INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

SKIN EFFECTIVNESS OF FINE HYDRO-GEL CREAM CONTAINING BOTANICAL OIL COMPLEXES USING PIT EMULSIFYING SYSTEM

In-Young Kim[1*], Hyun-Dae Cho[2]
[1*] R&D Center, Biobeautech Co., Ltd., Jungwon-gu, Seongnam-si, Gyeonggi-do, 462-807, South Korea
[2] CIR Center, Cosmecca Korea Co., Ltd., Eumseong-gun, Chungcheongbuk-do, 137-865, South Korea
*Correspondence Author: iykim200@naver.com

Keywords: Phase inversion temperature, Fine nano-emulsion, Moisture, Multi-functional effect, Skincare effect, Anti-aging

Abstract
This study was to get unique formulation of multi-functional activity for preparing hydro-gel cream using PIT emulsifying system. In order to develop the good safety, adsorption and multi-functions, we made a new formulation using phase inversion temperature (PIT) containing 4 botanical oils such as camellia japonica seed oil, macadamia integrifolia seed oil, limnanthes alba (meadowfoam) seed oil, argania spinosa kernel oil, 0.04wt% of adenosine and 2wt% of niacinamide. The fine botanical nutritive emulsion using PIT emulsifying method is easy absorbed into the stratum corneum because of fine droplet size. Appearance was high concentrated bluish liquid emulsion. PIT botanical oil complex was formed at around from 64 to 72°C. pH was 5.7±0.5. This formulation was very safety to protect on the skin, major droplet was mean 105nm containing 20wt% of PIT botanical complex. And also, there are fine wrinkle improvement and whitening effect containing. Skin in-vivo evaluations carried out the efficacy and functions of hydro-gel cream such as moisturizing effect, TEWL, fine wrinkle improvement and whitening activity. This hydro-gel cream is to find an optimum way to enhance the strengthening effect on skin barrier functions of cosmetic formulations.

Introduction
Controlling cosmetics keeping water and oil balance is one of the most important functions of skincare cosmetic field [1]. But, in specific formulation such as skin toner, essence, and skin lotion having a water/oil balance, it is not easy to find the skin care cosmetic fields having multi-functional activity such as moisturizing, whitening, TEWL and anti-aging efficacy. Recently, various high functional cosmetics have been launching having multi-functional effects such as skin care and make-up care cosmetic fields [2]. Also, PIT technology of Korea is to be significantly extended with a major trend in the world market. This study is to prepare method of these hydro-gel creams having multifunctional activity. The phase inversion temperature (PIT) method was introduced by Shinoda et al [3]. They used non-ionic ethoxylated surfactants whose properties at the droplet interface are highly dependent on temperature [4]. There are a lot of customers to prefer the multi-function products having high moisturizing activity, anti-wrinkle and whitening actions although it is makeup cosmetics [5-6]. This study is to prepare method of hydro-gel cream having skin care effect. Recently, application formulation applying phase inversion temperature (PIT) emulsifying system in skin care cosmetics industry have not been sufficiently[7]. Also, Hydro-gel cosmetic industry has been a major trend focused on skin moisturizing activity in world market [8-10]. There are a lot of customers to prefer the multi-function products having high moisturizing activity, anti-wrinkle and whitening actions. Our major idea of this study provides specific characteristics as follows, having multiple functions as well as sensorial effect. PIT botanical complex contained 4 kinds of plant seed oils having skin care effect would be quickly and easily absorbed more in the skin. Especially, clinical tests of our developed formulation were described about skin moisturizing activity, TEWL activity, whitening, the fine wrinkle improvement, having the effects of one product with multiple functions.

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

Materials and Methods

Materials and equipments

All materials used in this study were of cosmetic grade materials. PIT emulsifier of nonionic ethoxylated surfactant, plant emollient oil, glycerin, 1,2-hexanediol, sodium hyaluronate, beta-glucan, polygamma-glutamate, adenosine, niacinamide, were applied by Biobeautech Co., Ltd (Korea) and used without further purification. Droplet size was measured by Zeta sizer Nano ZS90 form Malvern Co., Ltd. The pH meter was used by BK technology Co., Ltd.

Preparation of PIT complex containing plant oils

PIT complex was prepared by mixing at above their melting points followed by cooling to room temperature. Mixtures of oil such as camellia japonica seed oil, macadamia integrifolia seed oil, limnanthes alba (meadowfoam) seed oil, argania spinosa kernel oil, nicinamide, adenosine, water and surfactant were prepared at their PIT technology. Also, PIT complex applied to high technology in order to make a fine emulsion using hydrophilic-lipophilic balance (HLB) temperature. PIT fine emulsions were formed at special zone 50~75°C which used the affinities of surfactant for water and oil were balanced. Then, the solutions are quickly heated or cooled in order to obtain water-in-oil (W/O) or oil-in-water (O/W) emulsion respectively

Evaluating method of moisturizing activity

Volunteers, washed the inside both forearms by 70% ethanol, were acclimated for 15 minutes in a thermostatic chamber (room temperature 25˚C, relative humidity 45%). After measure moisture content (before use) of measurement regions, testing samples treated with inside forearms each 2.0mg/cm2, after 30 minutes, 1, 2, 3, 4, 5, 6 hours, measured it 3 times using by Aramo TS (Aram Huvis Co. Ltd., Korea). These results are skin surface moisture content of this region. Change of moisture content showed relative value that measures initial value applied 100% ethanol [8-11].

Evaluating method of trans epidermal water loss (TEWL)

The Tewameter TM 300 Evaporimeter (CK Electronic Co., Ltd., Germany) was used to take TEWL measurements. The hand was placed on a flat table and Evaporimeter probe placed horizontally on the skin. Two sensors in the probe head sample relative humidity above the skin surface, allowing the rate of water loss to be calculated from the measured humidity gradient (g/m2/hr). TEWL measurements increase as the rate of water loss from the skin increase, indicating a compromise in the barrier. A decrease in TEWL measurements indicates an improvement in the skin's ability to retain moisture, or an improvement in barrier function. Before TEWL measurements, subjects equilibrated for approximately 15 minutes to ambient room temperature and humidity conditions. During the course of the study, the room was maintained at a temperature of 22˚C and relative humidity between 45% [12-15].

Human volunteers clinical test (in-vivo): Whitening & anti-wrinkle effect

In-vivo tests on the moisturizing activity were performed with 10 male and female volunteers in both short and long term tests. Control formulations were prepared as conventional O/W emulsions without liquid crystalline formation [16-19].

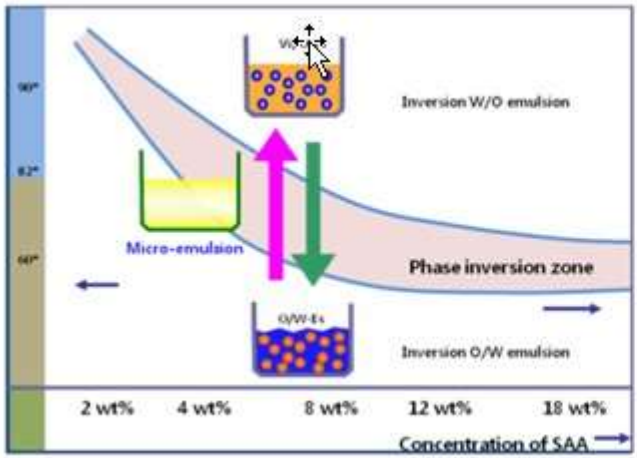
Results and Discussions

The Principle of PIT Emulsion System

Phase inversion temperature (PIT) emulsion system could be obtained fine emulsions without special homo mixer, simply only using agitation. The PIT emulsifying system formed fine emulsion due to the two phase changes from w/o emulsion to o/w emulsion

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

between botanical oil phase and water interface, depending on the increasing temperature. When a w/o emulsion is cooled, a transitional phases inversion occurs that results low viscosity, finely-dispersed fine emulsions having good long storage stability. This emulsion system was formed as a w/o type because of hydrophobic activity at high temperature above 85~95°C (see Fig. 1).



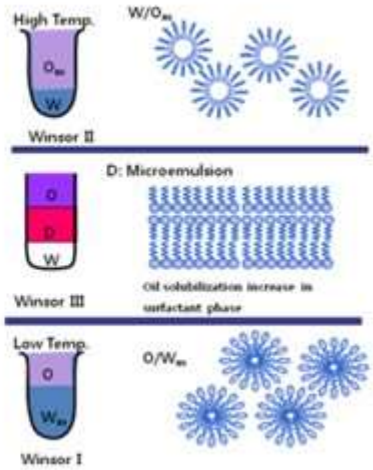


Figure 1. Mechanism of phase inversion zone depend on the temperature with (POE)12~20 fatty acid ethers of nonionic surfactants.
Figure 2. Phase prism, Winsor systems, and dispersion structure of an emulsion composed of nonionic surfactant, oil, and water depend on the various temperatures.

It was formed bi-continuous phase at 48~65°C, then, it was changed in o/w emulsion below 50°C. When it was bi-continuous lamellar phase put the cold water, final emulsion could get fine droplet size from 105nm. Specific non-ionic surfactant was changed to converse from w/o emulsion to o/w emulsion phase. Phase prism, Winsor types, and dispersion structure of an emulsion composed of nonionic surfactant, oil, and water (see Fig. 2).

Development of PIT emulsion with botanical oils

To prepare the PIT botanical complex, 4 kind emollient oils of the camellia japonica seed oil, macadamia integrifolia seed oil, limnanthes alba (meadowfoam) seed oil, argania spinosa kernel oil were developed (see Fig. 4). Then here, add the niacinamide (whitening) and adenosine (anti-wrinkle) to get high functional skin care activity, we developed the PIT botanical oil complex (see Fig. 5a, b).

Figure 3. 4 kinds of botanical plant oils to make an emollient effect: camellia japonica seed oil, macadamia integrifolia seed oil, limnanthes alba (meadowfoam) seed oil, argania spinosa kernel oil (pictures show from left side).

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

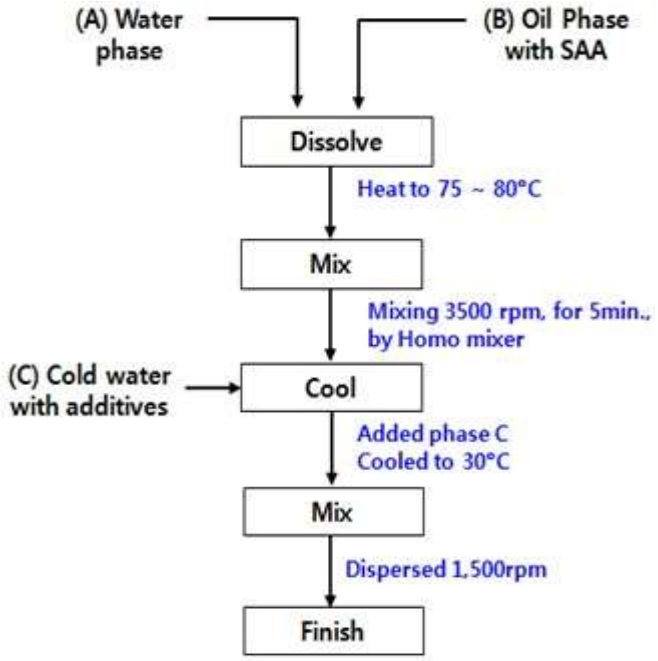


Figure 4. Preparing method of fine emulsion with botanical oil complex using PIT self assembly system without high pressure homogenizer.

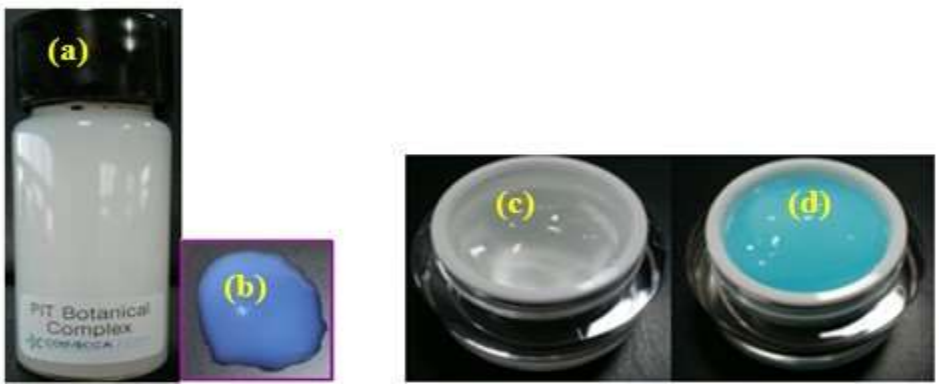


Figure 5. Pictures of PIT fine emulsion with botanical oil complex(a), appearance of bluish emulsion(b), placebo gel cream(c), and PIT hydrogel-cream (d) with PIT fine emulsion.

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

Appearance was bluish fine liquid emulsion as you showed Fig. 5. It was very fine emulsion to be absorbed into the skin epidermis. Feel texture was smooth and rich emollient and moisture feeling. Droplet size was 110±30nm (see Fig. 6). Placebo cream (c), PIT hydrogel-cream (d) showed at Fig 5c, d. pH was 6.0±0.5.

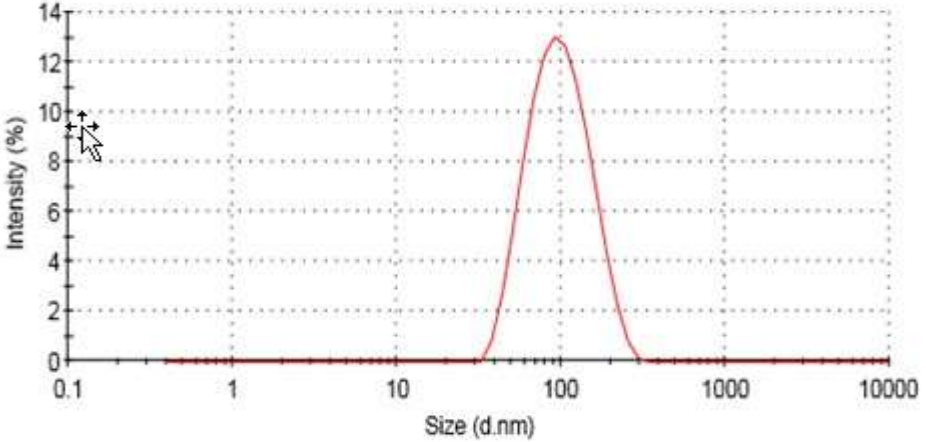


Figure 6. Droplet size of PIT plant emulsion with the camellia japonica seed oil, macadamia integrifolia seed oil, limnanthes alba (meadowfoam) seed oil, argania spinosa kernel oil; mean size 110 nm measured by Zetasizer.

Development of PIT hydro-gel cream

Multi-functional PIT high-gel cream was prescribed for the indicated in Table I. Heat A and B phase above 50~60°C, then put A and B and mix to 2, 500rpm for 5 minutes, after cool to 40°C after add C and D phase. Finally, put phase D and E each. Then mix to 1,500 rpm for 3minutes.

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

Table I. Formula of Hydro-gel Cream with Multi-functional Cosmetics: Compared placebo CRM with PIT Hydro Gel Cream (PIT-HG-CRM)

Phase	Ingredient Name	Placebo-CRM	PIT-HG-CRM	Functions
A	EDTA-2Na Chlorphenesin Water	0.02 0.2 To 100	0.02 0.2 To 100	Chelator Preservative Moisturizer
B	Carbomer (2% soln.) Ammonium Acryloyldimethyltaurate/VP Copolymer(2% soln.)	20.0 10.0	20.0 10.0	Gelling agent Gelling agent
C	PIT Complex (Camellia japonica seed oil, Macadamia integrifolia seed oil, Limnanthes alba (meadowfoam) seed oil, Argania spinosa kernel oil, Nonionic surfactant, Nicinamide, Adenosine)	-	20.0	Active ingredients
D	PEG-60 Hydrogenated Castor Oil Fragrance Alcohol Denat.	0.15 0.02 4.00	0.15 0.02 4.00	Surfactant Fragrance Astringent
E	Tromethamine Water	0.32 3.00	0.32 3.00	Ph Adjuster Conditioner
F	Phenoxyethanol Caprylyl Glycol/Ethylhexylglycerin	0.20 0.20	0.20 0.20	Preservative Conditioner

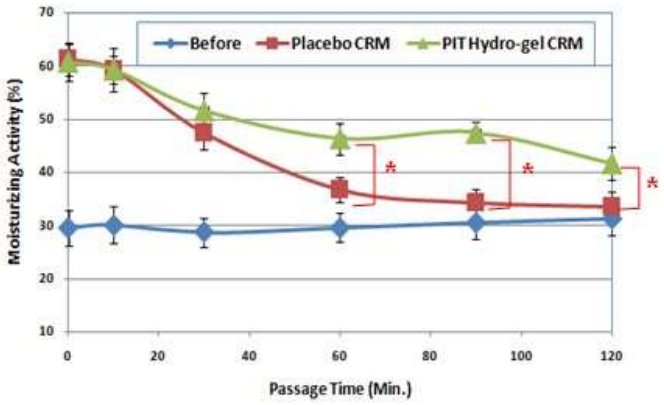
Moisturizing effect

The moisturizing activity of PIT hydro-gel cream (Table I) was evaluated and compared with conventional cream by measuring skin conductance of 7 volunteers. Fig. 7 shows the changes of moisture activity as a function of time. After 1 hour, the moisture activity of PIT hydro-gel cream was 45.5%. On the other hand, in the case of placebo cream, it was 34.3%. In addition, according to the student t-test, the moisturizing activity of PIT hydro-gel cream was confirmed to be significantly higher than that of placebo cream ($p<0.05$). Hence, PIT hydro-gel cream was considered to act much more effectively as a moisturizer than placebo cream.

Reducing Ability of Trans-epidermal Water Loss (TEWL)

The TEWL was measured when PIT hydro-gel cream (Table I) and conventional emulsion were separately applied on the skin (Fig. 8). After 4 and 6 weeks of application (twice a day), TEWL of PIT hydro-gel cream had a significant decrease in comparison to TEWL of placebo cream.

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT



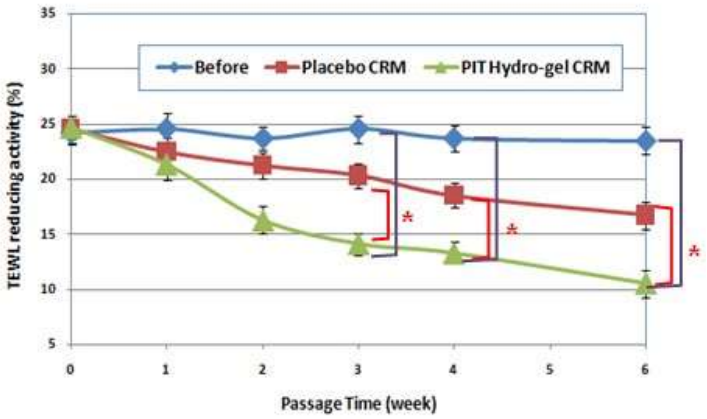


Figure 7. The changes of skin moisture as a function of time, male and female volunteers (n=7), student t-test (*p<0.05)

Figure 8. The changes of TEWL on the inner-side thighs, volunteers: n=7, *student t-test (*p<0.05)*

Skin Whitening and fine wrinkle diminishing activity (in-vivo)

Whitening Effect

Skin whitening activity of final formulation was evaluated and compared with before treatment by measuring volunteer skin with 3D-images with Aramo TS (Aram Huvis Co., Ltd, Korea). Fig. 9 showed the changes of skin clearing activity. As shown in Fig. 9 (placebo application, left), the skin clearance was no changed prior to the application. After 6 weeks of applying final formulation, the skin epidermis tended to be a change of skin clearance. It was diminished and removed pigmentation. However, there was no change on skin whitening improvement in four weeks following before application.

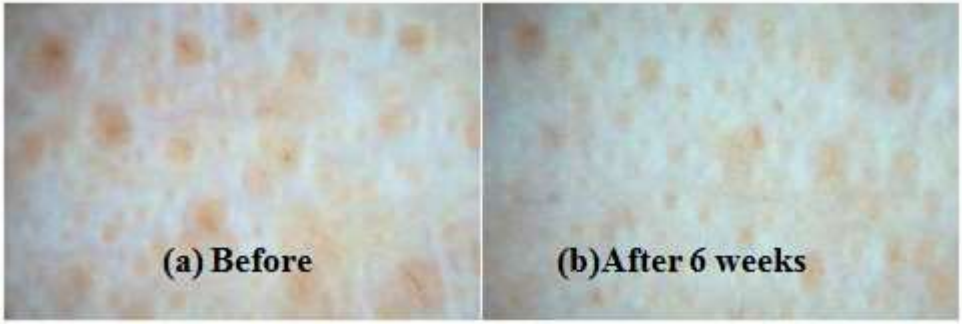


Figure 9. Skin whitening activity of PIT hydro-gel cream with niacinamide (active content: 2.0wt%) using human volunteers; (a) placebo application, (b) application after 6 weeks (n=6).

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

Figure 10. Fine wrinkle diminish evaluation of PIT hydro-gel cream with adenosine (active content: 0.04wt%) using human volunteers; (a) before application, (b) application after 6 weeks (n=6).

Anti-wrinkle effect

Fine wrinkle diminishing effect was carried out same samples with final formulations. Skin 3.6.1 conditions after application were observed by measuring volunteer skin with 3D-images with Aramo-TS. As shown in Fig. 10 (placebo formulation, left), the skin clearance was no changed prior to the application. After 6 weeks of applying final formulation, the skin epidermis tended to be a change of skin clearance. It was recognized to reduce fine wrinkles (Fig. 10, final formulation, right).

Conclusions

This study shows that total skin care of cosmetic formulations can be improved by focusing on the relationship between skincare and makeup having multi-functional efficacy. The skin efficacy of volunteers was improved such as moisturizing activity, TEWL, whitening, and wrinkle. The aim of this study is to get unique formulation of multi-functional activity for preparing PIT hydro-gel cream of Korean style. This PIT hydro-gel cream is to find an optimum way to enhance the strengthening effect on skin barrier functions of cosmetic formulations.

Acknowledgments

This study was performed by R&D fund from Korea small and medium business administration. (Project No. : S2140427).Development of PIT Hydro-Gel Cream

Multi-functional PIT high-gel cream was prescribed for the indicated in Table I. Heat A and B phase above 50~60□C, then put A and B and mix to 2, 500rpm for 5 minutes, after cool to 40□C after add C and D phase. Finally, put phase D and E each. Then mix to 1,500 rpm for 3minutes.As shown in Fig. 1, with addition of TiO_2 the densification of composites first increased and then decreased. Since, adding more TiO_2 has caused high volume expansion at spinel phases. According to reaction: (1) with the solution of titania in the periklase lattice, for of valence variation between Ti^{4+} and Mg^{2+}, vacancy cation will be created.. This vacancy causes high diffusion coefficient and sintering becomes easier.

References

1. K. Shinoda, The comparison between the PIT system and the HLB-value system to emulsifier selection, the 5th International Congress of Surface Activity, 2, 1969 (275-283).
2. K. Shinoda and H. Arai, The correlation between phase inversion temperature in emulsion and cloud point in solution of nonionic emulsifier, The Journal of Physical Chemistry, 68(2), 1964 (3485-3490).

INTERNATIONAL JOURNAL OF RESEARCH SCIENCE & MANAGEMENT

3. C. H. Kim, J. G. Park, J.W. Whang, J. R. Hu, Effect of direct emulsification method and invert emulsification method on droplet size of o/w emulsion, J. Soc. Cosmet. Scientists Korea, 20, 2011 (64-72).
4. J. Meyer, G. Polka, and R. Scheuermann, Preparing PIC emulsions with a very fine particle size, Cosmetics & Toiletries, 122 (1), 2077 (61-70)
5. J. J. Lee, K. Takayanagi, T. Shiga, Breeding for Improvement of Fatty Acid Composition in Rapeseed, Brassica napus L., Korean J. Breeding,6, No.2, 79(1974).
6. J. Alander, A. C.Andersson and C.Lindström, Cosmetic Emollients with High Stability against Photo-oxidation, Lipid Technology, 18(10), 226 (2006).
7. B. Y. So, The Correlation Between Phase Inversion Temperature (PIT) and Griffin's HLB-Value of Nonionic Surface-Active Agents, J. of Korean Oil Chemists' Soc., 6(2), 1(1989).
8. T. Folster, F. Schambil, H. Tesmann, Emulsification by the Phase Inversion Temperature Method: the Role of Self-bodying Agents and the Influence of Oil Polarity, International J of Cosmetic Science,12(5), 217 (1990).
9. E. J.Roh, B. K. Kim.D. S. Kim, Antioxidative Activity and Antiaging Effects of Tetrapanax Papyriferum Extract, J. of Korean Oil Chemists' Soc., 28(2), 219 (2011).
10. S.N. Park, H. J. Yang, J. H. Kim, W. K. Cho, The Stability of Emulsions Formed by Phase Inversion with Variation of HLB of Surfactant, J. of Korean Oil Chemists' Soc.,26(2), 117(2009).
11. I. Y. Kim, S. W. Jung, R.Y. Ryoo, C. K. Zhoh, Anti-aging Effects of the Extracts from Leaf. Stem, Fruit and Seed of Yew (Taxuscuspidata Sieb) by Solvent Extraction Method, J. Soc. Cosmet. Scientists Korea, 32(4),211 (2004).
12. I.Y. Kim, C. K. Zhoh, H. C. Ryu, Liquid Crystalline Technology of Cosmetic Industry and Moisturizing Effect of Skin, J. Soc., Cosmet. Scientist Korea, 30(2), 279 (2004).
13. I.Y. Kim, C. K. Zhoh, H. C. Ryu,Formation of Provitamin-B5Liquid Crystal with Hydrogenated Lecithin and Its Effectiveness of Moisturizing Activity, J. of Korean Oil Chemists' Soc., 20(2), 101 (2003).
14. S. Y. Kim, M. H. Lee, N. R. Jo, S. N. Park, Antibaterial Activity and Skin Moisturizing Effect of Cedrela Sinensis A. JussShoots Extracts, J. Soc., Cosmet. Scientist Korea, 36(4), 315 (2010).
15. J. C. Yang, The Evaluation on the Effectiveness as a Cosmetic Material of Oil Extracted from Schizandra Chinensis Seed, J. of Korean Oil Chemists' Soc., 29(2), 232 (2012).
16. D. R. Lee, W. G. Cho, Stability of Nano-Emulsions Prepared by Solubilization Method, J. Soc. Cosmet. Scientists Korea, 36(4), 265 (2010).
17. K. Y. Kyong, C. G. Lee, Development and Prospect of Emulsion Technology in Cosmetics, J. Soc. Cosmet. Scientists Korea, 32(4), 209 (2006).
18. C. K. Zhoh, I. Y. Kim, H. S. Lee, Nano Capsulization of Ceramide and the Efficacy of Atopy Skin, J. Soc. Cosmet. Scientists Korea, 30(3), 419 (2004).
19. M. C. Kim, C. S. Moon, H. K. Park, Emulsion Stability of Water/Oil Emulsified Fuel by associated with Emulsifiers, J. of Korean Oil Chemists' Soc., 25(3), 395 (2008).