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Effects of Diols on the foaming and emulsion properties in surfactant solutions

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요 약 : 본 연구에서는 계면활성제 용액에서 1,3-Butanediol, 1,2-Pentanediol, 1,2 Hexanediol 들이 cmc, 표면장력, 기포력 및 유화력에 미치는 영향을 연구 하였다. 계면활성제 수용액에 diol의 첨가는 cmc와 표면장력 저하를 초래 하였으며 기포력 및 유화력은 향상 되었다. 이러한 경향은 diol의 사슬길이가 길수록 현저 하였다. 이러한 특성은 diol의 알킬사슬과 계면활성제 분자간에 소수성 상호작용이 물분자간의 응집력을 감소시키고, 계면에서 계면활성제 headgroup간 상호작용을 증진시키기 때문이다. 또한 각 diol의 방부력을 평가하기 위하여 MIC 측정을 수행 하였으며 방부력은 1,2-HDO 〉 1,2-PDO 〉 1,3-BDO 순서였다. 본 연구의 결과들은 diol들이 화장품에서 보조 계면활성제와 방부제로 이용될 수 있음을 확인 시켜 주웠다.

주제어 :

Abstract : The effects of 1,3-Butanediol, 1,2-Pentanediol, and 1,2-Hexanediol in surfactant solutions on cmc, surface tension, foaming and emulsifying properties were determined. The addition of diols in aqueous surfactant solution decreased cmc and surface tension, and enhanced the foaming and emulsifying power. This trend is more significant by the longer hydrocarbon chain length of the diols. This property was confirmed because the diol's alkyl chain and the hydrophobic interaction with the surfactant reduce the cohesive force of water and increase the interaction between the head groups of the surfactant at interface. In addition, MIC test was conducted to determine the preservative power of each diol, and as a result, the antibacterial activity was effective in the order of 1,2-HDO 〉 1,2-PDO 〉 1,3-BDO. The results of this study show that diol can be applied to cosmetics as an auxiliary surfactant and antibacterial agent.

Keywords : diols; surface tension; foaming ability; emulsion stability; antibacterial activity

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1. Introduction

Diols are chemical compounds containing two hydroxyl groups that have a wide applications such as preservatives, industrial polyesters and fuels [1]. Among them, 1,3-Butanediol, 1,2-Pentanediol and 1,2-Hexanediol are low irritating to the human skin and usually regarded as safe substance, thus they are used as an ingredient in many cosmetic products [2]. They primarily serve as solvents to dissolve the active ingredients, and they are also used as moisturizers due to the ability to retain water molecules [3]. The diols contribute to the preservation of cosmetics from spoilage caused by microorganisms. So, they have been recently introduced as an alternative to parabens which have a problem of skin safety [4]. In this study, the effects of these diols in surfactant solutions regarding surface tension, foam and emulsifying power were determined as cosmetic ingredients. Also, the antibacterial effects of diols in surfactant solutions were evaluated.

Surfactants are used to lower the surface tension in a variety of processes where interface is created, e.g., in the mixing of fluids to create foams or emulsion. Because the formation of interface is a dynamic process, the effectiveness of surfactant is influenced by the thermodynamics that determine how a particular surfactant reduces the equilibrium surface tension [5]. Micelle is an aggregate of surfactant molecules that are formed in aqueous solution by self-assembling to minimize the contact area between the hydrophobic portion of the surfactant and water. To study the formation of micelles, the critical micelle concentration (cmc) is measured by investigating the change of surface tension as a function of surfactant concentraion [6-8]. The cmc is defined as the concentration of surfactants at which the micelle begins to form. Before reaching cmc, the surface tension changes noticeably according to the concentration of surfactants, and when cmc is reached, the surface tension remains relatively constant. The value of the cmc depends on temperature, presence of surface-active material and so on [9, 10].

Foams are generally formed as a result of gas dispersion through continuous surfactant solutions. Thus, the foams are described in terms of their foamability defined as the ability of the surfactants to create foam. Because the foams are thermodynamically unstable, it naturally collapses over time. Therefore, foam stability is investigated by the variation of foam height or volume over times, immediately after formation of foam. [11-13] Selection of surfactant has an impact on the properties of foam because it affects the surface tension and gas-liquid interfacial properties [14, 15]. In this study, sodium dodecyl sulfate (SDS) was used as anionic surfactant, cetyltri-methylammonium bromide (CTAB) as cationic surfactant and Tween 20 as nonionic surfactant were utilized.

An emulsion is a mixture of two or more immiscible liquids which are normally aqueous phase and oil phase [16]. Emulsions are thermodynamically unstable system and there may be several breakdown processes such as creaming, flocculation, sedimentation and Ostwald ripening. Accordingly, the emulsions require surface-active substances as suitable stabilizer suitable to adsorb on the droplet surfaces [17-19]. Surfactants having hydrophilic and hydrophobic groups prefer to be adsorbed on the interface between water and oil phase, thereby reducing interfacial tension and stabilizing emulsion, and thus they are necessary when forming an emulsion. [20, 21]. In order that, the emulsions are crucial in a wide range of applications including cosmetic, oil-field chemical and pharmaceutical filed. In this work, hexadecane which is one of most important oil related hydrocarbon groups and it is a substrate of low solubility in water was added to the surfactant solutions to form O/W emulsion [21].

To investigate the effect of diols, the surface tension, foaming, emulsion properties and antibacterial performances of surfactant solutions were analyzed, and the emulsions were created at various conditions.

2. Experimental

2.1. Materials

1,3-Butanediol (1,3-BDO, 99%), 1,2-Pentanediol (1,2-PDO, 98%), 1,2-Hexanediol (1,2-HDO, 96%) were purchased from Tokyo Chemical Industry. Sodium dodecyl sulfate and hexadecane were obtained from Sigma-Aldrich. Cetyltrimethylammonium bromide was purchased from DAEJUNG, Korea. Polyoxyethylene sorbitan monolaurate (Tween 20) was purchased from JUNSEI in Japan. The water was double-distilled and deionized using a Milli-Q Plus system, Millipore, France, with 18.2 MΩ·cm electrical resistivity at 25 ℃. All chemical reagents were used without further purification.

2.2. Preparation of several solutions and emulsion

1,3-BDO, 1,2-PDO, 1,2-HDO were prepared by diluting to the same concentration with 10 mM. Each surfactant is diluted with 10 mM diol and water using stock solution. Surfactant solutions were stirred for 15 minutes until completely dissolved. The initial concentration of surfactant was determined based on the critical micelle concentration.

O/W emulsion was prepared according to the ratio of water. The amount of hexadecane and SDS was fixed at 0.2 wt% relative to water. The emulsion was formed by homo mixer at 3000 rpm for 2 minutes.

2.3. Surface tension

Surface tension of solutions was measured using KRUSS K10ST surface tensiometer. It was designed to measure the air-water surface tension by the Du Nouy Ring method or Wilhelmy Plate method. Du Nouy Ring method utilizes the interaction of a platinum ring with the surface of the liquid. Wilhelmy Plate method use thin and rectangular plate made by platinum. Glassware and du Nouy ring were washed using ethyl alcohol, rinsed copiously with pure water and dried prior to use. The overall radius of ring is 9.5 mm and wire diameter is 0.18 mm. The ring was placed in surface of solution and the tensiometer zeroed. The air-water surface tension was measured until a steady, equilibrium value was reached. Then, the ring was cleaned and dried through flame.

2.4. Foaming properties

The dynamic Foam Analyzer DFA 100 (KRUSS, Germany) was used to identify the foaming properties of surfactant solutions with diol. DFA 100 is equipped with a 40 mm diameter prism column (measured internal diameter ~39.2 mm). Aqueous foams were created by injection of air (0.3 L/min) for 20 s through column height of 4.96 cm (60 mL of the prepared solution). Foam generation was terminated when a 100 mm foam column was achieved. The result of foam evaluation was collected using a foam analysis software. Initial foam volume $V_i$is the volume after air is injected into the solution for 20 s and the final foam volume after 10 mins is V_f. The initial volume V_i becomes the maximum value and means foam ability. And foam collapsing rate is an indicator of foam stability. The lower rate denotes that the foam is more stable. The collapsing rate is defined as

$$Foam\ collapsing\ rate = \frac{v_i - v_f}{t} \quad (mL/\min)$$

The structure of the foam was monitored with the camera positioned at 65 mm height mark on the mounting rail of the DFA 100. Through the camera, we took optical microscopic images to investigate the bubble morphology.

2.5. Emulsion properties

O/W emulsions were formed by using homo mixer. Oil in water emulsions are thermodynamic unstable systems. As time passed, it began to be separated into two layer and the boundary became clear. It indicates that breaking of emulsion started. The area of the sinking water increased while the area of the remaining emulsion decreased. Each volume was obtained by measuring the height of water and emulsion by time, the volume of sinking water is V_w, and the volume of remaining emulsion is V_e. Through V_w and V_e, Emulsion Stability Index (ESI), which is a volumetric method, can be obtained. A higher ESI value means that the emulsion is more stable, ESI was calculated by the following equation

$$Emulsion\,Stability\,Index = (1-\frac{v_w}{v_e})\times 100\,(\%)$$

The emulsion droplets are visualized by Nikon TI-DH optical microscope. This microscope was able to measure droplets size and discover several physical mechanisms such as flocculation, coalescence and Ostwald ripening.

2.6. MIC Test

To compare antibacterial activity of 1,3-BDO, 1,2-PDO and 1,2-HDO, Minimum Inhibitory Concetration (MIC) was measured against *Staphylococcus aureus* (*S. aureus*: ATCC 6538, gram-positive bacteria) by KMTR, Korea Institute of Microbiology and Technology. The MIC test is to measure the minimum concentration of an antimicrobial agents through performing the dilution method. The samples were added to Muller Hinton Broth which contains beef extract, casein hydrolysate, starch and agar. Then S. aureus were inoculated to the Broth and check the initial number of bacteria. After that, the bacteria were incubated at 37 ℃for 24 hours. By observing the growth of bacteria while diluting the sample, the MIC value can be determined. All diols are liquid sample, so that in this experiment, starting at 16 %, samples were diluted at 10, 8, 4, 2, 1 and 0.5 %.

3. Results and discussion

3.1. Critical Micelle Concentration

The critical micelle concentration (cmc) can be determined as the concentration-dependent surface tension (Fig 1). The surface tension was measured through KRUSS K10ST surface tensiometer and all experiments were conducted under the same conditions. The surface tensions were measured at various surfactant concentrations in aqueous solution without and with 10 mM of diols. The addition of diols induced the reduction in surface tension of surfactant solutions and the rate of decrease in surface tension was increased as the chain length of diols increased. Thus, the surface tensions of surfactant solutions with diols were 1,2-HDO 〈 1,2-PDO 〈 1,3-BDO.

The surface tensions of aqueous surfactant solutions above cmc at room temperature were reported in the literatures as shown in Table 1. Table 1 shows the surface tension of aqueous surfactant solutions above cmc at room temperature.

In case of 1,2-PDO and 1,2-HDO, the surface tensions of diols in water showed the same trend as in surfactant solutions. It is shown that the surface tension was decreased as the concentration of diols increased and remained constant above certain concentration as cmc in surfactant solutions as shown in Table 2.

Table 3 indicates thatthe cmc of 1,3-BDO in water was not found while the surface tension of 1,3-BDO solution in water was continuously decreased as the concentration of 1,3-BDO increased. These results might be induced by the high solubility of 1,3-BDO in water.

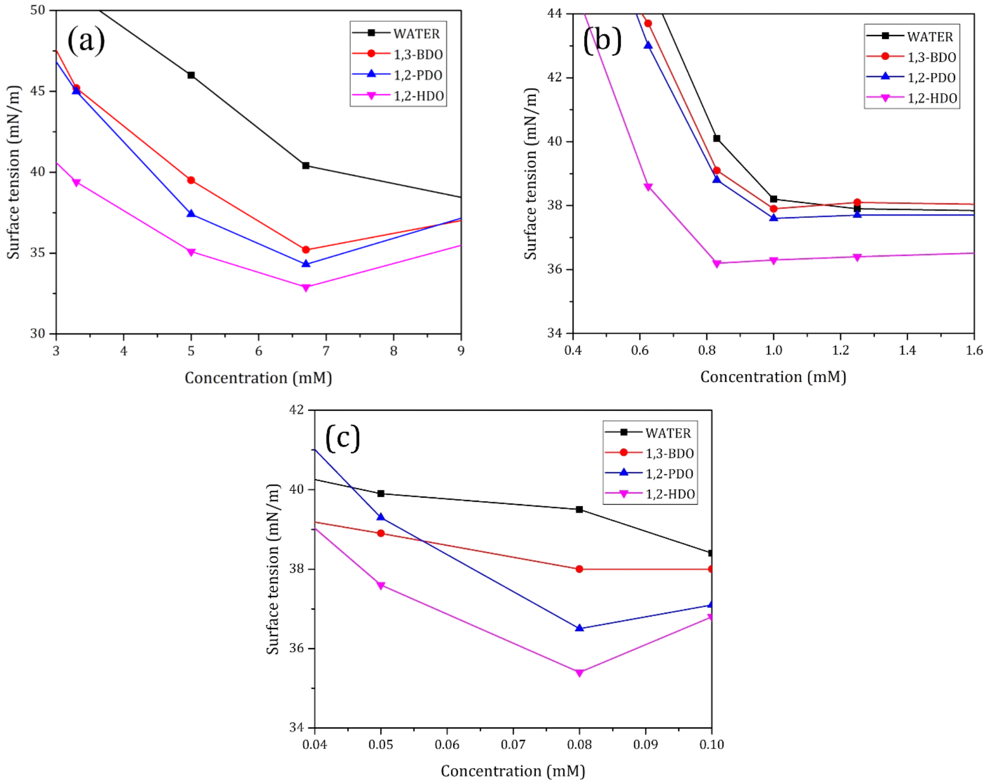

Fig. 1. Surface tension of (a) SDS, (b) CTAB, and (c) Tween 20 in aqueous solutions with different diols such as 1,3-BDO, 1,2-PDO and 1,2-HDO at room temperature.

Table 1. Surface tension of aqueous surfactant solutions above cmc

Surfactants	Surface tension (mN/m)
SDS	34.5
CTAB	36.0
Tween 20	39.0

Table 2. Surface tension and cmc of diols with aqueous surfactant solutions

Diol	Surface tension above CMC (mN/m)	CMC (mole fraction)
1,2-PDO	32.0	0.02
1,2-HDO	22.0	0.015

Table 3. Surface tension of 1,3-BDO in water

Mass % of 1,3-BDO in water	10	20	30	40	50	60	70	80
Surface tension (mN/m)	58.1	51.6	48.7	45.8	43.9	42.4	41.2	40.0

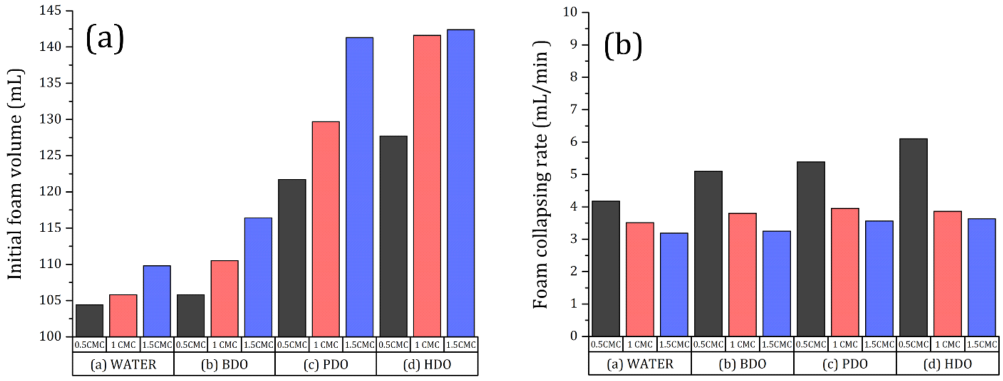

Fig. 2. Graph of (a) Initial foam volume and (b) foam collapsing rate of SDS aqueous solutions and the solutions with diols.

The presences of diols lowered the cmc compared to aqueous solutions. The cmc was reduced from 7.9 mM in SDS aqueous solution to 5.8 mM in solutions with diols, which decreased by about 26.6%. Regardless of the kinds of diol, the cmc of SDS with different diols were not varied significantly. In CTAB, it decreased by 16.4 % from 1.04 to 0.87 mM and in Tween 20, it decreased by 21.5 % from 0.079 to 0.062 mM. On average, it was found that the cmc decreased by approximately 21.5 % in the surfactant solutions with diol than in the aqueous solutions.

3.2. Foamability and Foam Stability

The initial foam volume and foam collapsing rate of SDS solutions were measured using DFA 100, the dynamic foam analyzer, and there were graphed (Fig 2). The foamability was identified in terms of initial foam volume and a larger initial foam volume indicates that the foamability is higher. The foam stability was measured to examine the foam collapsing rate, the lower rate indicates better stability. Based on the cmc, the surfactant concentration was set to 0.5 cmc, 1.0 cmc and 1.5 cmc. Fig 2 shows initial foam volume of the solutions with diols is larger than in aqueous solution and as the carbon chain length increases, the volume also increases. Conversely, foam collapsing rate is slower in the aqueous solution and the shorter chain length, the slower it is. There was also a slightly similar trend that the longer the carbon chain length, the lower the foam stability.

The bubble morphology of the foam was observed using the camera on the DFA 100 as shown in Fig. 3. The bubble size increased over time, and the size was larger in the case of containing diols than in aqueous solution. The larger size implies that the foam stability is reduced, and thus the stability is lower when the diols are contained [22].

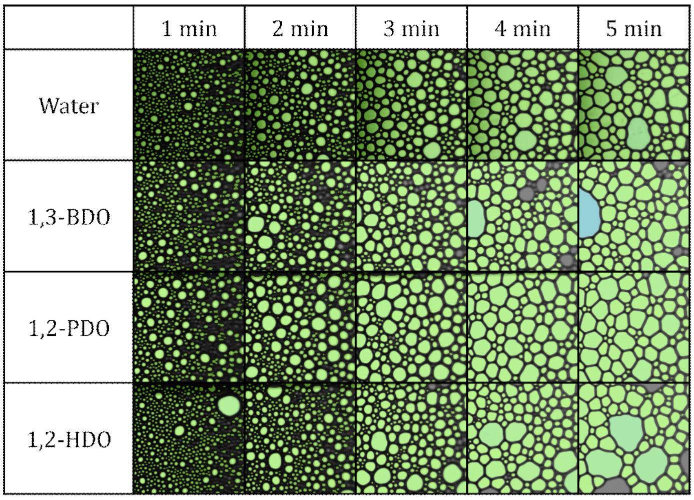

Fig. 3. Optical microscope images of sectional area of foam produced in SDS solutions with different diols.

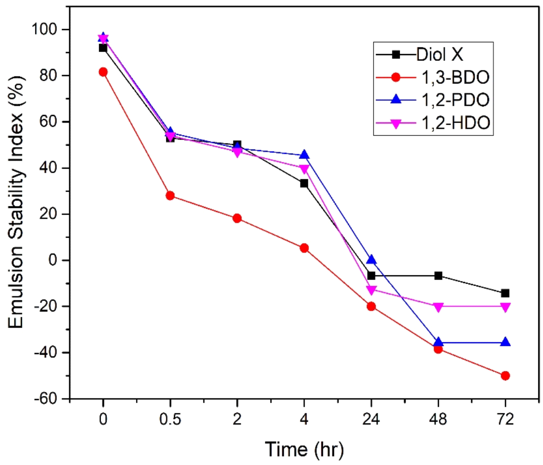

Fig. 4. Changes emulsion stability index of several emulsions over time

3.3. Emulsion Stability

The emulsion stability could be compared through the ESI value. The ESI is determined by observation of the boundary between two layers [23]. The higher ESI value means that the emulsion is more stable. Fig. 4. shows the ESI values according to time for different emulsions. SDS was commonly used as a surfactant and added 0.2 wt% to the amount of water. The diols were added in amount of 0.02mol relative to the water. The ESI values were lower than in aqueous solution in the presence of diols, and especially in the case including BDO. After 72 hours of emulsion formation, the ESI value of the emulsion without diol was −14%, the ESI of the emulsion containing 1,2 Hexanediol was −20% and the ESI for 1,2−Pentanediol and 1,3−Butanediol was −35 and −50%. These results indicates that the addition of diols reduce the stability of the emulsions.

Table 4. Minimum Inhibitory Concentration of 1,3-BDO, 1,2-PDO and 1,2-HDO against *S. aureus*

(a) Sample	Concentration (%)				-: not growth, +: growth		
	16	10	8	4	2	1	0.5
1,3-BDO	-	-	-	+	+	+	+
1,2-PDO	-	-	-	-	+	+	+
1,2-HDO	-	-	-	-	-	+	+

(b) Sample		MIC (%)
Diol	1,3-BDO	8% (80mg/mL)
	1,2-PDO	4% (40mg/mL)
	1,2-HDO	2% (20mg/mL)

Table 5. Minimum Inhibitory Concentration of Tween 20, SDS and CTAB against *S. aureus.*

(a) Sample	Concentration (%)								-: not growth, +: growth	
	16	10	8	4	2	1	0.5	0.1	0.05	0.01
Tween 20	-	-	-	+	+	+	+	+	+	+
SDS	-	-	-	-	-	-	-	-	-	+
CTAB	-	-	-	-	-	-	-	-	-	-

(b) Sample		MIC (%)
Surfactant	Tween 20	10% (100mg/mL)
	SDS	0.05% (0.5mg/mL)
	CTAB	Less than 0.01% (less than 0.1mg/mL)

3.4. Antibacterial activities

Minimum Inhibitory Concentration (MIC) test was performed to figure out and compare the antibacterial activities of each diol. Table 4 (a) shows whether growth occurs by concentration, and the MIC value of each diol can be found in Table 4 (b). This result indicates that the MIC of 1,3-Butanediol is 80 mg mL^{-1} for *S. aureus.* In the case of 1,2-Pentanediol and 1,2-Hexanediol, MIC was 40, 20 mg mL^{-1}, respectively.

Also, the antibacterial activities of surfactant itself have been measured. The MICs of nonionic Tween 20 and anionic SDS were 100 mg/mL and 0.5 mg/mL. Since the nonionic surfactant is mild to cell, the antibacterial of nonionic surfactant are quite low. But the MIC of cationic CTAB surfactant was less than 0.01 % as shown in Table 5. It has been reported that the cationic surfactant has a strong antibacterial effect.

4. Conclusions

To research the additive effect of diols on the surfactant solutions, surface tension, foaming and emulsion properties were examined using several measuring equipment such as tension meter, foam analyzer and so on. The presence of diols lowered critical micelle concentration of surfactant solutions by about 21.5 %. For foam properties, when the solution contained diols, the foamability increased and foam stability slightly decreased compared to the aqueous solution. This can be confirmed through the bubble morphology. As the carbon chain increased, the foamability improved because the longer alkyl chain is easy to transfer completely into the interior of micelle. By calculating the Emulsion Stability Index according to time, it was found that the addition of diols lowered the emulsion stability and especially, the stability was reduced in case of 1,3-Butanediol. These tendencies were determined to be due to the hydrophobic effect which is propensity of nonpolar substances to aggregate in an aqueous solution and exclude water molecules. Diols are polar solvent with two O-H groups, which gives the ability to from hydrogen bonds. As a result, the presence of diols in mixtures decreases the cohesive force of water. Furthermore, Minimum Inhibitory Concentration test was conducted to analyze the characteristics of each diol, with the conclusion that 1,2-Hexanediol had the best antibacterial activity.

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