Effect of Lecithin on Dermal Safety of Nanoemulsion Prepared from Hydrogenated Lecithin and Silicone Oil

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In this study, a hydrogenated lecithin-containing nanoemulsion was prepared from hydrogenated lecithin and silicone oil. Tween-60 and liquid paraffin, widely known emulsifiers, were used as standard substances, and high shear was produced by utilizing a high shear homogenizer and microfluidizer. The properties of the nanoemulsion prepared with hydrogenated lecithin were evaluated by measuring interfacial tension, dynamic interfacial tension, droplet size, zeta-potential, friction force, skin surface hygrometery, and dermal safety. The interfacial tension of lecinol S10/silicone oil was lower than that of lecinol S10/liquid paraffin. The nanoemulsion prepared from hydrogenated lecithin shows lower zeta-potential, skin surface hygrometery, and friction force compared with a general emulsion. The silicone nanoemulsion prepared from hydrogenated lecithin showed a zero value in the patch test and thus exhibits high dermal safety.

Key Words: Lecithin, Nanoemulsion, Interfacial tension, Zeta-potential, Skin safety

Introduction

Nanoemulsions used in cosmetics must afford safety, stability, and usability. The safety is related to the skin of the human body, the stability relates to the form of the nanoemulsion, and usability is associated with the efficiency and effect of the nanoemulsion.[1] Lecithin is primary a mixture of glycolipids, triglycerides, and phospholipids.[2,3] Recently, naturally original hydrogenated lecithin, which in terms of its basic elements is similar to the body's constitution, has attracted attention as an emulsifier.[4,5] Hydrogenated lecithin shows higher safety and usability than common emulsifiers. However, compared with common emulsifiers, it has lower stability. Hydrogenated lecithin offers excellent dermal safety compared with common emulsifiers as its composition corresponds well with the body's constitution. Hydrogenated lecithin not only can serve as an emulsifier, it also combines with water molecules and thus has plow power and pliability. However, hydrogenated lecithin has major drawbacks of high cost and deficient physical properties, such as surface and interfacial surface tension, compared with common emulsifiers. As such, it has not seen wide use as a general emulsifier.[6,7]

Recent advances in the mechanical industry have led to the development of advanced emulsifying equipment. Presently, hydrogenated lecithin-based vesicles and nanoemulsions can be prepared by utilizing the geometric structure of hydrogenated lecithin molecules and high shear. Nanoemulsions prepared with emulsifying equipment that provides high shear show better long-term stability than that of general emulsions.[8,9]

In a previous study, we reported on the preparation and characterization of a hydrogenated lecithin-containing liposome.[10] In the present study, a hydrogenated lecithin-containing nanoemulsion was prepared using a homogenizer and microfluidizer at high shear. The properties of the nanoemulsion were evaluated in terms of surface tension, droplet size, friction force, zeta-potential, color, skin surface hygrometery, and dermal safety.

Experimental

Materials. Hydrogenated lecithin used in this study was commercially available lecinol S10 (Nikkol Co., PC content: 39%). Phenyltrimethicone (silicone oil, molecular weight of 556, density of 0.985, Dow Corning Co.) was used as oil. Tween-60 (polysorbate 60, Uniqema Co.) was used as a standard emulsifier, and liquid paraffin (density of 0.83, mean molecular weight of 260-280, Penreco Co.) was used as oil. A V79-4 cell was supplied by American Type Culture Collection. Distilled water (resistivity: 18.0 MΩ·cm, pH: 6.7) was prepared with a Milli Q Plus system. The chemical structures of hydrogenated lecithin, Tween-60, and silicone oil are shown in Figure 1.

Preparation of the nanoemulsion. Designated amounts of hydrogenated lecithin and water were placed in a 2000 mL beaker. The solution was mixed with a mechanical stirrer at 70 °C for 1 h. Oil was added to the mixture, which was then mixed using a homo mixer. The droplet size of the coarse emulsion was 15 μm. The mixture was further stirred using high pressure apparatuses (homogenizer and microfluidizer). The droplet size of the nanoemulsion was 0.2 ~ 0.35 μm.

Characterization and measurements. The interfacial tension of the surfactant containing aqueous phase and oil phase was measured using a Surface Tensiometer (K12) *via* the ring method at 25 °C. The dynamic interfacial tension was measured using a Drop Volume Tensiometer (KRÜSS Co., Model DVD 10) at 25 °C.

The droplet size of the emulsion was determined using a Submicron Particle Sizer (Nicomp Co., Model 370) and Mastersizer (Malvern Co.).

The zeta-potential of the nanoemulsion prepared using the microfluidizer were determined with a Zeta Sizer (Malvern Co.).

Emulsification ability and stability were evaluated using the following method. Nanoemulsion samples were placed on a mass cylinder, and the extent of oil separation over time was checked by the naked eye. Friction force was measured using a Universal Tester (Chatillon Co., Model V-1000) under a load of 30 gf/cm^2, temperature of 25 °C, and relative humidity of 30%.

Skin surface hygrometery. The moisture content, absorption ability, and moisture maintenance ability of an in vivo sample was investigated using five men and five women under a temperature of 25 °C and a relative humidity of 67% by an in vivo water sorption-desorption test. The test subject rested in a constant temperature permanent moist chamber for 30 min, and the product was doped on a 4 cm × 4 cm area of the subject's skin. The conductivity along with time was determined using a Skincon-200 (IBS Co.) as means of assessing the moisturizing effect.

Patch test. A fixed quality of the nanoemulsion and a standard substance were doped on the back of a human subject, and dermal safety was determined after 48 h according to a procedure reported by the International Contact Dermatitis Research Group (ICDRG).

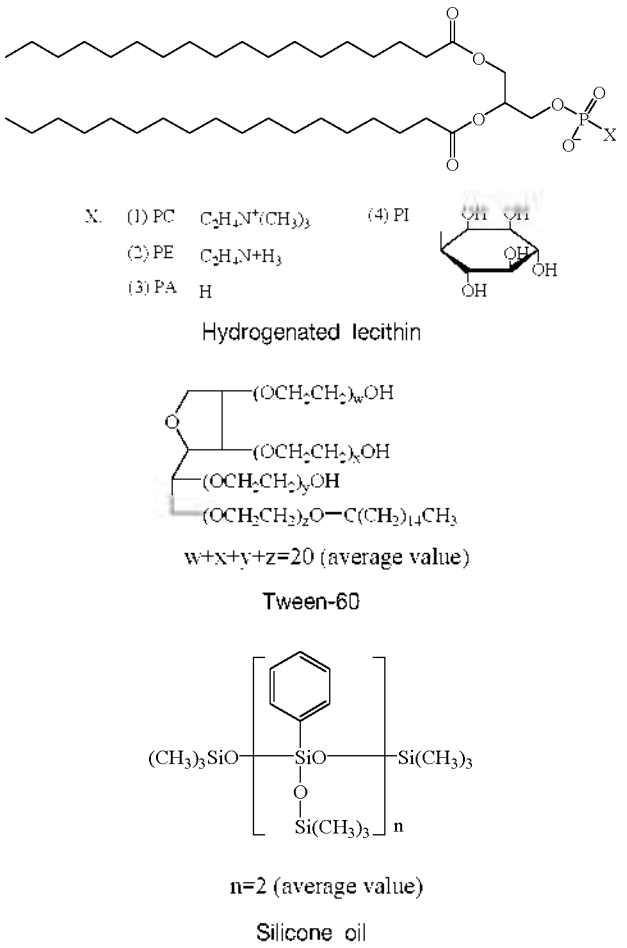

Figure 1. Chemical structures of the materials used.

Results and Discussion

Effects of lecithin on interfacial tension of the nanoemulsion. Marangoni-Gibbs effects are closely related to interfacial stability in the nanoemulsion. Interfacial tension between the surfactant solution and liquid paraffin, and silicone oil, respectively, was measured by the ring method, and the results are shown in Figure 2. Interfacial tension of both systems decreased sharply with increasing concentration of lecithin up to 0.1 wt% concentration of lecithin and thereafter decreased slowly above this concentration. Interfacial tension of lecinol S10/silicone oil was lower than that of lecinol S10/liquid paraffin at the same concentration of lecithin. Figures 3 and 4 show the effects of the emulsifiers and oils on the dynamic interfacial tension. The dynamic interfacial tension of all systems increased with increasing flow rate. The dynamic interfacial tension of lecinol S10-containing systems was lower than that of the Tween-60-containing system at the same flow rate. From these results it can be concluded that the lecinol S10 and silicone oil-containing nanoemulsions have small particle size and excellent stability.[10,11]

Effects of emulsifying equipment on droplet size of the nanoemulsion. Methods for the preparation of small droplet size can be divided into emulsification methods using interfacial chemical characteristics and preparation methods using mechanical energy. The mean droplet size of hydrogenated lecithin-containing emulsions using various emulsifying equipments at a temperature above the transition temperature (T_c, 65 °C) is shown in Table 1.[10] When use a homomixer, the droplet size is large and the distribution of the droplet size is broad, thus leading to instability brought about by separation after 8 h. Hydrogenated lecithin and silicone oil form a nanoemulsion when using relatively high shear is applied. From these results it is determined that a small droplet can be prepared using 0.3 wt% hydrogenated lecithin above T_c.[12,13]

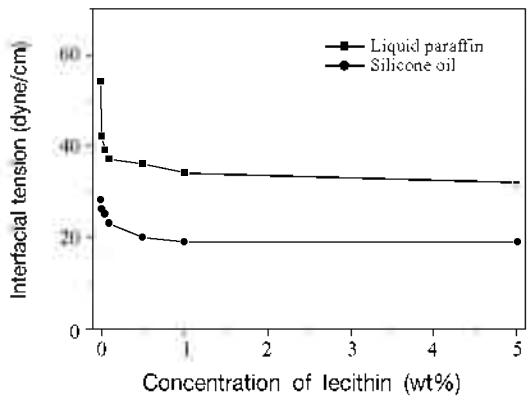

Figure 2. Interfacial tension between surfactants solution and liquid paraffin, silicone oil measured by ring method.

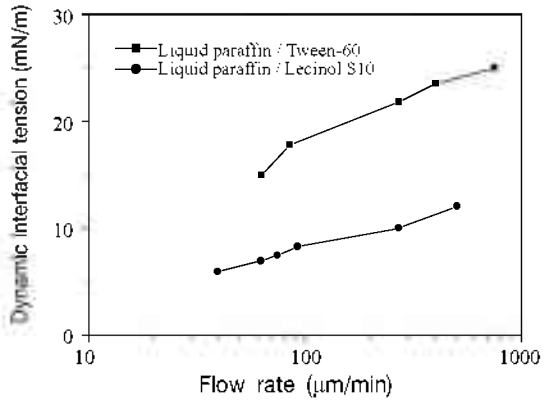

Figure 3. Dynamic interfacial tension of 0.5 wt% emulsifiers-containing solution at 25 °C.

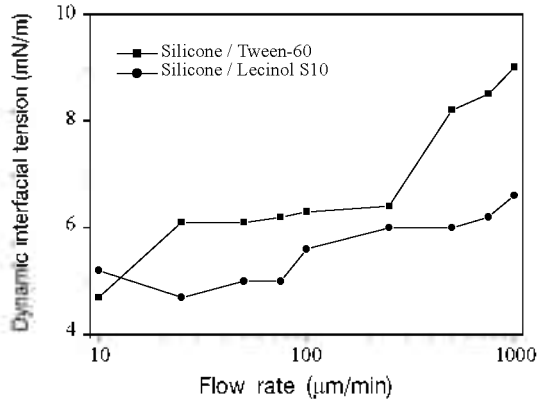

Figure 4. Dynamic interfacial tension of silicone oil/0.5 wt% emulsifiers-containing solution at 25 °C.

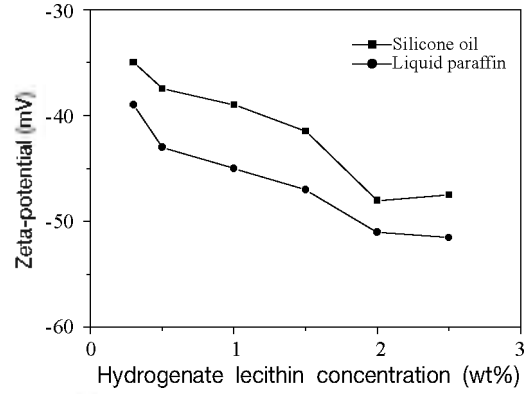

Figure 5. Effect of hydrogenated lecithin concentration on zeta-potential of emulsion containing 10 wt% liquid paraffin and silicone oil.

Effects of lecithin on zeta-potential and color of the nanoemulsion. Zeta-potential is an index of the stability of a nanoemulsion. The effect of hydrogenated lecithin concentration on the zeta-potential of the emulsion droplets is shown in Figure 5. The zeta-potential of the nanoemulsion prepared from silicone oil was higher than that of the emulsion prepared from liquid paraffin, and the former shows relatively high stability.[14]

Particle size can be discriminated approximately from color of emulsion. Table 2 shows the results of color for samples prepared using a microfluidizer and homomixer. The results indicate that the samples prepared with the microfluidizer are characterized by a darker green color than that of the samples prepared with the homomixer.

Table 1. Mean droplet size of hydrogenated lecithin-based emulsions emulsified using various emulsifying equipments

Emulsifying equipments	Mean particle size (μm)
Microfluidizer (100 MPa, 1 pass)	0.28
Homogenizer (100 MPa, 1 pass)	0.35
Homo mixer (5000 rpm, 10 min, 70 °C)	8.0

Note: The contents of silicone oil, emulsifier, and hydrogenated lecithin were 10 wt%, 1 wt%, and 3 wt%, respectively.

Table 2. Effect of high pressure treatment and homomixer treatment on color of samples

Color value[a]	Silicone oil		Color value	Liquid paraffin	
	HPT[b]	HMT[c]		HPT	HMT
L*	45.84	34.79	L*	29.32	23.48
a*	9.04	10.83	a*	12.14	13.23
b*	4.68	16.64	b*	12.34	18.45

[a]L* = visual lightness, a* = redness to greenness, b* = yellowness to blueness. [b]HPT = high pressure treatment. [c]HMT = homomixer treatment.

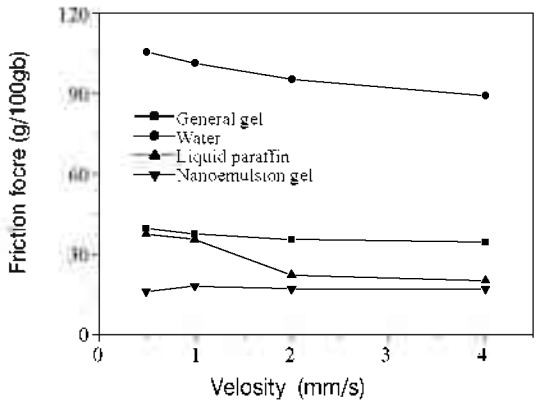

Figure 6. Results of friction test for nanoemulsion gel and general gel.

Friction force. Friction tests for a nanoemulsion gel and general gel were carried out using a Universal Tester, and the friction force was calculated according to the following equation.[15]

$$F = \mu W \quad (1)$$

where F is the friction force, μ is the coefficient of friction, and W is the vertical load.

Figure 6 shows the results of the friction force test. The nanoemulsion gel shows the lowest friction force values in this study, indicating that the nanoemulsion gel is smoother than a general gel. This can be attributed to the effect of hydronated lecithin and nanoemulsion.

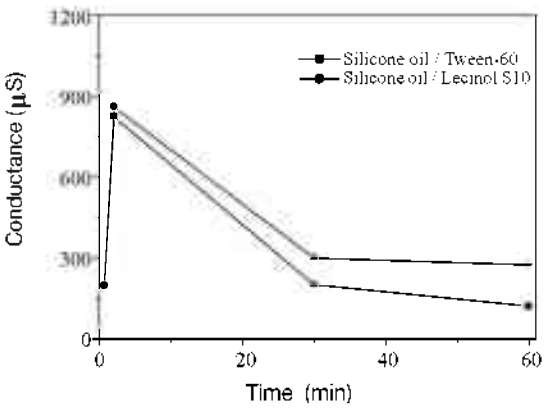

Figure 7. Conductance of silicone oil/Tween-60 and silicone oil/lecinol S10 as a function of time.

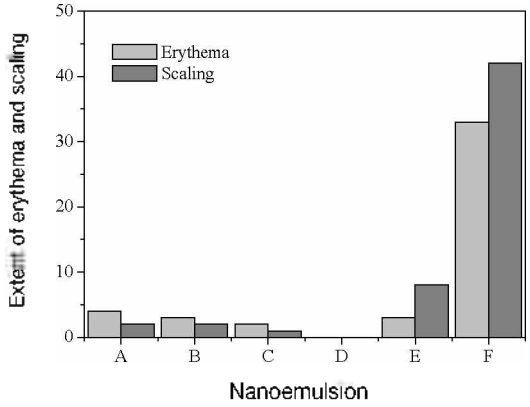

Figure 8. Emulsifiers, oil, and their irritation potential assessed form extent of erythema and scaling. (A: Liquid paraffin/Tween-60, B: Silicone oil/Tween-60, C: Liquid paraffin/Lecinol S10, D: Silicone oil/Lecinol S10, E: Saponins-Xanthan gum/Liquid paraffin, F: Sodium laureth sulphate/Liquid paraffin).

Skin surface hygrometery. Skin surface hygrometery of the nanoemulsion was determined by measuring conductance. Figure 7 shows the conductance of the nanoemulsion prepared from hydrogenated lecithin and the emulsion prepared from Tween-60 doped skin as a function of time. The conductance at the time of initial doping was increased, which was caused by water, and the conductance decreased with vaporization of water. The nanoemulsion prepared from hydrogenated lecithin for more than 30 min shows higher skin surface hygrometery than that of the general emulsion, which is attributed to hydrogenated lecithin combining with water to form bound water.[16]

Dermal safety. Dermal safety of the emulsifiers and oils was evaluated by a patch test. Dermal safety was divided into erythema and scaling, and the evaluation scale included five grades (0: no response, 1: erythema, 2: erythema + papule, 3: erythema + papule + small bleb, 4: bog bleb). Erythema is redness of the skin caused by increased blood flow to the capillaries. Figure 8 shows the extent of erythema and scaling by the emulsifiers and oils. The silicone nanoemulsion prepared from hydrogenated lecithin shows the smallest erythema and scaling values and thus exhibits high dermal safety in this study. Hydrogenated lecithin's basic elements are similar to the body's constitution. The hydrogenated lecithin-containing silicone nanoemulsion is not causes any side effects, such as erythema, papule, or bleb due to hydrogenated lecithin's affinity to skin.[17]

Conclusions

A hydrogenated lecithin-containing nanoemulsion was prepared using high pressure apparatuses, and cosmetic related properties were investigated. The interfacial tension of lecinol S10/silicone oil was lower than that of lecinol S10/liquid paraffin. The zeta-potential of the nanoemulsion prepared from silicone oil was lower than that of the emulsion prepared from liquid paraffin, and the former shows relatively high stability. The samples prepared with a microfluidizer showed the highest efficiency and a dark green color. The nanoemulsion prepared also showed lower skin surface hygrometery and friction force compared with the general emulsion. The silicone nanoemulsion prepared from hydrogenated lecithin showed a zero value in a patch test and thus exhibits high dermal safety.

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