Study of complete transparent nano-emulsions which contain oils

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Abstract

Recently inside nano liposome particles or nano-emulsions which contain tough-melting physiology activity materials or the coefficient of low organism utilization promote the coefficient of organism utilization, so this part has been studied a lot because they can absorb selectly cosmetics, specially physiology activity materials, into the skin. Also, in particle size, cells interstitial lipid interval are 30~50nm, so nano-emulsions that the size is similar to 30~50 nm are made to study for absorbing quickly into the skin. And transparent skin which contains oils in common skin lotion dosage form has become the center of public interest. The used nano-emulsions in this study were unsaturated lecithin/co-surfactant/ ethanol/ oil / water. And polysorbate 20/ polysorbate 80/ Dicetyl phosphate/ hydrogenated caster oil/ isoceteth-20/SLS were used in co-surfactant. The used oils were cyclomethicone and caprylic/capric triglyceride. The manufacturing process was that microfluidizer was fixed in 1000bar and transit times were changed from 1 to 10 times. From transparency and particle size, the transparency sequence was SLS> polysorbate 20= polysorbate 80> isoceteth-20> dicetyl phosphate >hydrogenated caster oil and the particle size was small. Specially cyclomethicone nano-emulsions, when we made unsaturated lecithin /SLS /ethanol/water/ cyclomethicone, cyclomethicone 5% was good for transparency. And 20% of this was used for making transparent skin toner in common skin dosage form.

Key words: nanoemulsion

1.Introduction

Liposomes make the double layer occlusive membranes which are composed of the phospholipid like phosphatidyl choline(PC), phosphatidyl ethanolamine(PE), phosphatidyl serine(PS), sphingomyelins, cardiolipids, plasmalogens, phosphatidic acid and cerebroside. They also are

moleculer groups which are equilibrium state with water. The formative ingredients for lipid have the amphiphilic property themselves because they contain a hydrophilic region and lipophilic region. Therefore when liposome is dispersed in water, that forms bilayer not sphere and surround an water soluble active ingredient while nanoemulsion includes an oil soluble active ingredient. Nanoemulsion has the different physical and chemical properties against the general emulsion like stability, inherited character, homogeneity, high surface area and so forth. Nanoemulsions consist in very tiny emulsion, having smaller diameter than 100nm. Unlike microemulsions, nanoemulsions are metastable systems, whose structure depends on the process used to prepare them. They can be prepared either by PIT method or spontaneous emulsification or with a high shear device.

Because the total interface excess free energy of a system, that one phase such as emulsion, foam, liphophilic sol is dispersed in continuous phase, is higher than thermodynamic equilibrium energy, this kind of colloid can not be spontaneously dispersed and this system is thermodynamic unstable. Therefore the dynamic approach is needed instead of the thermodynamic one. Nanoemulsions have smaller liquid droplets compared to general emulsion having larger ones. So nanoemulsion have a lot of advantages; 1)low creaming and sedimentation, 2)low fluctuation of surfactant being in interface and 3)low fluctuation and coalescence due to higher steric repulsion between liquid droplets. The formation problem of small droplet is shown on high Laplace pressure condition that makes the change to small droplet. This needs the application of high energy(high pressure homogenisers) and the high concentration of surfactants. The size of this system is only 100-200nm, but the small size of emulsion particle will give not only cohesion and adhsion but also inherent stability in creaming and precipitation. As particles become smaller, they have the Brown diffusion. And gravitation in some stability will be smaller than the massive gravity. For the most systems being stable state through the high molecular weight surfactant(not ionic), the energy distance curve will show very superficial minimum value. As particles become smaller, the attractive force of van der waals will be smaller. If the ratio of thickness of absorbed layer to the particle radius including nanoemulsions is large, the repulsion force of a system will be as strong as what kind of cohesion can be protected. These small particles do not change well unlike massive emulsions(big particles), so cohesion on the surface will be minimized and what kind of adhesion can be protected. One of the main advantages of nanoemulsion is that the transmission efficiency of active ingredients is good. The small particles with a large surface area can be effectively delivered to skin. Also, these small particles will cover on skin surface fairly, and will penetrate through rough tissues of skin surface. Generally, there are two processes to make nanoemulsion. The first process is using the high pressure- homogenizer. The Formation of nanoemulsion is achieved by pertinent surfactants and working with operation materials. The second process is the application PIT (phase inversion temperature) law. As it closes to PIT, the interfacial tension can be very low value(around 10-4mN/m) and as the emulsification is made near PIT, nanoemulsion will be formed with a rapid

cooling. It's generally hard to make very small particles, so it needs a lot of energies and surfactants. After making pre-emulsion of Oil in Water using usual method, nanoemulsion was made through high-pressure emulsification. The liquid droplet size from general emulsification is distributed on the average ten-several tens um and depends on rate, time, temperature of emulsification. Generally, when the smaller particle size is made with general emulsification , as time pass the phenomena such as creaming and sedimentation are showed and long-time stability is not good. However nanoemulsion through high-pressure emulsification is not controlled by instability factors which describes above. The range of emulsion particle size from general emulsification is one that is under the influence of gravity, and the stability of emulsion is under the control of stokes Law. However nanoemulsion with high-pressure emulsification has 100-200nm particle size, so the effect of gravity can be negligible. In this study the used oils are cyclomethicone and caprylic/ capric triglyceride and main surfactants are saturated lecithin and unsaturated lecithin. Also nanoemulsion was made with polysorbate 20/ polysorbate 80/ dicetyl phosphate/hydrogenated caster oil/ isoceteth-20/sodium lauryl sulfate(SLS) as co-surfactant. The formula is lecithin/ co-surfactant/ ethanol/ oil/ water and the manufacturing process was that microfluidizer was fixed at 1000bar and transit times were changed from 3 to 10 times. The particle size was measured with particle size analyzer at each time.

2. Experiment

2.1 Materials for experiment

The used lecithins are "Lipoid company's LIPOID S75 (unsaturated lecithin) and LIPOID S75-3(saturated lecithin)". Purified water which has passed through anion-cation exchange resin column is used.

2.2 Instruments for experiment

T.K. Auto Homomixer made by Tokushukika kogyo in Japan is used for manufacturing microemulsion, and in the evaluation of emulsion stability, charge in particle distribution is measured by using laser light scattering (Malvern, Model PCS 4700, UK) which is an instrument for measuring particle distribution. Nanoemulsion phenomenon is reviewed by photographing Freeze-Fracture Scanning Electron Microscopy. Microfluidizer (Microfluidics crop, Newton, MA, USA) was used to make nanoemulsion.

2.3. Method of experiment

Unsaturated lecithin (saturated lecithin) 5%(2%), co-surfactant 1%, ethanol 20%, oils 5% and the rest with water were mixed with homomixer, and then a series of three transit was made by

microfluidizer. Also cyclomethicone and caprylic/ capric triglyceride as oils was used and the number of transit through microfluidizer was changed from 3 to 10 times. After manufacture stability and particle size were checked on 0, 25and 45 degree.

3. Result and Discussion

The number of transit through the microfluidizer was changed 1 to 10 times to get the optimum condition. The result is shown in figure 1. When the samples are passed through the microfluidizer 3 times, the particle size are in optimum condition. From 4 times, the size is regularly maintained. In 8-10 times, the radius is slightly increased. When saturated and unsaturated lecithin were used, unsaturated lecithin was good on transparency and particle size. (figure 2) Unsaturated lecithin is good for emulsification because of double bonds. Unsaturated lecithin 5%, co-surfactant 1%, ethanol 20%, oils 5% and the rest with water were mixed with homomixer. The pressure for the microfluidizer is fixed at 1000bar and the number of transit is 3 times. The final product was kept at 25 degree for 1 month to measure the particle size. (figure 3) In case of caprylic/ capric triglyceride as an oil, the transparency sequence was SLS> hydrogenated caster oil> dicetyl phosphate> polysorbate 20= polysorbate 80> isoceteth-20, and the particle size was small. For cyclomethicon, transparency sequence was SLS> polysorbate 20= polysorbate 80> isoceteth-20> dicetyl phosphate >hydrogenated caster oil, and the particle size was small too. Each nanoemulsion is shown in figure 4. Transparency of SLS was good.(we could see the letter of magazine on the back side) The particle size distribution of nanoemulsion from polysorbate 20= polysorbate 80 and Freeze-Fracture Scanning Electron Microscopy are shown in figure 5 and 6, respectively. From these results, we can know that the particle size is 30-60nm and nanoemulsion is formed. The relationship of temperature and particle size shown in figure 7. At 45 degree, as temperature increase, transparency became good and particle size was small. Needless to say transparency became bad, and particle size was large at 0 degree. But transparency and particle size were not changed in SLS. The transparent nanoemulsion was manufactured using fllowing formula; Unsaturated lecithin 5%/SLS 1%/ ethanol 20%/Water/cyclomethicon 10%.The transparent skin toner and gel cream were made with 10-20% of this cyclomethicon nanoemulsion. Especially, it is well known that the viscosity should be increased to get high stability of nanoemulsion.

4. Conclusions

Nanoemulsion was made with unsaturated lecithin(saturated lecithin) 5%(2%), co-surfactant 1%, ethanol 20%, oils 5% and rest with water in this study.

1)In case of caprylic/ capric triglyceride as an oil, the transparency sequence was SLS>

hydrogenated caster oil> dicetyl phosphate> polysorbate 20= polysorbate 80> isoceteth-20, and the particle size was small. For cyclomethicon, transparency sequence was SLS> polysorbate 20= polysorbate 80> isoceteth-20> dicetyl phosphate >hydrogenated caster oil, and the particle size was small too.

2)At 45 degree, as temperature increase, transparency became good and particle size was small. Needless to say transparency became bad, and particle size was large at 0 degree.

3) The transparent nanoemulsion was manufactured using fllowing formula; Unsaturated lecithin 5%/SLS 1%/ ethanol 20%/Water/cyclomethicon 10%.The transparent skin toner and gel cream were made with 10-20% of this cyclomethicon nanoemulsion.

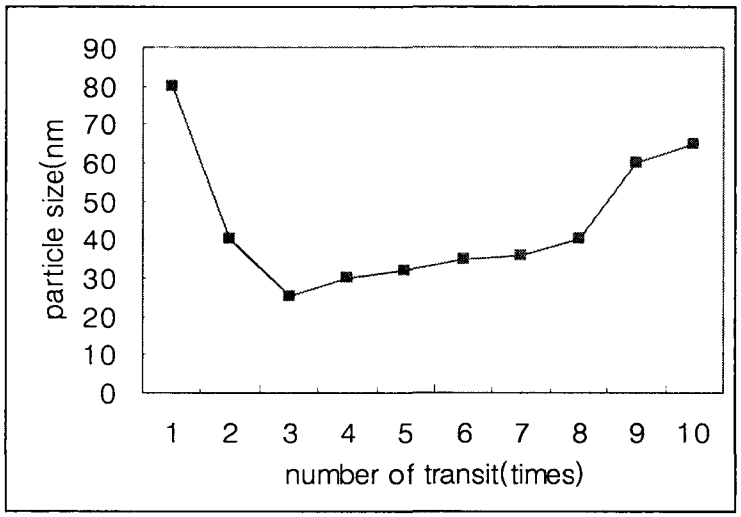


Fig.1. The particle size VS number of transit

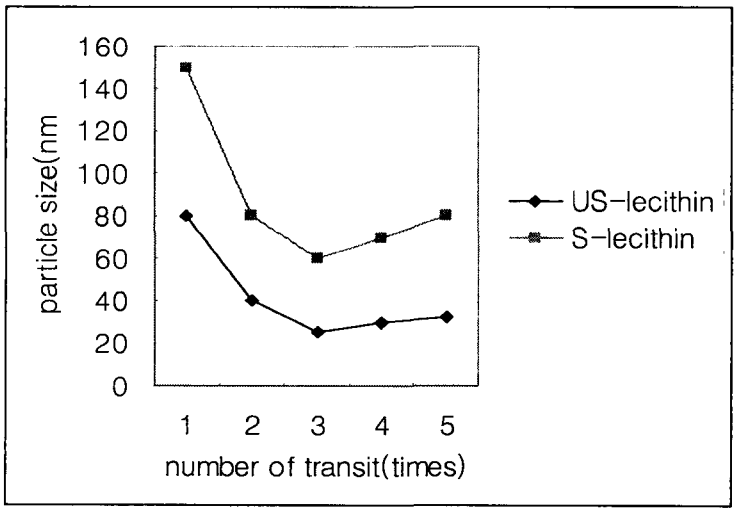
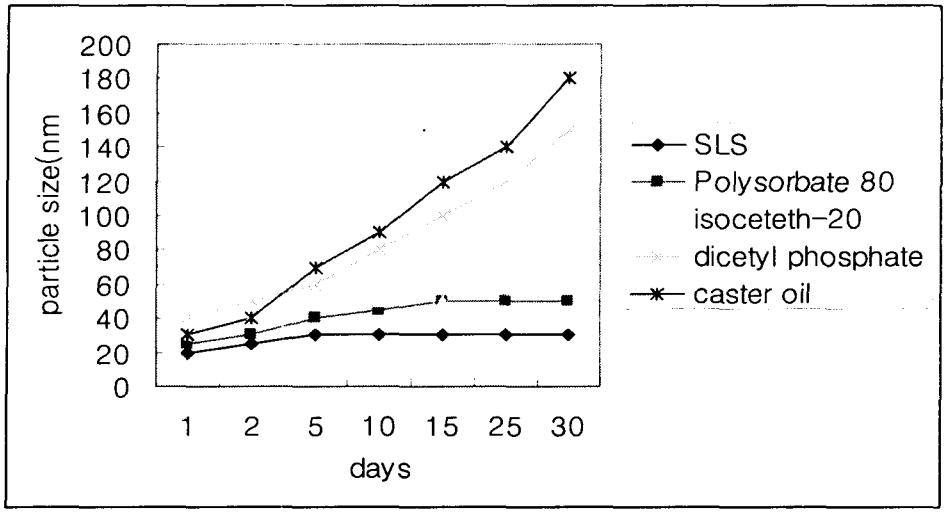
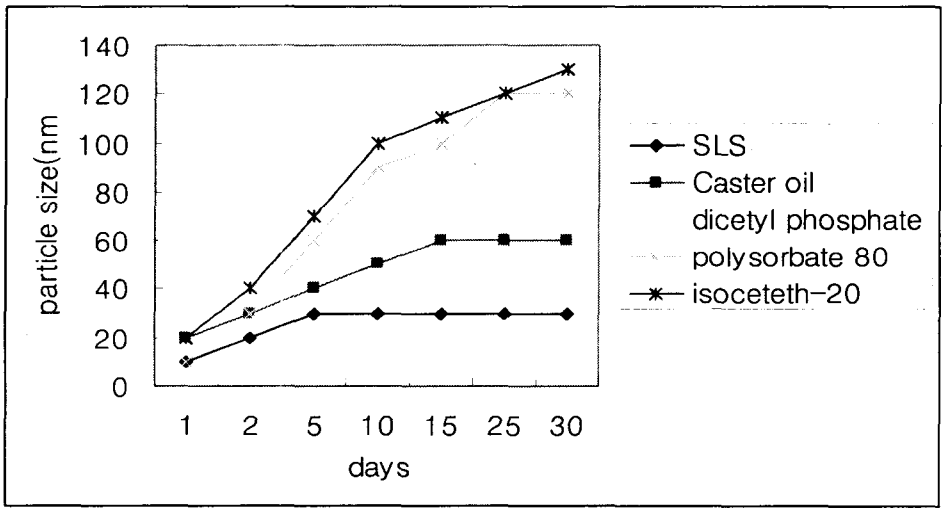


Fig.2. The particle size compared unsaturated with saturated Lecithin.



(a)cyclomethicone



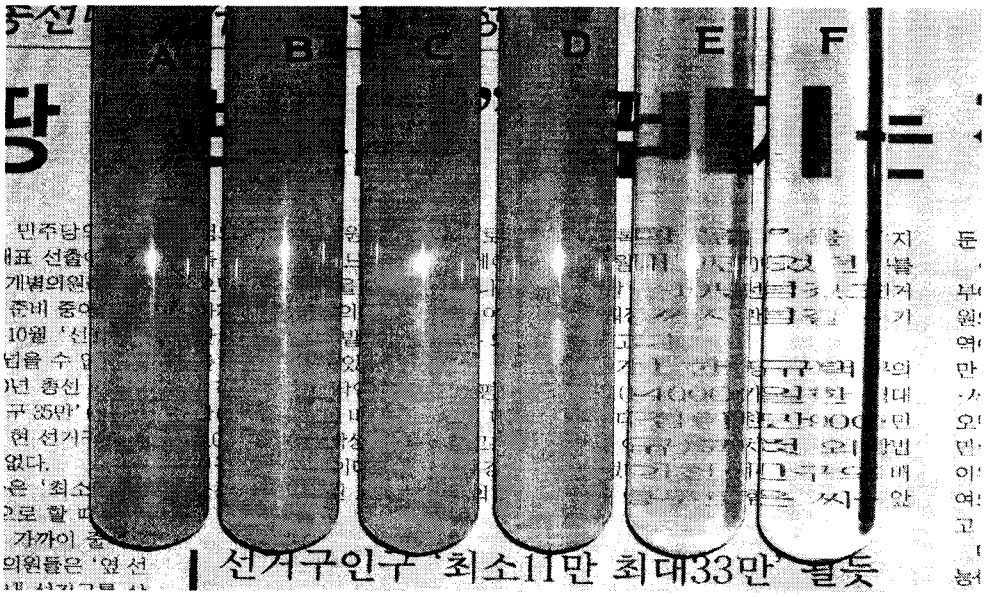
(b) caprylic/ capric triglyceride

Fig.4. The product was kept at 25 degree for 1 month to measure The particle size.

A:isoceteth-20 B:Polysorbate 80 C:polysorbate 20 D:dicetyl phosphate

E:hydrogenated caster oil F:SLS

(a)cyclomethicone



A:isoceteth-20 B:Polysorbate 80 C:polysorbate 20 D:dicetyl phosphate

E:hydrogenated caster oil F:SLS

(b) caprylic/ capric triglyceride

Fig.4. The transparency of nanoemulsion

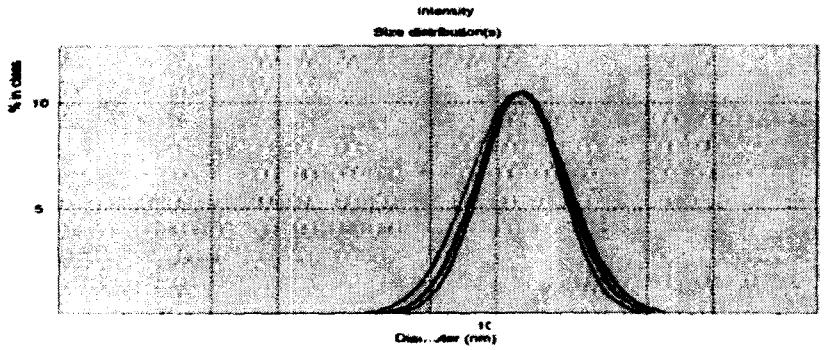


Figure.5. Particle size distribution of nanoemulsion.

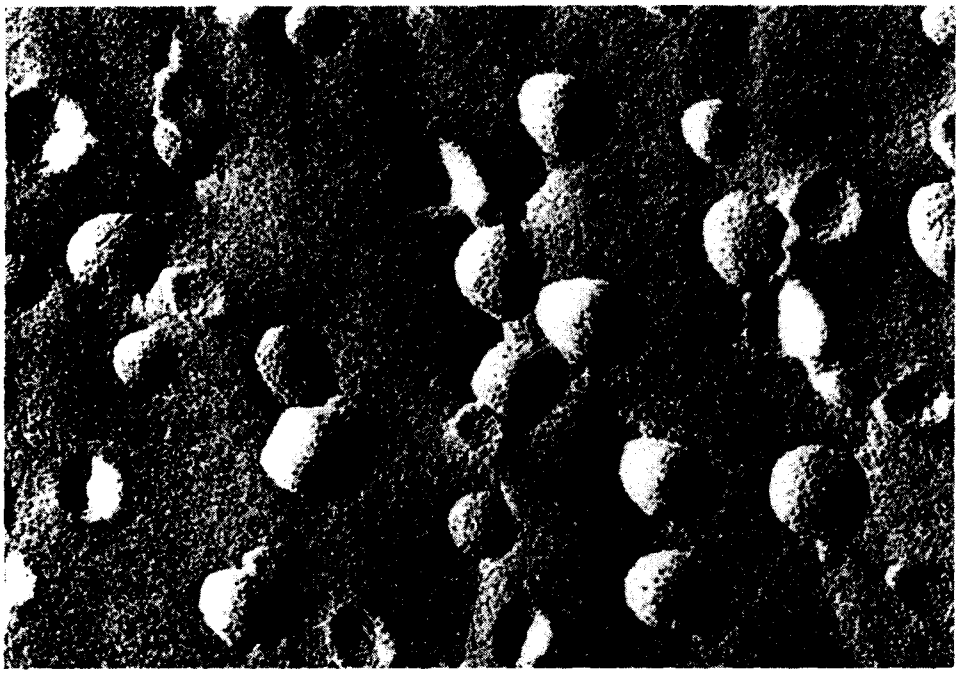
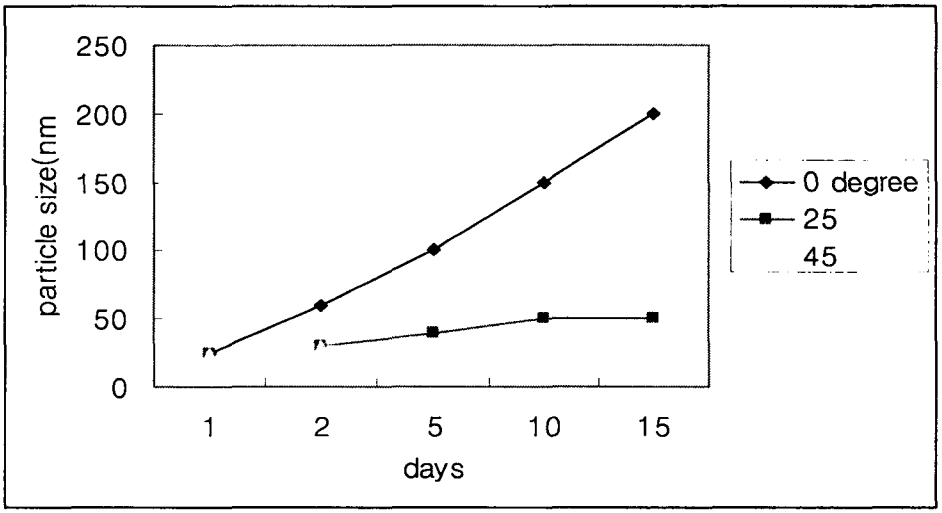
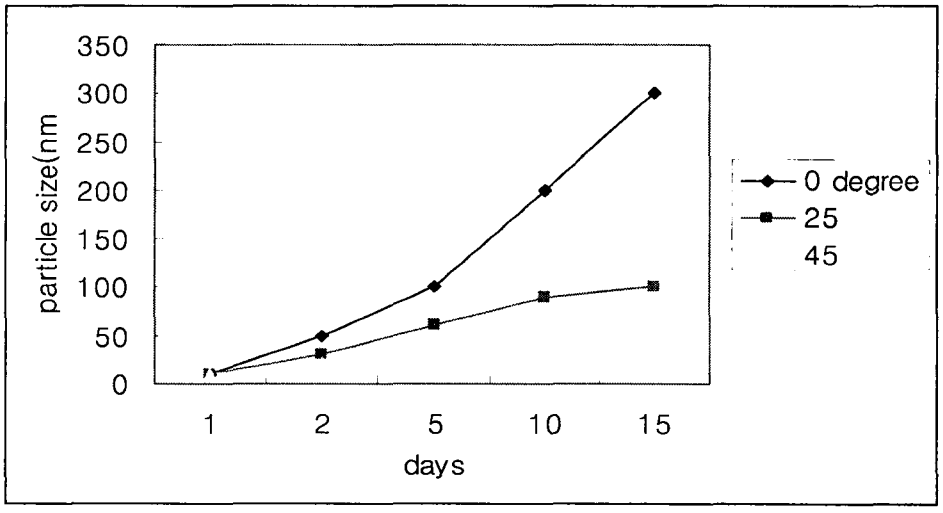


Fig 6. Freeze-Fracture Scanning Electron Microscopy picture of nanoemulsion.



(a) cyclomethicone



(b) caprylic/ capric triglyceride

Fig 8.The relationship of temperature and particle size.