J. of the Korean Oil Chemists' Soc.,
Vol. 28, No. 1. March, 2011. 1~5


# Properties of Oil-in-Water Nano-emulsions Prepared from Hydrogenated Lecithin with High Pressure Homogenizer

Wan-Goo Cho†· Duck-Whan Bae*

†*School of Alternative Health Management, College of Alternative Medicine, Jeonju University, 3-ga, Hyoja-dong, Wansan-gu, Jeonju, 560-759, Korea*
**Bangwool Land, Migun Techno 419, Yongsan-dong, Yusong-gu, Daejeon, 305-500, Korea*
*(Received October 13, 2010 ; Accepted December 20, 2010)*

**Abstract** : In this study, We investigated the properites of nano-emulsions containing hydrogenated lecithin prepared by high pressure homogenizer. The size of droplet of emulsions prepared by homogenizer at various rpm (rotation per minute) was not measured due to the unstability of emulsions, however, the size of droplet of nano-emulsions prepared by high pressure homogenizer was around 300 nm and the appearance of emulsions was bluish. The stability of emulsions with various lecithin concentration was tested against time. POV (Peroxide value) of emulsions were plotted against time. POVs of emulsions prepared with an egg lecithin and a soy lecithin were increased with time, however, POV of emulsion with Lecinol S-10□ was kept constant within 60 hours and at 60 °C. In consumer test, the nano-emulsion showed higher affinity regardless of skin type. Both of irritation scores of emulsions were similar.

*Keywords* : *nano-emulsions, lecithin, POV, consumer test, irritation*

## 1. Introduction

Nano-emulsions are a class of emulsions that can be translucent or transparent and the droplet sizes are within the range of 20-200 nm[1]. Nano-emulsions with small droplet size obtained by the so-called condensation methods(e.g. phase inversion temperature(PIT) or phase inversion composition(PIC) methods[2]. The extremely small droplet size which can be overlap those of micro-emulsions. Unlike micro-emulsions, which are thermodynamically stable, nano-emulsions are only kinetically stable. The stability of nano-emulsions are mainly governed by Ostwald ripening[3,4]. The long-term stability of nano-emulsions with no apparent flocculation or coalescence makes them unique.

The attraction of nano-emulsions for application in various industrial fields has been focused on, for example, reaction media for polymerization[5], personal care and cosmetics[6-8], drug delivery system[9], and agrochemical[10]. At present, new applications are being developed to use nano-emulsions as consumer products.

In this paper, we will report results on the

† Corresponding author
(E-mail : wgcho@jj.ac.kr)

properties of nano-emulsions containing hydrogenated lecithin preparing with high pressure homogenizer. Particular attention has been paid to the stability and oxidative stability that commonly encountered within food and cosmetic nano-emulsion systems.

## 2. Materials and Methods

### 2.1. Materials

Egg lecithin and soy lecithin were purchased from Aldrich-Sigma and Lipo (USA), respectively. Hydrogenated lecithin (Lecinol S-10®) and PEG 60 hydrogenated caster oil(Nikkol HCO 60®) were obtained from Nikko Chem.(Japan). Cosmetic grade of light mineral oil from Kuk-dong (Korea) and silicone oil (Silicone 556®) from Dow Corning (USA) were used. All these materials were used as received. Water was deionized and Milli-Q filtered.

### 2.2. Nano-emulsion preparation

Nano-emulsion(B) was prepared from a mixture of 2.0 wt % of mineral oil and 0.5 wt % of hydrogenated lecithin by slowly adding 97.5 wt % of water with agitation using magnetic stirrer. The addition rate was kept constant at 1.0 $mlmin^{-1}$. The emulsification temperature was kept at 23 °C. After premixing of all materials, the emulsions were mixed by homogenizer (TK Robomixs, Japan), high pressure homogenizer(HS 3011, Hwasung, Korea) and (Microfluidizer, Microfluidics, M 110P, USA) on the condition of 500 bar, respectively.

### 2.3. Normal emulsion preparation

Emulsion(A) was prepared from mixture of 0.5 wt % of silicone oil and 0.5 wt % of PEG 60 hydrogenated caster oil by slowly adding 99.0 wt % of water with agitation using magnetic stirrer.

### 2.4. Droplet size measurement

The mean droplet size and distribution of the nano-emulsions were determined by dynamic light scattering (DLS, ESLZ, Photal, Japan).

### 2.5. Free fatty acid value measurement

Free fatty acid value(AV) and conjugated dienoic acid value (CDNV) were determined according to the AOAC method[11].

### 2.6. Human patch test

The patch test procedure involves application of 0.2 g on a 25 mm Plain Hill Top Chamber (Hill Top Companies, Cincinnati, OH, USA) to the skin of the upper outer arm of 30 human volunteers for up to 4 hours. Treatment sites are assessed for the presence of irritation using a four point scale (Table I) at 24, 48 and 72 hours after patch removal. A volunteer with a '+' or greater reaction at any of the assessments is considered to have demonstrated a 'positive' irritant reaction and treatment with the causative substance does not proceed on

Table 1. Assessment of Reactions

| Grading | Description of response | Score |
|---|---|---|
| 0 | No reaction | 0 |
| + | Weakly positive reaction (usually characterized by mild erythema or dryness across most of the treatment site) | 0.05 |
| ++ | Moderately positive reaction (usually distinct erythema possibly spreading beyond the treatment site) | 0.10 |
| +++ | Strongly positive reaction (strong, often spreading erythema with oedema) | 0.20 |

that person. For panellists with a '+' or greater response at application times of less than 4 hours to a particular test substance, it is assumed that they would present a stronger irritant reaction if exposed for 4 hours. However, once a '+' or greater response i,; obtained, there is no need to subject these panellists to further treatment with that substance. In evaluating the results, what is measured is the number of panellists who had, or would have had, a positive 'irritant' reaction after a 4 hour exposure.

### 2.7. Consumer test

For the sensory evaluation, panel was consisted of 38 women who had been selected for their high degree of sensory acuity. Panels were given by reflecting the skin types. Their mean age was 41 year, range 25~52. Two products (A and B) were dispensed to the arms of the volunteers. The subjects were asked to assess the degree of liking during spreading. The results were marked on an analogue scale.

## 3. Results & Discussion

### 3.1. Properties of O/W nano-emulsions using various homogenizer

Nano-emulsion was prepared from a mixture of 2.0 wt % of mineral oil and 0.5 wt % of hydrogenated lecithin by slowly adding 98.5 wt % of water with agitation using magnetic stirrer. The addition rate was kept constant at 1.0 $mlmin^{-1}$. The emulsification temperature was kept at 23 °C. After premixing of all materials, the emulsions were mixed by homogenizer (TK Robomixs, Japan), high pressure homogenizer (HS 3011, Hwasung, Korea) and (Microfluidizer, Microfluidics, M110P, USA) on the condition of 500 bar, respectively. The size of droplet of emulsions prepared by homogenizer varying rpm was not measured due to the unstability of emulsions. The size of droplet of emulsions prepared by high pressure homogenizer was around 300 nm as shown in Table 1. The appearance of emulsions was bluish. Homogenization is a fluid mechanical process that involves the subdivision of particles or droplets into micron sizes to create a stable dispersion or emulsion for further processing. This is an important stage in the treatment of cosmetics, food and dairy products. It provides improved product stability, shelf life, digestion, and taste.

### 3.2. Comparison of POV of emulsions against time

Lecithins are emulsifiers of natural origin that are widely used in cosmetics, pharmaceutical and food emulsion systems. Lecithin contains a high percentage of polyunsaturated fatty acids (PUFAs), especially icosapentaenoic acid (EPA) and docosasahexaenoic acid (DHA).

Emulsions with various lecithin were tested against time. In Fig 1, POV (Peroxide value) of emulsions were plotted against time. POVs of emulsions prepared with a egg lecithin and a soy lecithin were increased with time, however, POV of emulsion with Lecinol S-10 was kept constant within 60 hours and at 60 °C.

Table 2. Size of Droplet of Emulsions Prepared by Various Homogenizer

| | Homogenizer | | High Pressure Homogenizer | |
|---|---|---|---|---|
| | 3,000 rpm | 10,000 rpm | M110P | HS 3011 |
| Size of droplet(μm) | - | - | 0.30 | 0.34 |

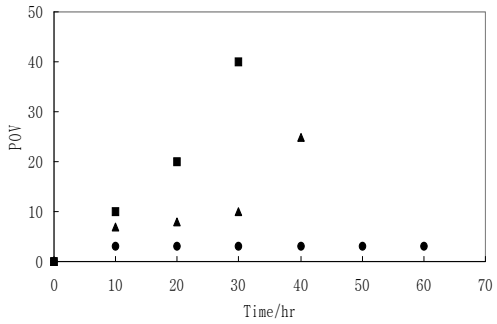


Fig. 1. POV of emulsions with lecithin against time at 60 ℃.

### 3.3. Comparison of consumer test

The skin sensory performance of personal care products is an important factor for the sales potential of any cosmetic product. Efficacy claims can be made for these products based on clinical trials. However, if the clinical activity in one way or another can be supported by sensorial perception, this personal care product is more likely to be successful in the market place. Skin sensory characteristics of a emulsion are the result of the ingredients in the marketed products as well as the physical form in which they have been formulated. The objective of this study was to identify the sensory characteristics of two types of emulsions. Panel consisted of 38 women who had been selected for their high degree of sensory acuity. Panels were given by reflecting the skin types. In Fig. 2, the nanoemulsion showed higher affinity regardless of skin type.

### 3.4. Patch test of emulsions

The Table 3 contains data from a test. The scores were the arithmetic average of volunteers. Both of irritation scores of emulsions were similar.

## 4. Conclusions

From the above experiment, we could obtain the following results.

1. The size of droplet of emulsions prepared by homogenizer varying rpm was not measured due to the unstability of

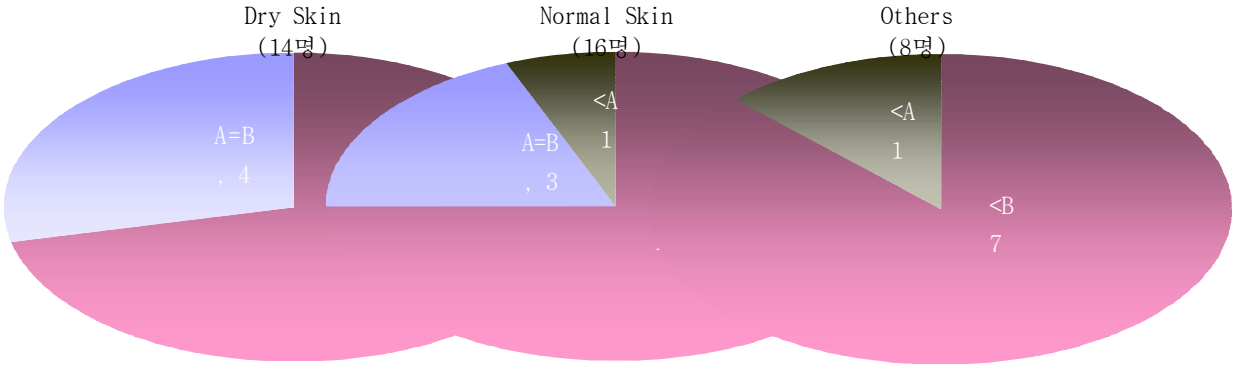


Fig. 2. Rating comparison of sensory between nanoemulsion(B) and macroemulsion(A).

Table 3. Score of Skin Irritation

| Items | Score |
|---|---|
| Emulsion prepared by homogenizer(A) | 0.05 |
| Emulsion prepared by high pressure homogenizer(B) | 0.04 |

emulsions. The size of droplet of emulsions prepared by high pressure homogenizer was around 300 nm and the appearance of emulsions was bluish.

2. Emulsions with various lecithin were tested against time. POV (Peroxide value) of emulsions were plotted against time. POVs of emulsions prepared with a egg lecithin and a soy lecithin were increased with time, POV of emulsion with Lecinol S-10 was kept constant within 60 hours and at 60 °C.
3. In consumer test, the nanoemulsion showed higher affinity regardless of skin type.
4. Both of irritation scores of emulsions were similar.

## References


1. A. Forgiarini, J. Esquena, C. Gonzalez, and C. Solans, Formation of Nano-emulsions by Low-Energy Emulsification Methods at Constant Temperature, *Langmuir*, **17(7)**, 2076 (2001).
2. T. J. Mason, J. N. Wilking, K. Meleson, C. B. Chang, and S.M. Graves, Nanoemulsion: Formation, Structure and Physical Properties, *J. Physics Condensed Matter*, **18**, 635 (2006).
3. T. Tadros, P. Izquierdo, J. Esquena, and C. Solans, Formation and Stability of Nanoemulsions, *Adv. in Colloid and Interface Sci.*, **108-109**, 303 (2004).
4. C. Solans, P. Izquierdo, J. Nolla, N. Azemar, and M. J. Garcia-Celma, Nanoemulsions, *Curr. Opinion in Colloid & Interface Sci.*, **10**, 102 (2005).
5. J. Ugelatad, M. S. El-Aaser, and J. W. Vaderhoff, Emulsion Polymerization, *J. Polymer Sci. Polymer Letter*, **11**, 503 (1973).
6. S. M. Graves, K. Meleson, J. N. Wilking, M. Y. Lin, and T. J. Mason, Structure of Concentrated Nano-emulsions, *J. Chem. Phys.*, **122**, 134703 (2005).
7. T. J. Mason, S. M. Graves, J. N. Wilking, and M. Y. Lin, Effective Structure Factor of Osmotically Deformed Nano-emulsions, *J. Phys. Chem. B*, **110**, 22097 (2006).
8. O. Sonneville-Aubrut, J. T. Simmonet, and F. L. Alloret, Nanoemulsions: a New Vehicle for Skin Care Products, *Adv. in Colloid & Interface Sci.*, **108-109**, 145 (2004).
9. S. Shafiq, F. Shakeel, H. Teixeira, F. J. Ahmad, R. K. Khar, and M. Ali, Development and Bioavailability Assessment of Ramipril Nanoemulsion Formulation, *European J. Pharma. and Biopharm.*, **66**, 227 (2007).
10. L. Wang, X. Li, G. Zhang, J. Dong, and J. Eastoe, Oil in Water Nano-emulsions for Pesticide Formulations, *J. Colloid & Interface Sci.*, **314**, 230 (2007).
11. A.O.C.S,: "AOCS official and tentative method", 10th edition, *Am. Oil Chem. Soc.*, Chicago (1990).