum-absorbing powders such as middle-porous porosity silica (product number: Silica bead 700), porosity cellulose, and others, absorb sweat and sebum by means of their middle-porous property, their porosity property and the volume of the powder remaining unchanged. Thus, their sweat and sebum absorption capacity is limited. The poly(*iso*-BMA-co-EGDMA) microgel prepared in this study absorbs sebum by volume expansion, thus increasing the amount of sebum absorption compared with normal sebum-absorbing powders. Figure 1 shows SEM micrographs of normal sebum-absorbing powder (Silica bead 700) before and after sebum absorption. The particle size of the Silica

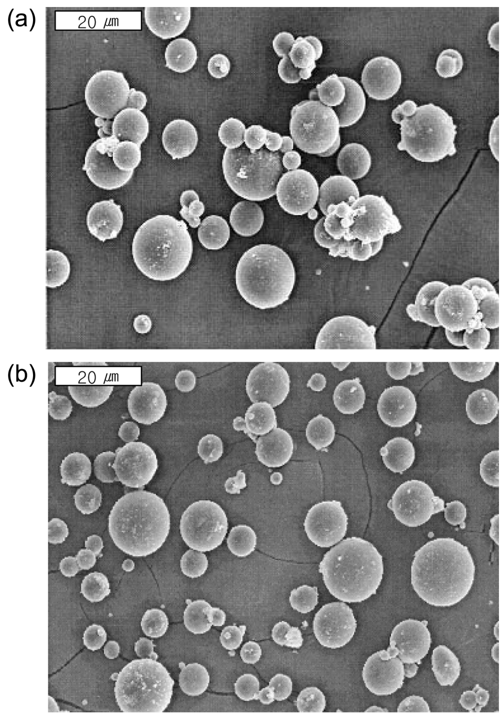

Figure 1. SEM micrographs of Silica bead 700 (a) before and (b) after sebum absorption.

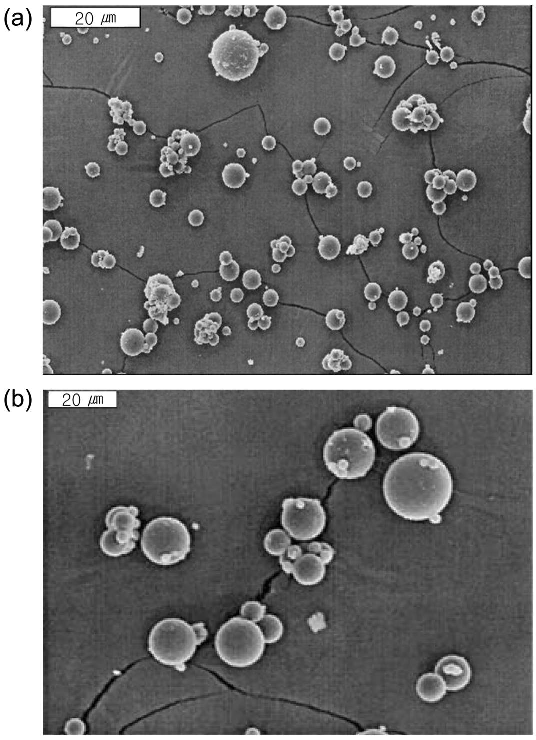

Figure 2. SEM micrographs of poly(*iso*-BMA-co-EGDMA) microgel (a) before and (b) after sebum absorption.

bead 700 before and after sebum absorption did not vary, as shown in Figure 1. Figure 2 shows SEM micrographs of the microgel before and after the sebum absorption. As can be seen, the post-sebum-absorption particle size of the microgel was significantly expanded compared with the pre-sebum-absorption particle size.

Animal irritation and adhesiveness tests. The animal irritation of poly(*iso*-BMA-co-EGDMA) microgel and microgel-containing face powder was investigated and compared with that of widely used sebum-absorbing Silica bead 700 powder. The skin reactivities for the microgel and Silica bead 700, respectively, are shown in Table 2. As can be seen, the primary irritation index (P.I.I.) of the microgel is the same as that of Silica bead 700. Table 3 shows the skin reactivities for Silica bead 700-containing face powder I and microgel-containing face powder II, respectively. The P.I.I. of face powder II is lower than that of face powder I,

Table 2. Results of skin reaction of poly(*iso*-BMA-co-EGDMA) microgels and silica bead 700

Site		Microgel				Silica bead 700			
Change		Eruthema & Eschar		Edema		Eruthema & Eschar		Edema	
Phase (hrs.)		Intact		Intact		Intact		Intact	
Animal	Sex	24	72	24	72	24	72	24	72
1	♂	1	1	0	0	1	1	0	0
2	♂	0	0	0	0	0	0	0	0
3	♂	0	0	0	0	0	0	0	0
4	♂	0	0	0	0	0	0	0	0
5	♂	1	0	0	0	0	0	0	0
6	♂	0	0	0	0	0	0	0	0
7	♂	0	0	0	0	1	0	0	0
Mean score		0.29	0.14	0	0	0.29	0.14	0	0
Σ Mean score		0.43		0		0.43		0	
Total		0.43		0		0.43		0	
P.I.I		0.22		0		0.22		0	

[a]Time after topical application; [b]P.I.I (Primary irritation index) = Total/2.

Table 3. Results of skin reaction of face powder I and face powder II

Site		Face powder I				Face powder II			
Change		Eruthema & Eschar		Edema		Eruthema & Eschar		Edema	
Phase (hrs.)		Intact		Intact		Intact		Intact	
Animal	Sex	24	72	24	72	24	72	24	72
1	♂	1	0	0	0	1	1	0	0
2	♂	0	0	0	0	0	0	0	0
3	♂	0	0	0	0	0	0	0	0
4	♂	0	0	0	0	0	0	0	0
5	♂	0	0	0	0	0	0	0	0
6	♂	1	1	0	0	1	0	0	0
7	♂	1	0	0	0	0	0	0	0
Mean score		0.43	0.14	0	0	0.29	0.14	0	0
Σ Mean score		0.57		0		0.43		0	
Total		0.57		0		0.43		0	
P.I.I		0.29		0		0.22		0	

[a]Time after topical application; [b]P.I.I (Primary irritation index) = Total/2.

Table 4. Sensory test results of face powder I and face powder II

Name	Sex	Age	Adhesiveness		Smoothness	
			Face powder I	Face powder II	Face powder I	Face powder II
Seo O O	F	31	4	4	3	4
Lee O O	F	23	4	4	4	3
Lim O O	F	35	5	4	3	4
Park O O	F	22	3	4	5	4
Lim O O	F	21	4	5	4	5
Yoon O O	F	27	3	4	4	4
Park O O	F	26	3	4	3	3
Kim O O	F	27	5	4	4	4
Lee O O	F	29	2	4	3	4
Lee O O	F	30	5	5	4	3
Jeong O O	F	34	5	5	4	4
Kim O O	F	35	4	3	4	4
Cha O O	F	26	4	5	5	4
Park O O	F	27	4	4	3	3
Kim O O	F	24	3	5	3	4
Song O O	F	23	5	4	4	3
Kim O O	F	31	4	4	4	4
Lee O O	F	30	3	3	5	4
Average score			3.83	4.17	3.77	3.73

Score: good 5, normal 3, bad 1.

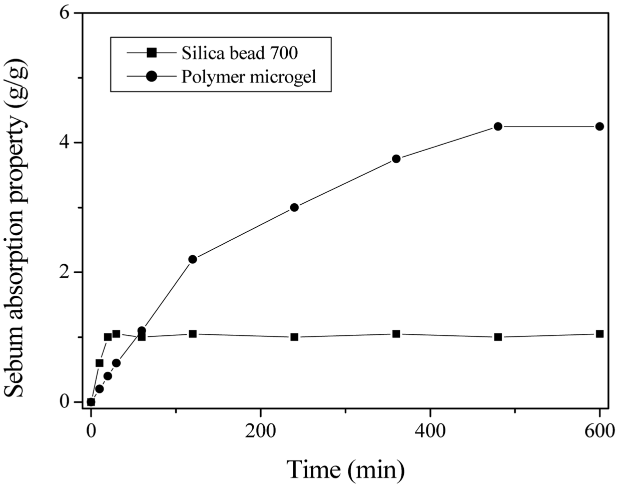

Figure 3. Sebum absorption properties of poly(*iso*-BMA-co-EGDMA) and Silica bead 700 as a function of time.

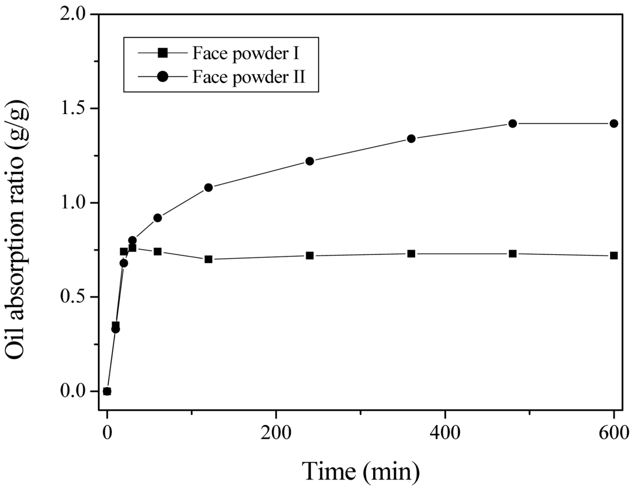

Figure 4. Sebum absorption properties of face powder II [with poly(*iso*-BMA-co-EGDMA) microgel] and face powder I (with Silica bead 700) as a function of time.

indicating that face powder II is gentler to skin than face powder I.

The sensory results of the adhesiveness tests on face powder I and the face powder II are shown in Table 4. As can be seen, face powder II, whereas equal in smoothness to face powder I, has a higher adhesiveness quality than that of face powder I. This result is due to the fact that microgel absorbs sebum by expanding its volume, thereby reducing facial strain and sliminess, which results in the increased adhesiveness.

Sebum absorption properties. The sebum absorption properties of poly(*iso*-BMA-co-EGDMA) microgel and Silica bead 700, as functions of time, were investigated according to a new evaluation method utilizing the sebum absorption ratio, and the results are shown in Figure 3. When the absorption time was 30 min, the sebum absorption ratio of Silica bead 700 was higher than that of the microgel. However, when the absorption time was 60 min, the sebum absorption ratio of Silica bead 700 was similar to that of the microgel. After 30 min, the sebum absorption ratio of Silica bead 700 did not vary, whereas the sebum absorption ratio of the microgel, to 500 min, continuously increased. This result can be attributed, once again, to the fact that microgel absorbs sebum by expanding its volume, which results in its increased sebum absorption ratio. Silica bead 700 showed a low sebum absorption ratio and a high rate of sebum absorption, whereas the microgel showed a high sebum absorption ratio and a low rate of sebum absorption, which qualities can alleviate facial strain. Also, the sebum absorption was carried out at a low rate and over a long time, thereby reducing sliminess as well.

The sebum absorption properties of microgel-containing face powder II and Silica bead 700-containing face powder I were also investigated according to a similar method, and the results are shown in Figure 4. Face powder I showed a low sebum absorption ratio and a relatively high rate of sebum absorption, whereas face powder II showed a high sebum absorption ratio and a low rate of sebum absorption, which properties can reduce both facial strain and sliminess.

According to these results, it was confirmed that poly(*iso*-BMA-co-EGDMA) microgel-containing face powder II has superior properties of sebum absorption and adhesiveness to those of Silica bead 700-containing face powder I.

Conclusions

The spreading, adhesiveness, and skin reactivity characteristics of poly(*iso*-BMA-co-EGDMA) microgel-containing face powder II were investigated and compared with those of commercially available Silica bead 700-containing face powder I. In the result, the particle size of Silica bead 700 before and after sebum absorption did not vary, whereas the particle size of poly(*iso*-BMA-co-EGDMA) microgel significantly expanded after sebum absorption. The P.I.I. of face powder II was lower than that of face powder I. The smoothness of face powder II was similar to that of face powder I, and the adhesiveness of face powder II was higher than that of face powder I. Finally, face powder I showed a low sebum absorption ratio and a relatively high rate of sebum absorption, whereas face powder II exhibited a high sebum absorption ratio and a low rate of sebum absorption, which properties can alleviate the phenomena of facial strain and sliminess. According to these results, it was confirmed that poly(*iso*-BMA-co-EGDMA) microgel-containing face powder has outstanding properties of sebum absorption and adhesiveness that are clearly superior to those of commercially available Silica bead 700-containing face powder.

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