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 Received: 04-3-2011  
 Revised: 08-3-2011  
 Accepted: 18-3-2011

## Evaluation of Shampoos Containing Silicone Quaternary Microemulsion

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### ABSTRACT

This study aimed to characterize properties and to evaluate conditioning performance providence of shampoos containing various concentrations, i.e., 0, 1, 2, 3 and 4 %w/w, of silicone quaternary microemulsion. The results indicated that all samples were clear yellowish liquids with good odor. Addition of silicone quaternary microemulsion in the investigated formulation did not markedly affect the characteristics of the obtained shampoos, except viscosity. Their averaged pH values were in the range of 6.59-7.17 which were acceptable according to TIS 162-2541. All samples provided stable foam, surface tension reduction and low viscosity with Newtonian flow. Under a light microscope, the cuticle arrangement of five tresses washed by the studied shampoos seemed to be better than their counterpart tresses before washing. However, no obvious difference on the cuticle arrangement of the tresses after washing by the identical manner with different shampoos was observed. Most of 50 referees rated and scored that the tress washed with shampoo containing 1%w/w silicone quaternary microemulsion provided the highest conditioning performance, i.e., smooth and softness, via contacting. It could be concluded that silicone quaternary microemulsion was able to be incorporated with the investigated shampoo formulation as an effective hair-conditioning agent.

**Keywords:** Hair, Microemulsion, Shampoo, Silicone quaternary microemulsion

### INTRODUCTION

The shampoo sector is probably the largest unit sale amongst the hair care products since shampoos are one of cosmetic products used in daily life. Main function of shampoos is for cleansing the hair and scalp; however, other benefits such as hair-conditioning enhancement are also expected. A very wide range of different shampoos for different hair types and with different functions are found in the market, e.g., anti-dandruff, baby, hair-colored and conditioning shampoos. For conditioning shampoos, several chemicals have been investigated to be used as hair-conditioning agents. Moreover, applications of nanotechnology components such as microemulsions in shampoos are interesting for product development. Microemulsions are thermodynamically stable, transparent, low-viscous dispersions of oil and water stabilized by an interfacial film of a surfactant and usually in combination with a cosurfactant. Their advantages include ease of preparation, aesthetic appearance, thermodynamic stability and high solubilization power. Hence, microemulsions have been widely used in cosmetic and cosmeceutical formulations for skin, personal and hair products (Boonme, 2007; Boonme, 2009; Boonme et al., 2009; Boonme and Songkro, 2010; Boonme et al., 2010). Hloucha et al. (2009) found that the microemulsion, dicaprylyl ether (and) coco glucoside (and) glyceryl oleate (Plantasil® Micro), could reduce the values of the residual combing work, implying to decrease the friction force between the hair and comb. A patent disclosed that conditioning performance of an aqueous shampoo composed of water, a surfactant, a cationic deposition polymer and a silicone component could be significantly boosted when using a silicone component in the form of a combination of emulsified silicone and microemulsified silicone (Gallagher et al., 2004). Ostergaard et al. (2004) reported that silicone quaternary microemulsion could be used as a multifunctional product for hair care, for example,

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it could be potentially used in a color retention conditioner. Silicone quaternary microemulsion, silicone quaternium-16 (and) undeceth-11 (and) butyloctanol (and) undeceth-5 (Dow Corning® 5-7113 Silicone Quat Microemulsion), is claimed that it can provide excellent wet and dry conditioning effects on the hair with the added benefit of body and volume enhancement (Dow Corning Corporation, 2006). Thus, it is an interesting ingredient which can be used in conditioning shampoos.

The objectives of this study were to investigate characteristics and to determine conditioning performance providence of shampoos containing various concentrations, i.e., 0, 1, 2, 3 and 4 %w/w, of silicone quaternary microemulsion.

## Materials and Methods

### Materials

Silicone quaternary microemulsion (Dow Corning® 5-7113 Silicone Quat Microemulsion, Dow Corning Corporation, USA) was obtained as a gift from Summit Chemical Co., Ltd., Thailand. Sodium lauryl ether sulfate (Texapon® N40), cocamide diethanolamine (Comperlan® KD), hydrolyzed collagen (Nutrilan® I-50), propylene glycol (and) 5-bromo-5-nitro-1,3-dioxane (Bronidox® L) were purchased from Cognis Thai Ltd., Thailand. Sodium chloride, citric acid and perfume were purchased from local distributors in Thailand. Distilled water was used throughout the experiment. All chemicals were pharmaceutical grade and used without further modification.

### Preparation of shampoos

Five samples, designated as S-0, S-1, S-2, S-3 and S-4, were prepared by mixing all components in the formulations as shown in Table 1 until clear shampoos were obtained. S-0 was control sample without silicone quaternary microemulsion while S-1, S-2, S-3 and S-4 contained silicone quaternary microemulsion in the concentrations of 1, 2, 3 and 4 %w/w, respectively. Each sample was prepared in triplicate.

**Table 1.** Formulations of the investigated shampoos

Ingredient	%w/w
Sodium lauryl ether sulfate	50.00
Cocamide diethanolamine	3.00
Hydrolyzed collagen	2.00
Propylene glycol (and) 5-bromo-5-nitro-1,3-dioxane	0.20
Sodium chloride	1.00
Citric acid	0.04
Perfume	qs
Silicone quaternary microemulsion	0, 1, 2, 3, 4
Water	to 100.00

### Characterization of shampoos

All samples were observed for clarity, color and odor. Their pH was measured by a pH meter (Mettler Toledo, USA). Foaming ability was evaluated by shaking 50 µl of each sample in 40 ml of water with a constant force for 5 minutes and then determining the height of generated foam above the water surface. The surface tension of the solution of 10% each sample in water was evaluated using a tensiometer (Lauda, Germany). Rheological property was measured using a programmable rheometer (Brookfield DV-III Ultra, Brookfield Engineering Laboratories Inc., USA) fitted with a spindle while set at different spindle speeds. All measurements were performed in triplicate at room temperature (Thai Industrial Standards Institute, 1988; Mainkar and Jolly, 2000; Aghel et al., 2007). One-way analysis of variance (ANOVA) and Tukey's multiple comparison test were used to compare characteristics of different formulations and a *p* value of 0.05 was considered to be significant.

### Evaluation of conditioning performance

A hair tress of a Thai woman was obtained from a local salon. It was cut into 6 swatches of the tresses with approximately the length of 15 cm and the weight of 5 g. A swatch without washing was used as the representative of original tress. Other five tresses were treated by washing with the samples using the identical manner. For each cycle, each tress was washed with the mixture of 20 g of a sample and 20 g of water using a sonicator for 5 minutes. Then, it was washed with 40 g of water using a sonicator for 5 minutes. The sonification force was constantly fixed throughout the experiment. Afterwards, each tress was left for air drying at room temperature. After each cycle, the appearance of all tresses was optically observed and the cuticle arrangement of each tress was investigated by a light microscope (Olympus, Japan) at a magnification of 400 compared to that before treatment. The tresses were washed for maximum ten cycles. The photographs were taken with a digital camera.

The 50 volunteers were invited to be the referees for determining the conditioning performance, i.e., smooth and softness, of the tresses by a blind test. They were asked to contact the six tresses labeled with random codes. One tress was the representative of original tress while other five tresses were washed with the studied shampoo by the designed process for ten cycles. Afterwards, they rated and scored for conditioning performance. For rating, they were asked to rank the conditioning performance of the tresses after contacting from 5 to 0 according to the best to the worst, respectively. No identical ratings were allowed. For scoring, they were asked to mark the conditioning performance of the tresses after contacting in 4-choice of satisfaction levels, i.e., excellence (score = 4), good (score = 3), fair (score = 2) and poor (score = 1). Identical scores were allowed.

## RESULTS AND DISCUSSION

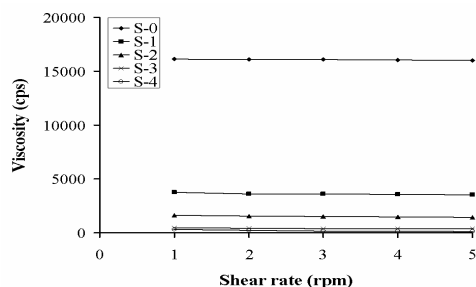
All samples were clear yellowish liquids. They had good odor since the perfume used could conceal the salty odor of silicone quaternary microemulsion. The values of pH, foam height

and surface tension were shown in Table 2. In TIS 162-2541, the pH values of shampoos are specified in the range of 5.0-8.0 (Thai Industrial Standards Institute, 1988). It was found that the averaged pH values of all samples were in the range of 6.59-7.17 which were acceptable according to TIS 162-2541 and implied to stabilize the ecological balance of the scalp. All shampoos were able to provide stable foam. Although foam generation does not relate to the cleansing ability of shampoos, it is of satisfaction to the consumers. The reduction in surface tension of water from 72.8 to around 29 dynes/cm by the shampoos indicated that they had good detergency action (Mainkar and Jolly, 2000; Aghel et al., 2007). In addition, a commercial shampoo was diluted with the identical method and then measured for surface tension. It was found that the 10% aqueous solution of the commercial shampoo provided the surface tension of  $30.2 \pm 0.15$  dynes/cm. All studied shampoos could insignificantly reduce the surface tension of water when comparing with each other ( $p > 0.05$ ) while they could significantly reduce the surface tension of water when comparing with the commercial shampoo ( $p < 0.05$ ). It could be caused by that the surfactants used in the studied shampoos and those in the commercial shampoo were different.

**Table 2.** Characteristics of the shampoos (n=3)

Sample	pH	Foam height (mm)	Surface tension (dynes/cm)
S-0	7.10±0.04	15.50±1.00	29.52±0.18
S-1	6.64±0.33	12.90±1.44	29.45±0.17
S-2	7.17±0.50	10.50±2.50	29.34±0.16
S-3	6.59±0.12	9.67±0.58	29.47±0.07
S-4	7.11±0.19	11.30±0.79	29.25±0.10

The rheological profiles were exhibited in Figure 1. It was found that all samples had low viscosity with Newtonian flow. Addition of silicone quaternary microemulsion in the shampoo formulation significantly decreased viscosity ( $p < 0.05$ ). The compositions in silicone quaternary microemulsion may cause the splitting of the polymers in the formulation into the building blocks (Martin, 1993). However, all samples still could provide ease of flow on removal from containers and spreading on application to the hair tresses.

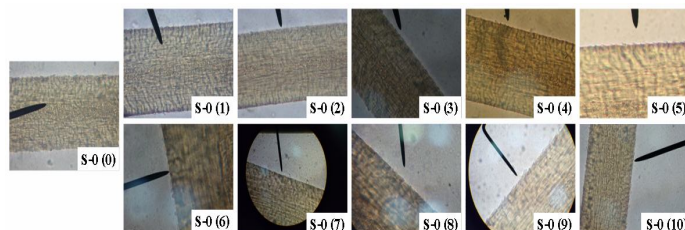


**Fig 1.** Rheological profiles of the shampoos. Each data point was the mean ± S.E.M. (n=3).

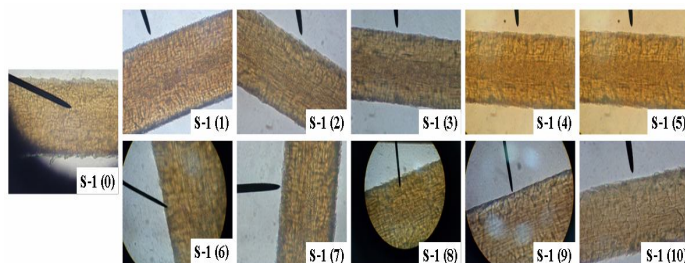
It was seen that, the representative of original tress or the tress with no washing looked rougher than the tresses washed by the studied shampoos as shown in Figure 2. Figures 3-7 exhibited the comparison of the cuticle arrangement of the tresses before and after washing with the studied shampoos. It could be seen that the cuticle arrangement of five tresses washed by all studied shampoos seemed to be better than their counterpart tresses before washing. However, no obvious difference on the cuticle arrangement of the tresses after washing with different shampoos was observed. Washing the tresses with all shampoos could improve the closing of the flat overlapping cells on the outermost layer of the hair.



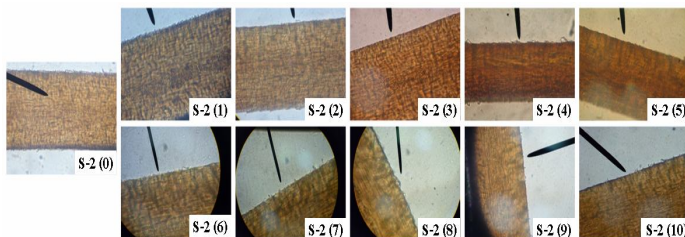
**Fig 2.** Optical appearance of the representative of original tress (A) and the tresses washed by (B) S-0, (C) S-1, (D) S-2, (E) S-3 and (F) S-4 by the designed process for ten cycles.



**Fig 3.** Cuticle arrangement of the tresses under light microscope with magnification of 400 before and after washing with S-0. The number in the bracket referred to number of washing cycles.



**Fig 4.** Cuticle arrangement of the tresses under light microscope with magnification of 400 before and after washing with S-1. The number in the bracket referred to number of washing cycles.



**Fig 5.** Cuticle arrangement of the tresses under light microscope with magnification of 400 before and after washing with S-2. The number in the bracket referred to number of washing cycles.

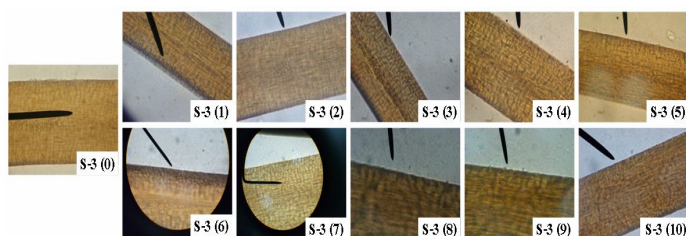


Fig 6. Cuticle arrangement of the tresses under light microscope with magnification of 400 before and after washing with S-3. The number in the bracket referred to number of washing cycles.

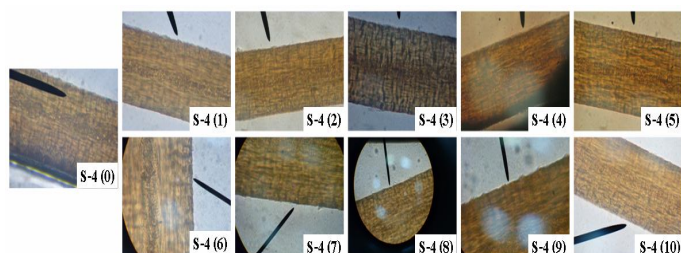


Fig 7. Cuticle arrangement of the tresses under light microscope with magnification of 400 before and after washing with S-4. The number in the bracket referred to number of washing cycles.

Tables 3 and 4 exhibited the ratings and scores of the referees in order to determine the conditioning performance of the tresses via contacting. Most referees (86%) rated that the tress washed with S-1 provided the highest conditioning performance. The score of the conditioning performance of the tress washed with S-1 was averaged at 3.68 from 4, indicating good-to-excellence level. The tresses washed with other samples, i.e., S-0, S-2, S-3 and S-4, were scored in similar fair-to-good level of the conditioning performance. The representative of original tress was determined that it provided the lowest conditioning performance since it was not treated with the studied shampoos. Hence, it did not get any hair-conditioning agents for enhancing the closing of the cells on cuticle layer.

Table 3. The numbers of the ratings of the referees' opinion on the conditioning performance of the tresses after contacting (n=50)

Rating	S-0	S-1	S-2	S-3	S-4	No washing
0	0	0	1	0	0	49
1	8	1	8	11	21	1
2	11	1	12	20	6	0
3	15	2	15	7	11	0
4	15	3	13	11	8	0
5	1	43	1	1	4	0

Note: The ratings of the conditioning performance of the tresses after contacting from 5 to 0 referred to the best to the worst, respectively.

Table 4. The numbers and means of the scores of the referees' opinion on the conditioning performance of the tresses after contacting (n=50)

Score	S-0	S-1	S-2	S-3	S-4	No washing
1	3	0	0	11	2	46
2	23	0	23	26	17	4
3	21	16	23	12	27	0
4	3	34	4	1	4	0
Mean	2.48	3.68	2.62	2.06	2.66	1.08

Note: The scores of the conditioning performance of the tresses after contacting were 4-choice of satisfaction levels, i.e., excellence (score = 4), good (score = 3), fair (score = 2) and poor (score = 1).

Silicone quaternary microemulsion is claimed by its manufacturer that its recommended use levels are from 2 to 8 %w/w (Dow Corning Corporation, 2006). In this study, hydrolyzed collagen was added in all shampoos. Therefore, the feeling of conditioning performance could be found when washing the tresses with all samples. Silicone quaternary microemulsion in the concentration of 1%w/w was optimum for incorporating in the investigated formulation.

## CONCLUSIONS

It could be concluded that silicone quaternary microemulsion could be used as an effective hair-conditioning agent in shampoos. The suitable concentration of silicone quaternary microemulsion for mixing in the investigated formulation was 1%w/w since it provided the highest satisfaction of the 50 referees on conditioning performance of the tresses via contacting. Moreover, the obtained shampoo possessed acceptable pH, foaming ability, surface tension reduction and viscosity. However, addition of silicone quaternary microemulsion in shampoos might change some characteristics of the blank counterpart. For instance, the decrease of viscosity was found when silicone quaternary microemulsion was incorporated into this typical formulation. Therefore, the thorough study is required before use.

## ACKNOWLEDGEMENT

The authors thank Summit Chemical Co., Ltd., Thailand for generously supplying Dow Corning® 5-7113 Silicone Quat Microemulsion.

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