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Synthesis and Properties of Lauric Acid-2-hydroxy-3-propane Sulfonic Acid Sodium Salt

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The lauric acid-2-hydroxy-3-propane sulfonic acid sodium salt was synthesized by using sodium bisulfite, epichlorohydrin, sodium phosphate and lauric acid. The influence factors on reaction temperature, reaction time, the amount of catalyst and reactant molar ratio to the yield were investigated. The favorable conditions of synthesizing were determined:epichlorohydrin was added into the solution of sodium bisulfite of 85 °C, stirring for 2 h and left for 1.5 h. 3-Chloro-2-hydroxy-propane sulfonic acid sodium was synthesized. 3-Chloro-2-hydroxy-propane sulfonic acid sodium was mixed into the solution of sodium phosphate at 55 °C and 4 h reaction time. The 2,3-oxiranemethane sulfonic acid sodium salt was formed. 2,3-Oxiranemethane sulfonic acid sodium salt solution was dropped into the solution of lauric acid at 90 °C, the dropping time 0.5 h and left for 2.5 h. Lauric acid-2-hydroxy-3-propane sulfonic acid sodium salt was obtained. The yield was 85.2 % and was characterized by FTIR.

Keywords: Lauric acid, Epichlorohydrin, Synthesis, Lauric acid-2-hydroxy-3-propane sulfonic acid sodium salt.

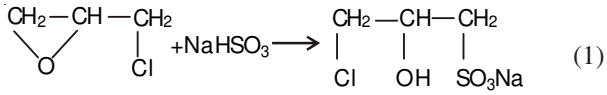
INTRODUCTION

With the growing tension of oil energy situation, improving oil recovery rate has became a major issue of oil exploitation. Surfactant in terms of enhanced oil recovery has been increasingly used[1-6]. Sulfonate surfactant is a kind of surfactant that has the most yield and widest application. In this paper, Lauric acid-2-hydroxy-3-propane sulfonic acid sodium salt was synthesized by using fatty acid and epichlorohydrin. The product was characterized by FTIR. The result shows that the surfactant has a better surface activity. This new kind of surfactant has huge potential based on the advantages of low cost and green.

EXPERIMENTAL

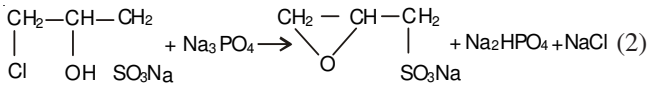
Sodium bisulfite, sodium phosphate, epichlorohydrin, octadecanoic acid, quaternary ammonium salts were AR grade. JC2000CI intravenous infusion of the contact angle of the interfacial tension measuring instrument, the Shanghai Morning Digital Technology Equipment Co., Ltd. products; WRS-1B digital melting point apparatus, Shanghai Fine Scientific Instruments Co., Ltd. products; TENSOR27 infrared spectrometer, Bruker Spectrum Instrument Company products.

Synthetic principle: The sulfonation reaction of epichlorohydrin and sodium bisulfite generates 3-chloro-2-hydroxy-propane sulfonic acid sodium salt.



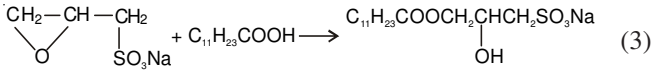
(1)

The closed loop reaction of 3-chloro-2-hydroxy-propane sulfonic acid sodium and sodium phosphate generates 2,3-oxiranemethane sulfonic acid sodium salt.



(2)

The esterification reaction of epoxy propane sulfonate and lauric acid generates stearic acid-2-hydroxy-3-propane sulfonic acid sodium salt.



(3)

Synthetic procedure: A certain amount of water and sodium bisulfite were placed in a four-necked flask equipped with a reflux condenser, a thermometer and a stirring rod. The mixture was then heated and stirred until all the sodium bisulfite was dissolved. The epichlorohydrin was slowly added dropwise to the solution when the temperature reached to 85 °C, left for 2 h and then stirring for 1.5 h. The solution was then cooled to room temperature, and continued to cool in an ice bath, then the solution was filtered to give a solid: 3-chloro-2 -hydroxy-propane sulfonic acid sodium salt. The yield is 80.6 %.

The aqueous solution of sodium phosphate was heated to 55 °C, then the 3-chloro-2- hydroxy-propane sulfonic acid sodium was added to the solution[7]. The product was cooled after 4 h, and filtered. The filtrate was distilled under reduced pressure to give a waxy 2,3-oxiranemethane sulfonic acid sodium salt. The yield was 85.2 %.

The four-necked flask equipped with lauric acid, a moderate amount of water and quaternary ammonium salt (catalyst) was stirred and heated to 90 °C, then epoxy propanesul-fonate solution was added dropwise to the four-necked flasks, droping about 0.5 h, reaction 2.5 h maintained at 90 °C. The solution was cooled and filtered to give crude product. The crude product was recrystallized from ethanol to give the product as a white powder in a yield of 85 %.

RESULTS AND DISCUSSION

Influence factors for synthesizing 3-chloro-2-hydroxy-propanesulfonate

Temperature: We explored the effects of reaction temperature on the yield of product on the condition that the reaction time was 3.5 h, the molar ratio of the reactants was 1.15:1 (Fig. 1).

Fig. 1 showed that the reaction temperature increases, the yield rises at first and then gradually reduces. When the reaction temperature is below 75 °C, the sulfonating agent can not be fully dissolved and affects the reaction proceeding[8], and the yield rises gently. When the temperature is higher than 85 °C, with the reaction temperature increases, the yield slightly decreases. Partial decomposition of sodium bisulfite at high temperatures reduces the effective concentration of the sulfonating agent. Epichlorohydrin, being also prone to self-polymerization reaction, results in the increasing of by products. These factors make yield decreases. When temperature is 85 °C, the maximum yield of the product is 80.6 %.

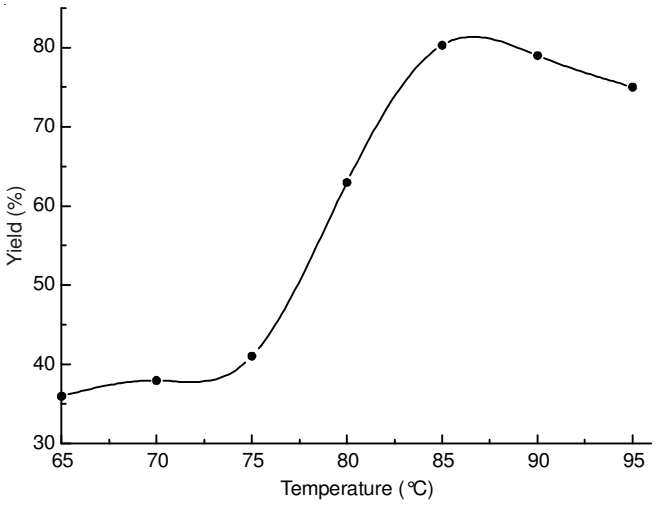


Fig. 1. Effect of the reaction temperature on the yield

Reactant molar ratio: Effect of the molar ratio of sodium bisulfite and epichlorohydrin on the yield is shown in Fig. 2.

Fig. 2 showed that when the reaction time is 3.5 h, the reaction temperature is 85 °C and other conditions, the yield rises with increasing the molar ratio of sodium bisulfite. when

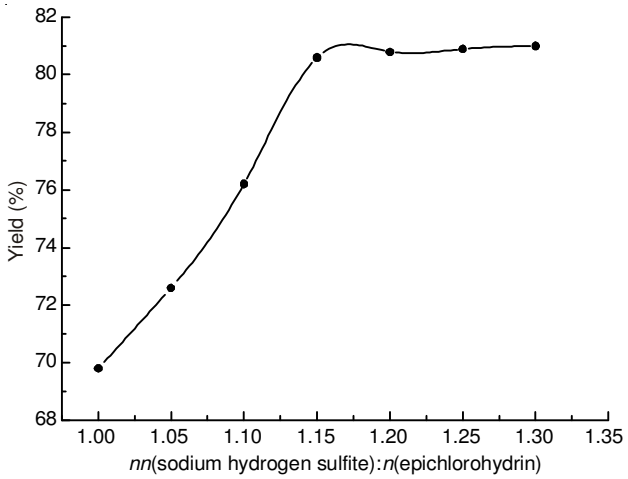


Fig. 2. Effect of the molar ratio on the yield

n (sodium hydrogen sulfite): *n* (epichlorohydrin) is 1.15:1, the maximum yield of the product is 80.6 %. then yield tends to be gentle.

Effect of loading sequence on the yield and reaction time: The experiment shows that adding dropwise epichlorohydrin to the aqueous solution of sodium bisulfite is beneficial to the yield. As the sodium bisulfite is a good nucleophilic reagent, the aqueous solution of sodium dithionite has a certain concentration of H^+, the acidic environment makes the epoxy ring oxygen protonated, the C-O bond weaken, and ring carbon atom with a positive charge. that increases the ability to bind with a nucleophilic reagent, epichlorohydrin ring-opening reaction is easily performed[9]. The yield is improved. Conversely, if sodium bisulfate is added dropwise to epichlorohydrin. the acidic environment in the initial stage of the reaction is weak, the reaction does not proceed in a timely manner, and thus reaction time is long, the yield is relatively low.

Influence factors for synthesizing the main product lauric acid-2-hydroxy-3-propane sulfonic acid sodium salt

Catalyst screening: We explored the effects of different catalysts (mass fraction 1.2 %) on the esterification reaction on the condition that the reaction temperature was 90 °C, reaction time was 3 h, *n* (epoxy propane sulfonate): *n* (lauric acid) was 1:1.1, the data was shown in Table-1.

TABLE-1
EFFECT OF DIFFERENT CATALYSTS ON THE YIELD

Catalysts	Yield (%)
Sixteen alkyl trimethyl ammonium bromide	68.6
Benzyl trimethyl ammonium chloride	78.3
Benzyl triethyl ammonium chloride	85.2

Table-1 showed that when the other conditions are the same, benzyl triethyl ammonium chloride is used as a catalyst, the yield is higher than the previous two catalysts, so the experiment used benzyl triethyl ammonium chloride.

Condition experiment: We have investigated the effect of the amount of catalyst, reaction temperature, reaction time and the molar ratio of on the product yield. As shown in Table-2. When the *w* (catalyst) is 1.2 %, the T is 90 °C, the reaction time is 3 h and the molar ratio of epoxy propane sulfonate and lauric acid is 1:1, the yield is the highest, up to 85.2 %.

TABLE-2
EFFECT OF DIFFERENT CONDITIONS ON THE YIELD

S.No.	w(catalysts) (%)	Yield (%)	t (°C)	Yield (%)	h	Yield (%)	Molar ratio *n*(epoxy propane sulfonate): *n*(Lauric acid)	Yield (%)
1	0.6	58.6	80	65.6	2.0	71.2	1:0.8	74.1
2	1.0	77.0	85	76.6	2.5	78.4	1:0.9	78.9
3	1.2	85.1	90	85.2	3.0	85.2	1:1.0	85.2
4	1.5	81.8	95	80.3	3.5	80.1	1:1.1	79.8

Infrared spectrum: In Fig. 3, the absorption peak at 3362 and 1558 cm^{-1} may be due to hydroxyl special absorption peak. The absorption peak at 1731, 1232 and 1180 cm^{-1} may be the absorption peak of the ester group. The absorption peak in 2957 and 1422 cm^{-1} may be a methyl group absorption peak. The absorption peak in 1052 cm^{-1} is due to sulfite group stretching vibration absorption peak and in 2850 cm^{-1} is for methylene group stretching vibration absorption peak, whereas the absorption peak of the epoxy bond in 3050, 2150, 915 and 850 cm^{-1} are not present.

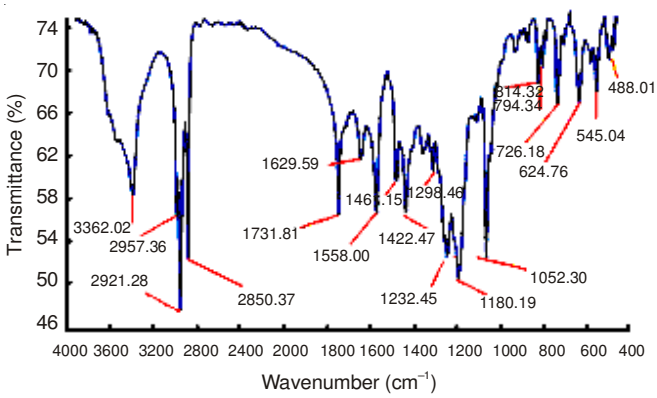


Fig. 3. FTIR absorption spectrum of the product

Measurement of critical micelle concentration: Critical micelle concentration is the important parameter of surfactant performance, surface tension of lauric acid -2-hydroxy-3-propane sulfonic acid sodium salt of different concentration was measured by using JC2000CI surface tension meter. The critical micelle concentration was obtained, and the surface performance was compared with sodium dodecyl sulfate (SDS). Results were shown in Table-3.

TABLE-3
CRITICAL MICELLE CONCENTRATIONS AND THEIR SURFACE TENSIONS OF SURFACTANTS

Samples	SDS	Product
Surface tension (mN m^{-1})	40.0	33.10
CMC (mmol L^{-1})	8.0	6.38

Table-3 suggested that the critical micelle concentration of product is the lower compared with the conventional anionic surfactant SDS. This showed the surfactant has a better surface activity.

Conclusion

Lauric acid-2-hydroxy-3-propane sulfonic acid sodium salt was synthesized by using fatty acid and epichlorohydrin. The product was characterized by FTIR result shows this surfactant has a better surface activity. The yield was 85.2 %. The critical micelle concentration was 6.38 mmol × L^{-1}. This synthetic route has a lower reaction temperature, high yield, readily available raw materials.

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