

The Functions of Polyoxyethylene Tocopherylethers in the Formulations of Cosmetics

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화장품제형에서 폴리옥시에틸렌토크페릴에테르의 기능

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Abstract

The functions of polyoxyethylene tocopheryl ethers [POE(n)TEs] in emulsion, solubilization and gel were studied. For emulsification of liquid paraffin, POE(10)TE showed better emulsifying effect in O/W emulsions than others tested. The effects of oil and polyol content on the formulation of W/O and O/W emulsions were also studied. In O/W emulsion, the viscosity was increased by increasing the liquid paraffin content, at about 70%, and slightly increased by increasing the propylene glycol content. However, in W/O emulsion, the viscosity was decreased by increasing the oil content, and also decreased by increasing the propylene glycol content. For solubilization of perfume oil, POE(18)TE showed better solubilizing effect than the others tested. The gelling effect of POE(n)TEs increased with ethylene oxide chain length up to 50 moles. The gelling property was evaluated for hardness and viscosity.

1. Introduction

Surfactants are very important and indispensable in cosmetics. Among them,

nonionic surfactants are very important in skin care products. They may react with the stratum corneum, so their safety and physiology in skin care products are very important. One reason for the use of nonionic surfactants in skin care products is their well-documented safety, particularly their excellent skin and eye tolerance.(1)

Surfactants and their mixtures form amphiphilic association structures such as micelles, vesicles, emulsions and lyotropic liquid crystals when combined with each other and with water.(2,3) These association structures play decisive role not only in preparing cosmetic formulations but also in interacting with the stratum corneum lipids, which themselves are in the form of an amphiphilic association. The similar system of liquid crystal is relevant to the structure of the stratum corneum lipids on the skin.(4) This has been known as the key material for moisturizing the skin and is the essence of cosmetic science. In this paper the effect of polyoxyethylene tocopheryl ethers [POE(n)TEs] in the preparations of emulsion, solubilization and gelation was studied. Also this paper will discuss the effect of oil and polyol in cosmetic formulations.

2. Experimental

The POE(n)TEs (Toconics^R, Trade name of Pacific Corporation, Korea) with average number of ethylene oxide units of 5, 10, 12, 15, 18, 30, 50, 60 and commercial grade of nonionic surfactants were used in this study. All of these surfactants have the poisson distribution of ethylene oxide chain lengths, and the average degree of polymerization was determined from the increase in weight of the reaction mixture.(5)

dl- α Tocopherol was obtained from Roche. D-limonene, α -hexyl cinnamic aldehyde, lilyal and musk ketone were obtained from Givaudan. All chemicals were commercially available and used without further purification. FTS-40 system (Bio-Rad Laboratories, Inc., U.S.A) was used for Fourier-transformed IR spectroscopy. A Brookfield LVT viscometer (Brookfield, Synchro-Lectric, Model LVT) was employed in all viscosity measurements.

Cloud point

1% of nonionic surfactant solution was prepared for cloud point determination. In the case of POE(40) stearate (Myrj 52, ICI) and POE(100) stearate (Myrj 59, ICI), the

cloud point was determined from the temperature at which the solution changed from clear to turbid, by heating a solution containing 1 mole of sodium chloride in a test tube to lower the cloud point below the boiling temperature.(6) To examine the effect of SLS and CTAB addition, 1% of SLS and CTAB solution was also prepared respectively. To determine the stability at solution state, cloud point was measured after aging for 10 days, 20 days and 30 days, respectively, at 45°C.

Emulsification test

In this study very basic formulations were used to evaluate the properties of POE(n)TEs as emulsifier by comparing those of commercially available emulsifiers. The oil used for the experiments is a light mineral oil and deionized water was used for water phase. Liquid paraffin(L.P. #70, Witco, U.S.A) was chosen as main component of oil phase with dimethicone(Dow Corning 200 fluid, Dow Corning, U.S.A) and water with propylene glycol were the water phase.

(1) O/W Emulsion: The formulations used in this study are shown in Table I, III and V. After both oil phase with emulsifier and water phase were heated to 70°C, oil phase was added into water phase while homogenizing by T.K Homomixer (Tokushu Kikai, Japan) for 3 min, 5000 rpm. After emulsification the mixture was cooled down to 30°C by slow agitation.

(2) W/O Emulsion: The formulations used in this study are shown in Table II, IV and VI. After both oil and water phases were heated to 70°C, water phase was added into oil phase while homogenizing with T.K. Homomixer for 3 min, 6000 rpm. After emulsification the mixture was cooled down to 30°C by slow agitation, as the same way in O/W emulsion.

(3) Emulsion stability : In order to evaluate a emulsion stability within a short time, we choose a very simple method. We have used 25ml of emulsifier (1% in water) and 25ml of LP #70. The mixture of water phase and oil phase was severely shaken for 30 min, and stood in water bath. Preparations which emulsified for 30 min, they showed the phase separation after standing. After given hour, the remained emulsified phase was determined. All procedures were conducted at room temperature.

Solubilization test

Solubilization study of perfume oil in different micellar concentrations was carried out by using mixed perfume oil of α -hexyl cinnamic aldehyde, lilial and musk ketone (2:2:1, w/w) in surfactant solutions such as choleth-24 (Solulan C-24,

Amerchol), polysorbate 60 (Tween-60, ICI), POE (12) nonylphenyl ether (ENP-12, Nihon Emulsion) and POE(60)hydrogenated castor oil (Nikkol HCO-60, Nikko Chemical) and POE(18)TE, respectively. A vortex mixer was used in mixing the oil components and surfactant solution. The experiment was performed by adding a given amount of oil to given concentration of nonionic surfactant solution. The oil was first added to the surfactant solution and dispersed into tiny droplets by hand and was then mixed with surfactant solution by the vortex mixer at 120 cycles/min for 20 minutes. The turbidity of the solution was measured by transmittance(%) at 750nm. Transmittance (%) of the solution of surfactants as a function of concentration was measured by spectrophotometer.

Gelation test

Formulations for gelation test are shown in Table VII, VIII and IX. The nonionic surfactant and the oil soluble component were first added to the alcohol phase and the other hydrophilic component was added to water phase. Surfactant solution phase was heated to 80°C and water phase to 85°C. Surfactant solution was then added into water phase with moderate agitation. A clear transparent gel was formed on cooling. The viscosity was measured by Brookfield Viscometer (Brookfield, Synchro-Lectric, Model LVT) or gel hardness was measured by Rheometer (Fudo, Japan) at 30°C, 2 Kg, 2 cm/min and adaptor # 23.

Stability test of tocopherol in gels

Two kinds of transparent clear gels were obtained using POE(50)TE and Choleth-24 with tocopherol for stabilizing testing. The stability against oxidation was determined by UV spectrophotometer and thin layer chromatography. The UV absorption measurement was taken in the wavelength range 240-330nm. The main absorption peak for tocopherol is at 292 nm in ethanol. The UV absorption was taken at the maximum position as determined by the instrument. The color changes of the gels with POE(50)TE and with Choleth-24 were also observed after 3 months at room temperature. The gels of POE(50)TE and Choleth-24 were analysed by TLC for determining the oxidation stability of tocopherol. 2g of the gel after preparation was extracted with 4 ml of n-hexane, and the gel after aging for 3 months at room temperature was also extracted with same manner. The hexane layer was evaporated, and the residues were dissolved in 30 μ l of hexane. The samples were subjected to TLC using petroleum ether/ethyl ether (4:1, v/v) as a solvent. The spots on the plates were detected

under UV illumination at 254 nm. (7)

3. Results and Discussion

Cloud point

In Figure 1, the moles of ethylene oxide are plotted as a function of cloud point for POE(n)TEs. As the cloud point increases, the difference in nonpolar weight for members of the respective series decreases, until the curves eventually meet at a cloud point of about 95°C. The cloud point of POE(n)TEs is related to the number of POE units. The cloud point is rather insensitive to the concentration of the surfactant itself, but is appreciably influenced by the presence of certain additives. As seen in Figure 1, Anionic surfactant such as SLS depressed the cloud point. However cationic surfactants such as CTAB did not depress the cloud point as effectively as SLS. The cloud point results from a balance of the intermicellar van der Waals attractions and hydration repulsion, with the latter decreasing as a function of temperature. For longer EO groups the hydration repulsion is increased and so a higher temperature is required before attractions and repulsions are balanced. Increasing ethylene oxide chain length decreases the attractive force between micelles and results in increasing the cloud point.(8) The stability of nonionic surfactants in aqueous solution was measured by cloud point determination after aging for a time period at elevated temperature. The changes in cloud point of nonionic surfactants are shown in Figure 2. As shown in Figure 2, POE(40)stearate and POE(100)stearate having ester chains were susceptible to decomposition with an increase in the polyoxyethylene chain length, but POE(n)TEs did not change in cloud point. The cloud point of each POE(n)TEs did not decline with time. As shown in Figure 2, the changes in cloud point of aqueous solutions stored at 45°C was not found. This phenomenon is in agreement with the suggestion of Sato et al.(9)

Emulsification

In this study, liquid paraffin was mainly used as a oil phase. Liquid paraffin was chosen as the oil because it is one of the principal raw materials in the manufacture

of cosmetics. Emulsions of liquid paraffin oil in water have been studied by a viscosity determination. These emulsions were formed with POE(5)TE in W/O emulsion and POE(10)TE in O/W emulsion respectively.(10) The viscosities of emulsions immediately following homogenization were studied as a function of oil phase or polyol content. Keeping the total emulsifier contents constant, the ratios of the oil phase or polyol content in each phase were varied.

(1) O/W Emulsion: In Figure 3, the effect of oil content on the viscosity of O/W emulsion are shown. When oil phase content increases up to a critical value of about 70%, the viscosity increased suddenly. Moreover, quite stable emulsions which have higher volume fraction above 95% have been prepared. We think this is due to the formation of polyhedral particles. In figure 5, the effect of PG content on the viscosity of O/W emulsion was shown. As seen in the figure, the viscosity was slightly increased with PG content.

(2) W/O Emulsion: In figure 4, the effect of oil content on the viscosity of W/O emulsion. As shown in this figure, the viscosity was rapidly decreased by increasing the content of LP # 70. In figure 6, the effect of PG content on the viscosity of W/O emulsion. As shown in figure 6, the viscosity was decreased with the addition of PG.

(3) Emulsion stability: Emulsion stability was evaluated according to the presence or absence of visible separation, by placing the emulsion in a graduated cylinder and determining the degree of separation with time. In figure 7, the emulsion stability of various nonionic surfactants in O/W emulsion was shown. Emulsion stability was evaluated by measuring the stable portion to total portion. Among tested nonionic surfactants, POE(10)TE showed better emulsion stability than the other commercial emulsifiers. We assume this was due to a formation of liquid crystalline structure of POE(10)TE in emulsion system. This is important for stabilization of emulsion. The presence of liquid-crystalline structure at the oil-water interfaces has been shown to produce improvements in the stability of emulsion, although the exact mechanism of their action is still subject to some question.

Solubilization

The results of the solubilizing effect tested are shown in Figure 8 for mixed perfume oil. The solubility of each solubilize in surfactant solutions maintained a linear relationship with the molar surfactant concentration. As shown in Figure 8, in the case of POE(18)TE, polysorbate 60 and Nonoxynol-12, the optimum ratio of

solubilizer to solubilize was changed for each system but was generally in the range from 3 : 1. Among them, POE(18)TE showed better solubilizing ability than others in the range of 1 to 1.5% of solubilize content. Cholet-24 has poor solubilizing ability among the nonionic surfactant tested. In the case of POE(60) hydrogenated castor oil, it has moderate solubilizing ability. For solubilization of mixed perfume oil POE(18)TE showed better solubilizing effect than the others. We can assume that POE(18)TE has a large solubilizing capacity, that can be applied to various formulations. This effect may be due to, POE(18)TE has somewhat small CMC value or large micelle size and has a good configuration for solubilization when made into micelles.

Gelation

Clear gel is a mixture with one component of which is a fluid, homogeneous down to substantially colloidal dimensions, capable of resisting a finite shear force. This definition can be used to define a gel in terms of two important operational phenomena: First, a gel may contain a substantial portion of liquid component: Second, it is characterized by an extremely high viscosity and hardness.(11) Figure 9 shows the relationship between viscosity of the gel and EO moles in Table VII. As shown in Figure 9, POE(50)TE has excellent gelling property which can be considered to have good solubilizing effect for oil components. The relationships between viscosity and nonionic surfactant content were studied from Table VIII. In Figure 10, POE(50)TE, Cholet-24 and Oleth-20 formed clear gels with high viscosity, but polysorbate 60 did not. Especially POE(50)TE formed a gel with the smallest amount of surfactant. In Table IX, the formulations of sunscreen gel using nonionic surfactants are shown. The test results appeared in Figure 11. As shown in Figure 11, POE(50)TE, POE(30)TE and Cholet-24 showed good gelling property in a sun screen gel formula containing ethylhexyl-p-methoxycinnamate. We assume that surfactants with a flat and large hydrophobic group contained on its body such as chromanoyl group and its conjugates form a micelle with small aggregation numbers in the relative high concentration range, being stacked side by side, while the usual nonionic surfactants, such as polyoxyethylene alkyl or alkenyl esters, polyoxyethylene sorbitan derivatives form spherical or ellipsoidal micelles with large aggregation numbers.(12)

Stability of tocopherol in gels

Figure 12 shows the UV spectra of tocopherol in Cholet-24 and POE(50)TE base.

The upper spectrum of solid line was taken initially at the end of the mixing of the two phases. The lower spectrum of broken line was taken after the gel was aged for 3 months. The UV absorption decreases slowly with time. An observable change in the UV spectrum was found in the gel formed by Choleth-24 gel base after aging for 3 months. Whereas slight change can be seen in the tocopherol gel base even after being aged for 3 months. POE(50)TE gel has been aged for 3 months, slight changes in the UV absorption were found. The two absorption peaks in the lower spectrum of figure 10 are at the wavelengths of 294 nm and 272 nm respectively. The first peak is due to tocopherol while the next peak are probably due to its oxidation products. Tocopheryl quinones have been identified as the oxidation products and are known to absorb more intensely than tocopherol at shorter wavelength.(13) TLC results for POE(50)TE and Choleth-24 base gel were good agreed with spectrophotometric measurement.(14) It is interesting to see that the clear gels formed by POE(50)TE are more stable against oxidation in air than those formed by Choleth-24. The tocopherol structure contains a non-polar, 13-carbon straight chains and some branches and a polar part composed of two rings with oxygen attached. Since Choleth-24 has only 8 carbon chains which are not straight and 4 hydrocarbon rings, one may expect that the polar part and some non-polar parts of the tocopherol may extend outside the hydrocarbon core of the Choleth-24. On the contrary, in the POE(50)TE micelles the large part of the nonpolar chain of tocopherol can be solubilized in the hydrocarbon core and the rest of the non-polar part and the polar part of tocopherol can be solubilized in the EO mentle when the EO number is high enough. It is clear that POE(50)TE have advantages of both improved oxidation stability and gelling characteristics compared with common nonionic surfactants.(15) We believe that a stable gel can be also obtained by proper matching of the molecular structures between the solubilizer and the solubilizate.

4. Conclusion

For emulsification test of liquid paraffin, POE(10)TE showed better emulsifying effect in O/W system than others tested. In O/W emulsion, the viscosity was increased by increasing the liquid paraffin content, at about 70%, and slightly increased by increasing the the propylene glycol content. However, in W/O emulsion, the viscosity was decreased by increasing the oil content, and also decreased by increasing the the

propylene glycol content. For solubilization of perfume oil, POE(18)TE showed better solubilizing effect than the others tested. The gelling effect of POE(n)TEs increased with ethylene oxide chain length up to 50 moles. In gelling tests, it is possible to prepare tocopherol containing gels that have high resistance against oxidation in air by solubilizing the oily phase in the POE(50)TE surfactant solution.

요 약

유화, 가용화, 겔화 등의 화장품 제형에서 폴리옥시에틸렌 토크페릴에테르 [POE(n)TE]의 기능에 대해 연구하였다. Liquid paraffin의 유화에 있어서 POE(10)TE는 O/W 유화에서 우수한 유화능을 나타내었다. W/O 및 O/W 유화에서 oil 과 polyol 류의 함량 증가가 점도에 미치는 영향에 대해서도 연구하였다. O/W 유화에서 점도는 liquid paraffin 약 70% 함량에서 급격히 증가하기 시작하였으며, propylene glycol 첨가시는 약간의 증가만 나타내었다. 그러나 W/O 유화에서 점도는 liquid paraffin의 함량 증가에 따라 감소하였고, propylene glycol 함량 증가에 대해서도 감소하는 경향을 나타내었다. Perfume oil의 가용화에 있어서 POE(18)TE는 우수한 가용화능을 나타내었다. POE(n)TE의 gelling effect는 폴리옥시에틸렌의 사슬의 증가에 따라 50 mol까지는 상승하는 현상을 나타내었다.

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Table I. Formulations of O/W emulsion (wt %)

Ingredient	#1	#2	#3	#4	#5	#6	#7	#8
LP #70	20	30	40	50	60	70	80	90
Sil #200	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Toconics-10	3	3	3	3	3	3	3	3
PG	5	5	5	5	5	5	5	5
DIW	71.9	61.9	51.9	41.9	31.9	21.9	11.9	1.9

Table II. Formulations of W/O emulsion (wt %)

Ingredient	#1	#2	#3	#4	#5	#6	#7	#8
LP #70	10	20	30	40	50	60	70	80
Sil #200	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Toconics-5	3	3	3	3	3	3	3	3
PG	5	5	5	5	5	5	5	5
DIW	81.9	71.9	61.9	51.9	41.9	31.9	21.9	11.9

Table III. Formulations of O/W emulsion (wt %)

Ingredient	#1	#2	#3	#4	#5	#6	#7	#8	#9
LP #70	40	40	40	40	40	40	40	40	40
Sil #200	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Toconics-10	3	3	3	3	3	3	3	3	3
PG	10	15	20	25	30	35	40	45	50
DIW	46.9	41.9	36.9	31.9	26.9	21.9	16.9	11.9	6.9

Table IV. Formulations of W/O emulsion (wt %)

Ingredient	#1	#2	#3	#4	#5	#6	#7	#8	#9
LP #70	5	5	5	5	5	5	5	5	5
Sil #200	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Toconics-5	3	3	3	3	3	3	3	3	3
PG	10	20	30	40	50	60	70	80	90
DIW	81.9	71.9	61.9	51.9	41.9	31.9	21.9	11.9	1.9

Table V. Formulations of O/W emulsion (wt %)

Ingredient	#1	#2	#3	#4	#5	#6
LP #70	40	40	40	40	40	40
Sil #200	0.1	0.1	0.1	0.1	0.1	0.1
Toconics-10	3	10	20	30	40	50
PG	5	5	5	5	5	5
DIW	51.9	44.9	34.9	24.9	14.9	4.9

Table VI. Formulations of W/O emulsion (wt %)

Ingredient	#1	#2	#3	#4	#5	#6	#7
LP #70	5	5	5	5	5	5	5
Sil #200	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Toconics-5	3	5	10	20	30	40	50
PG	5	5	5	5	5	5	5
DIW	86.9	84.9	79.9	69.9	59.9	49.9	39.9

Table VII. Gel test of POE(n)TEs (wt %)

Ingredient	Content
Propylene glycol	5.0
Ethanol	5.0
POE(n)TE	20.0
dl- α Tocopherol	1.0
D. I. Water	69.0

Table VIII. Formation of Gelation (wt %)

Ingredient \ Formula	A	B	C	D	E	F	G	H
Propylene glycol	5	5	5	5	5	5	5	5
Ethanol	5	5	5	5	5	5	5	5
Nonionic surfactant*	5	10	15	20	25	30	35	40
dl- α Tocopherol	1	1	1	1	1	1	1	1
D. I. Water	84	79	74	69	64	59	54	49

Nonionic surfactant* : POE(50)TE, Cholet-24, Oleth-20, Polysorbate 60

Table IX. Formulation of Sunscreen Gel (wt %)

Ingredient	Content
Propylene glycol	5.0
Ethanol	5.0
Nonionic surfactant*	20.0
Ethylhexyl-p-methoxycinnamate	1.0
D. I. Water	69.0

Nonionic surfactant* : POE(50)TE, POE(30)TE, Cholet-24, Oleth-20, Polysorbate 60

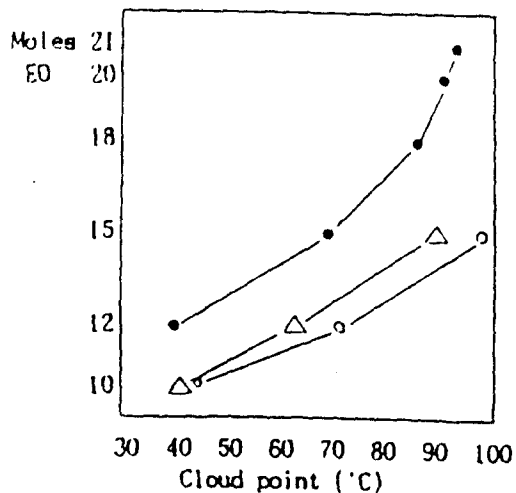


Figure 1. Cloud point of POE(n)TEs vs. mol of ethylene oxide [●:No additive △:CTAB(1%) 0.2ml added ○:SLS(1%) 0.2ml added]

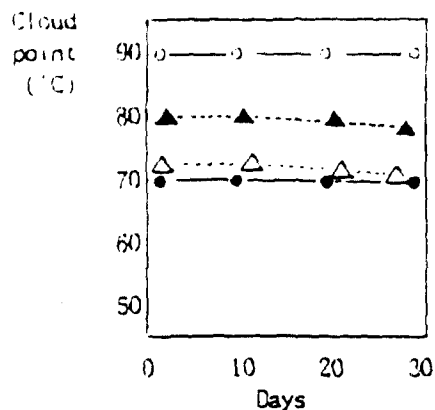


Figure 2. The changes of cloud point of 1% aqueous POE(15)TE, POE(18)TE, POE(40)stearate and POE(100)stearate, stored at 45°C. [Cloud points of POE(40)stearate and POE(100)stearate were measured after addition of 1M NaCl] [●:POE(15)TE ○:POE(18)TE △:POE(40)stearate (in 1M NaCl) ▲:POE(100)stearate (in 1M NaCl)]

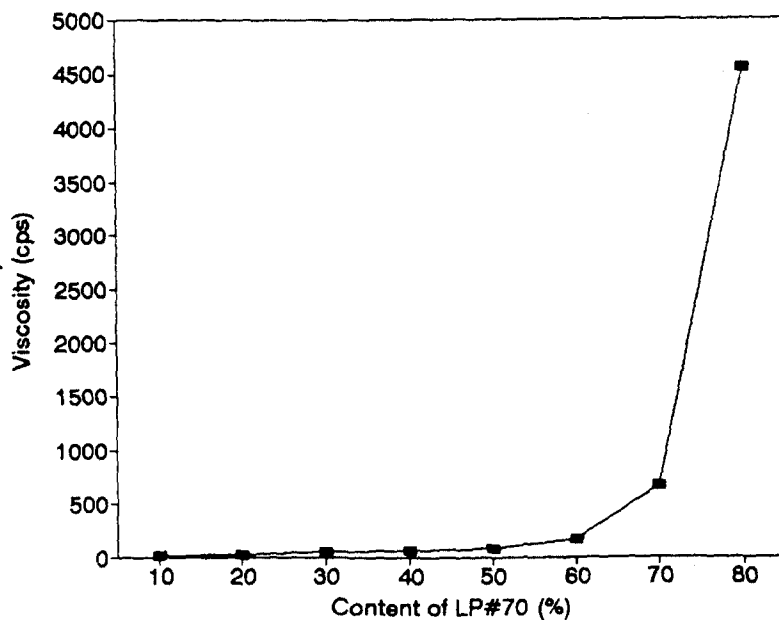


Figure 3. Effect of oil content in the viscosity of O/W emulsion

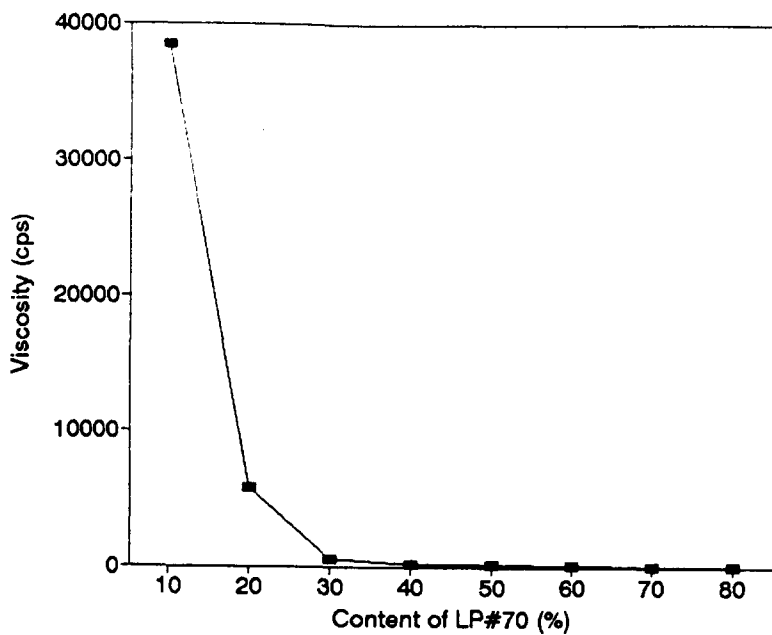


Figure 4. Effect of oil content in the viscosity of W/O emulsion

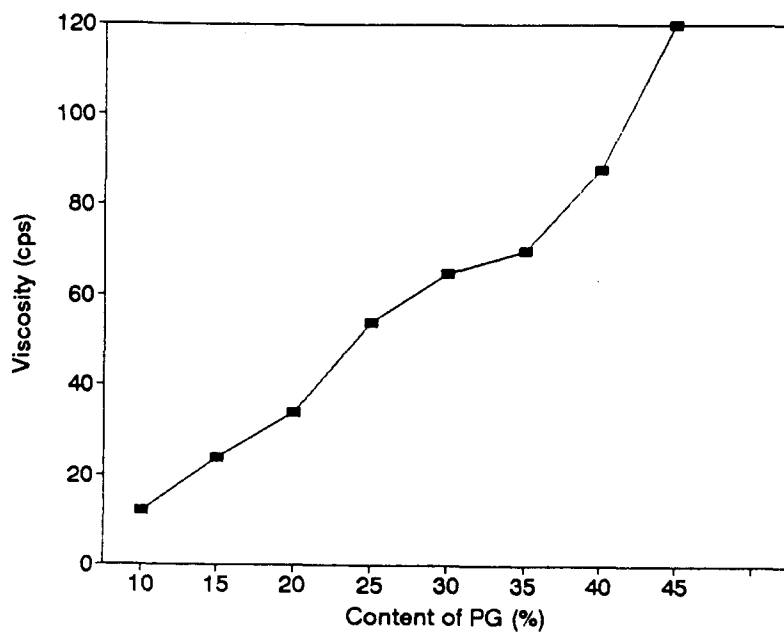


Figure 5. Effect of PG content in the viscosity of O/W emulsion

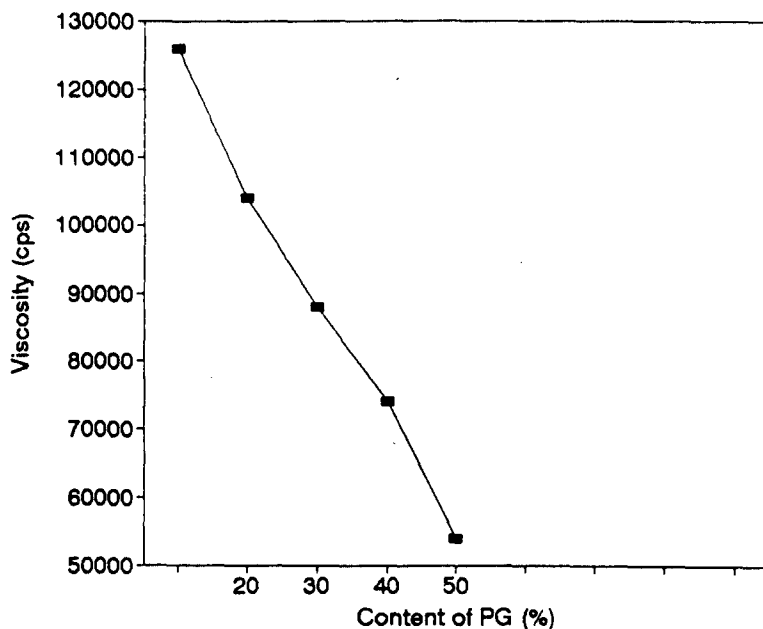


Figure 6. Effect of PG content in the viscosity of W/O emulsion

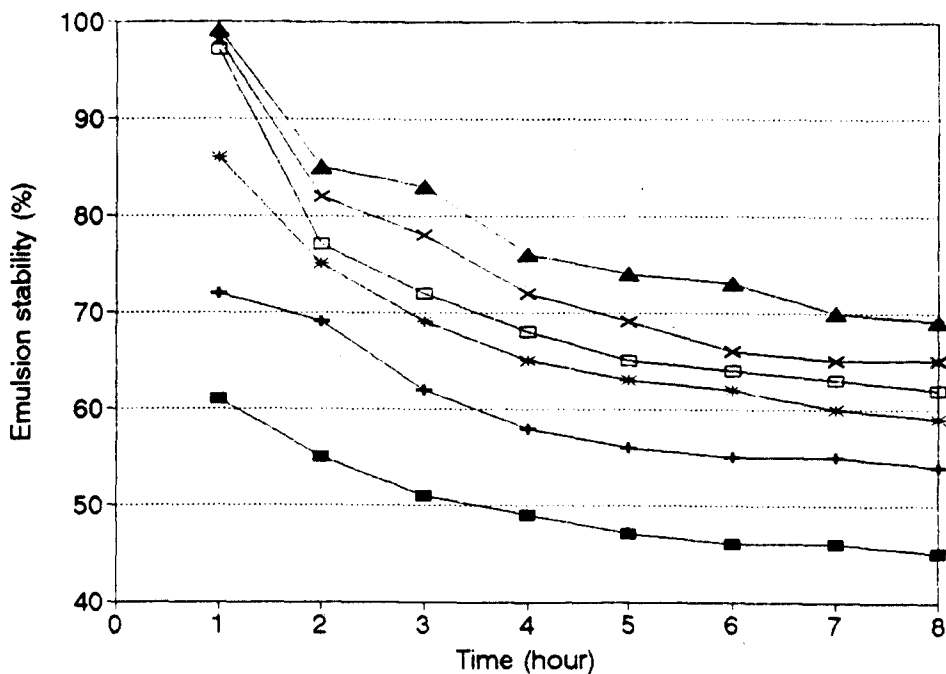


Figure 7. Relative emulsion stability of various nonionic surfactants in O/W surfactant system [▲-▲: POE(10)TE, ✕-✕: POE(12)TE, □-□: POE(5)Nonylphenol, ✕-✕: POE(6)Nonylphenol, + - +: POE(16)2-octyldodecylether, ■-■: POE(20)sorbitan trioleate]

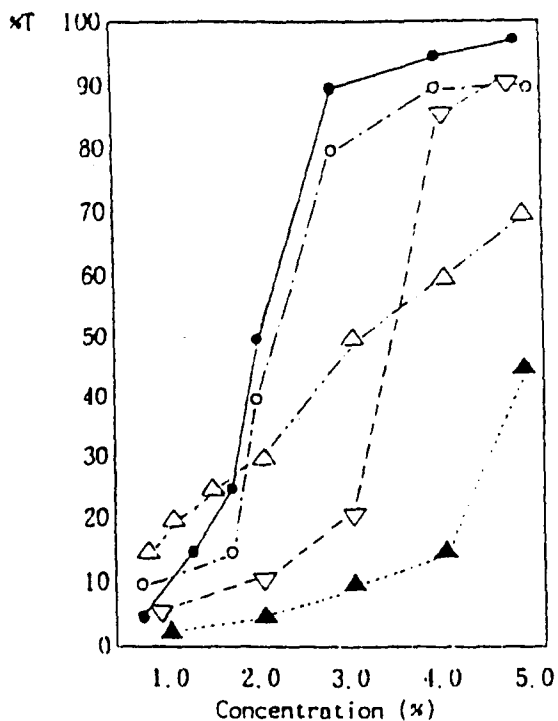


Figure 8. Plot of the transmittance(%) at 750nm vs surfactant concentration (mixed perfume content: 0.5%, ethanol content: 20%) [●: POE(18)TE ○: Polyorbate 60 △: POE(60)Hyd. CastorOil ▲: Choleth-24 ▽: Nonoxynol-12]

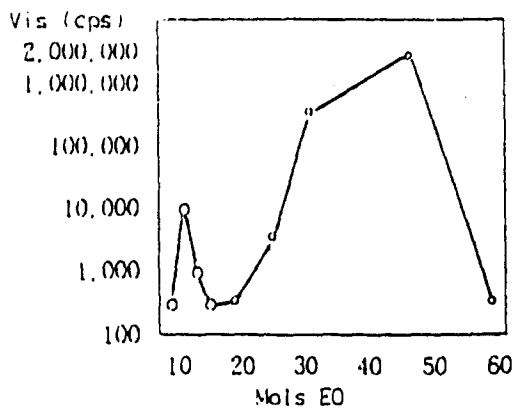


Figure 9. The correlation between viscosity and EO mol. of the gels formed with POE(n)TEs (20 wt %)

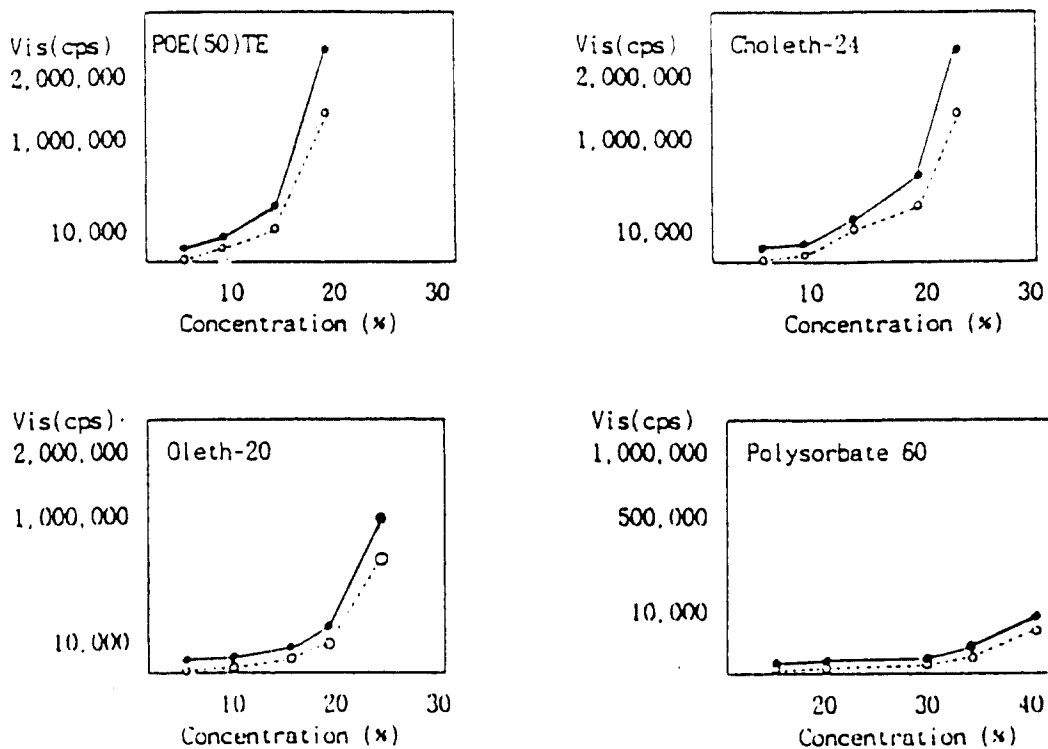


Figure 10. Relationship between viscosity and nonionic surfactant content. Solid line represents the viscosity of the gel with tocopherol (1%). Broken line represents the viscosity of the gel without tocopherol

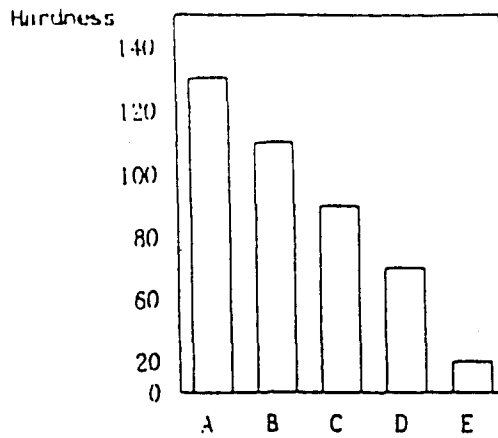


Figure 11. Hardness of the gel formed from POE(50)TE, POE(30)TE, Choleth-24, Oleth-24 and Polysorbate 60 (20 wt %), contained 1% of Ethylhexyl-p-methoxy cinnamate. [A: POE(50)TE B: POE(30)TE C: Choleth-24 D: Oleth-20 E: Polysorbate 60]

Figure 12. Spectrophotometric measurement of tocopherol oxidation in gels.

