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Preparation of Anion Surfactant Series of Sulfated Alkyl Fatty Acid Sodium from Squid Fat

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The great leader Kim Jong Il said:

"The sector of science and technology should pinpoint as seeds the problems that demand urgent solution in the respective realms and the tasks that can produce the greatest profit, and solve them one by one."

Since mineral oil resources such as crude oil being exhausted, the studies of extracting various sorts of surfactants from oils and fats of animals and plants including soybean oil, sardine fat and launce fat are actively done [1, 3].

There are two methods for extracting surfactants; one is to synthesize from natural oils, the other to synthesize the fatty acids industrially from mineral oils.

Iodine values characterize the content of unsaturated bonding; iodine value of squid fat is 172～204, further more 207～208 and unsaturated rate is great. Therefore squid fat is able to be esterificated by sulfuric acid and anion surfactant with high surface activity can be made from squid fat. [2].

We studied the preparation of an anion surfactant series of sulfated alkyl fatty acid sodium from squid fat, natural fat which can be easily obtained in the east sea.

1. Experiments

The preparation process of extracting an anion surfactant series of sulfated alkyl fatty acid sodium from squid fat includes the following three stages.

Saponification reaction stage: unsaturated fatty acid glyceride＋water→sodium of unsaturated fatty acid＋glycerine

Sulfation reaction stage: sodium of unsaturated fatty acid＋sulfuric acid→Sulfated alkyl fatty acid ＋sodium sulfate

Neutralization reaction stage: Sulfated alkyl fatty acid＋sodium hydroxide→sulfated alkyl fatty acid sodium

Here the main stages are saponification reaction and sulfation reaction.

Saponification reaction of squid fat was progressed as follows.

20g of squid fat is filled in reactor and the rate of fat and water is 1:3. The reactor is heated to constant temperature and then 20mL of constant concentration of NaOH solution is added little by little while stirring. After reacting for a certain time, the yield is determined by titrating the residual sodium hydroxide with hydrochloric acid.

After reaction, the salting out is progressed by adding hot sodium chloride saturated solution in reactor while stirring. The obtained soap is filtrated with suction and dried and then the yield is determined.

Sulfation reaction was progressed as follows.

First, unsaturated fatty acid sodium obtained from saponification stage is added in reactor and heated to constant temperature and the ratio of solid and liquid is 1:3. Then the sulfuric acid of constant concentration drops in the stirring state.

After reaction, the liquor is neutralized with sodium hydroxide till pH is 7.

The obtained sulfated fatty acid sodium is salted out, separated, filtrated and dried and then the yield is determined. The yield of product is determined by the rate of obtained amount in experiment on theoretical amount.

2. Results and Discussions

2.1. Saponification reaction stage

Effect of sodium hydroxide concentration. We considered the effect of the sodium hydroxide concentration on the yield of product while reacting it for 1h at 70℃. The result is shown in Fig. 1.

As shown in Fig. 1, the yields indicate approximately constant values when the concentrations of sodium hydroxide are higher than 30%. This result implies that the produced soap species sufficiently ensure the phase contact between fat and water when the concentrations of sodium hydroxide are higher than 30%. That is, because the produced soap species play the dispersant action.

Effect of reaction temperature. Reaction temperature plays an important role in saponification.

Effect of reaction temperature on saponification yield was studied in the condition that the concentration of NaOH is 30% and the reaction time is 1h. Figure 2 showed the result. As shown in Fig. 2, the yield was rapidly increasing to 80℃ as the temperature increased but above 80℃, it was smoothly increasing.

The fact shows that the temperature should be over 80℃.

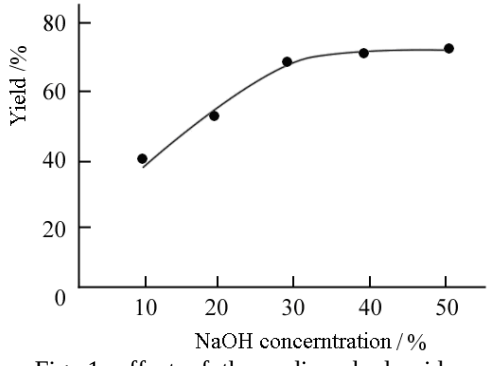

Fig. 1. effect of the sodium hydroxide concentration on the product yield

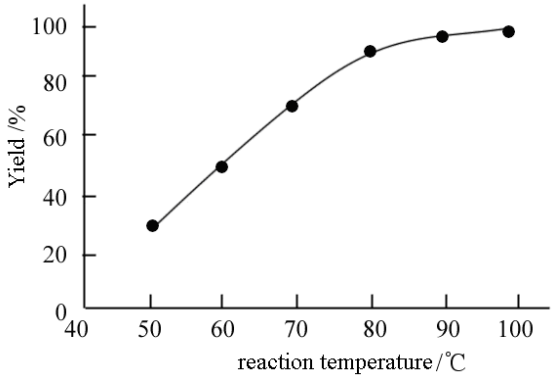

Fig. 2. Effect of reaction temperature on saponification yield

Effect of reaction time Effect of reaction time on yield of saponification was studied in 30% of concentration of NaOH at 100℃.

Fig. 3 showed the result. As shown in Figure 3, the yield reached almost 100% at 100℃ for 1.0～1.5h.

According to the above results, the concentration of NaOH is 30%, the reaction time is 1.0～1.5h, the reaction temperature is 100℃. Under these conditions the yield is over 95%.

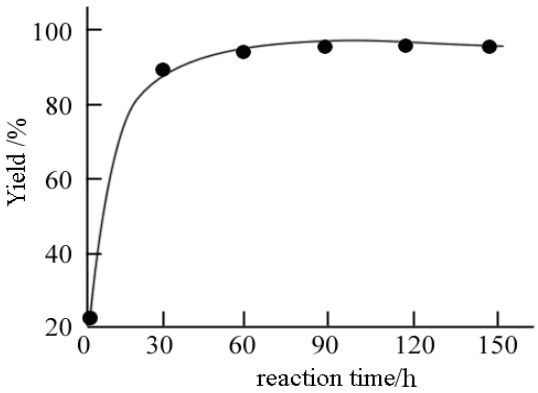

Figure 3. Effect of reaction time on yield of saponification

2.2. Sulfation stage

Effect of NaOH concentration. Effect of H_2SO_4 concentration on yield of product was investigated for 30min at 30℃.

Fig. 4 showed the result.

As shown in Fig. 4, the yield was increasing to 30% of H_2SO_4 concentration with time but above40% it was rapidly decreasing. This can be attributed to occurrence of secondary reactions such as oxidation, polymerization and hydrolysis in high concentration.

Effect of reaction temperature. Effect of reaction temperature on yield of product was investigated in 30% H_2SO_4, reaction time 30min. Fig. 5 showed the result.

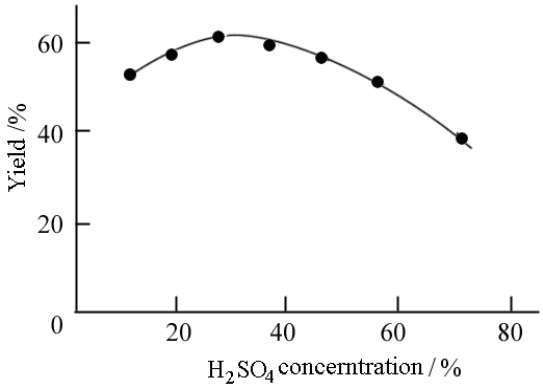

Fig. 4. Effect of H_2SO_4 concentration on yield of product

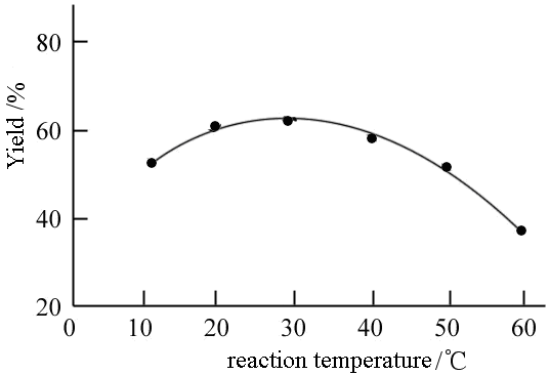

Fig. 5. Effect of reaction temperature on yield of product

As shown in Fig. 5, the yield was 62%, the maximum value, at 20～30℃ but beyond 30 ℃ and it was decreasing. That is why the amount of oil to react with H2SO4 is reduced due to oxidation of oil. Because the higher the unsaturation degree of fatty acid, the more heat emitting in sulfation process, to regulate the temperature appropriate for sulfation, the dropping of sulpheric acid should be slowly performed with vigorous stirring.

From the results the optimal conditions are 30% of H_2SO_4 concentration, 20～30℃ of reaction temperature and reaction time 30min and under these conditions the yield is 62%.

Conclusion

The optimal conditions in saponification reaction of squid oil are 30% of NaOH concentration, 100℃ of reaction temperature, 1~1.5h of saponification time and in this case the yield was over 95%.

In sulfation reaction of sodium salt of unsaturated fatty acid from squid oil, the optimal conditions are 30% of H_2SO_4 concentration, 20~30℃ of reaction temperature and reaction time 30min and under these conditions the yield is 62%.

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