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Synthesis and Characterization of Sulfosuccinate Monoester Surfactant from Natural Renewable Resources

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Abstract

The increasing environmental awareness and the use of renewable resources provide opportunities for the use of oleochemicals in surfactants. Through new technologies that are emerging, especially in the field of oleochemical-based surfactants has made it more attractive in recent times. Surfactants can be derived from petrochemical feed stocks and renewable resources (e.g.oleochemical). Renewable resources have the advantage that they contribute less effect to the environment as well as aquatic organisms. Surfactant production is based on 40% petrochemical and 60% oleochemical feed stocks. The basic oleochemical feed stocks are fatty acid, fatty alcohol, fatty acid methyl ester, fatty amine, glycerol, these oleochemicals are derived from vegetable/ animal oils and fats. The sulfosuccinate monoester is an alternative anionic surfactant to non ionic surfactant due to their mildness properties in cosmetics and personnel care product. In this study, disodium sulfosuccinate monoester surfactant was synthesized by esterification of unsaturated fatty alcohols(Oleyl alcohol), which are special and can only be obtained from natural sources with maleic anhydride followed by sulfonation of sodium bisulphate and neutralized with sodium hydroxide solution. The optimum conditions for each step were determined. The progress of reaction were monitored by TLC, AV, HV, SV and % sulfite content. The structures of the sulfosuccinate monoesters were unambiguously resolved on the basis of FTIR, ^{1}H-NMR and ^{13}C-NMR. Antimicrobial properties were evaluated against following bacteria: Staphylococcus aureus Bacillus subtilis and Escherichia coli. The surfactant properties of sulphosuccinate including surface tension, critical micelle concentration, maximum surface area, % yield, cloud point, wetting, foaming and dispersion power, Gibbs adsorption density, enthalpy and free energy of micelle formation were calculated for the analysis of the micellization process in relation to their chemical structures.

Keywords: Fatty alcohol, Sulfosuccinate, surface tension, cmc, antimicrobial activity.

1. Introduction

Fats and oils are renewable resources that can be treated chemically or enzymatically to produce oleochemicals, which often act as a replacement for petroleum-derived materials in that sense, Oleochemicals are the chemicals derived from plant and animal oil & fats. [1-2]. Renewable resources have the advantage that they contribute less effect to the environment as well as aquatic organisms [3]. The basic oleochemical feed stocks are fatty acid, fatty alcohol, FAME, fatty amine, glycerol. Oleochemical based surfactants are easily biodegradable having low toxicity and biocompability over petroleum synthesized surfactants [4]. Surfactants as a large consuming chemical are closely interrelated to our life, industry and environment. Nowadays energy and environment are two main concerns which need to be solved by science and technology[5-6]. Surfactants have also been widely used in textiles, fibers, food, paints, polymers, cosmetics, pharmaceuticals, mining oil recovery, pulp paper industries etc.[7]. Surfactants obtained from fatty alcohols are widely used in household products, industrial detergents and personal care products [8].

Sulfosuccinate surfactants are especially tailored to these needs. They are extremely mild to both skin and eyes, Sulfosuccinate can be divided into two types: Diester and Monoester. Out of it, The monoester type is ideal for personal care applications and reduces the particle size of nanoparticle. It is a good foamer and mild to skin and eyes. Sulfosuccinate monoester based on fatty alcohol, which consist of hydrophobic tail and sulphate group are hydrophilic head[9]. The composition of the chain length of fatty alcohols, varying from 10 to 18 carbons, has a direct impact on the properties of surfactants [10]. Oleyl alcohol consist of a double bond in long alkyl chain, These sulfosuccinate monoester surfactant synthesized by oleyl alcohol as a raw material ,which is a natural renewable resources having outstanding efficiency and effectiveness in reducing surface tension[11]. Interactions of water with surfactant molecules are generally investigated by various physicochemical methods such as FTIR [12], NMR [13], Surface tension [14] Antimicrobial activity [15] Solubilization [16].

The goal of the present work is to synthesized a anionic sulfosuccinate monoester without using any phase catalyst, to determine surface properties, biological properties, solubilizing properties by light scattering technique and their physicochemical properties.

2. Experimental Procedure

2.1 Materials

Oleyl alcohol (35%) and maleic anhydride, pet ether were purchased from s.d.fine chemicals, mumbai; sodium bisulfite purchased from sisco pvt.ltd. Mumbai, NaoH from Merck,

2.2 Synthesis of Disodium Oleyl Sulfosuccinate Monoester Surfactant:

Oleyl alcohol (0.111mole) was loaded into a three neck reactor vessel and heated to 60-70°C that leads to melting of alcohol. The temperature was kept constant at this level and maleic anhydride 0.122 mole, was continuously added by stirring and keeping the temperature on a constant level. The heat of reaction causes the anhydride to melt. The reaction was continued by stirring the mixture for 2.5 hrs at 70^0C. Then, an aqueous solution (30%) of sodium bisulphite (0.122mole) was added to reaction mass. The reaction was completed by continuous stirring at 90^0C up to 1.5 hrs. The product was neutralized with 25% aqs NaOH solution. For the purification purpose, the synthesized product was washed with pet ether 2 to 3 times for removal of unwanted impurities and unreacted materials. Then, the waxy yellowish lumps are obtained and dried at 105^0C.

3. Methods

3.1 1H-NMR Spectra

Nuclear magnetic resonance spectroscopy is widely used as one of an armory of instrumental techniques available for structure analysis in Chemistry.

3.2 Fourier Transform Infrared Spectral Analysis

FTIR analysis was carried out by using a shimadzu model FTIR-8400. Its used to monitored the functional groups of the synthesized products. The sample was coated onto KBr pellets.

3.3 Surface Tension Measurements

Surface tension measurements was performed for freshly prepared solutions of sulfosuccinate derivatives in a concentration range of 0.02-0.10 mole/L using Kruss du Nouy tensiometer, with the help of Krüss Standard Ring method (Kruss GmbH, Hamgurg, Germany).

3.4 Antimicrobial Activity

Bactericidal activity tests was carried out against *Staphylococcus aureus* ,Bacillus subtilis, Escherichia coli using the diffusion agar technique and their antifungal activities were tested against *Candida albicans* using an agar plate technique.

3.5 Solubilization Measurement

A light-scattering technique was used to measure the solubilizing power of the prepared surfactant at 25^0C using a systronic model turbiditymeter. The solubilizing power of surfactant solution(0.05%) was measured using 0.1 gm of paraffin oil as nonpolar solute was mixed together using magnetic starrier at 260 rpm for different time intervals and turbidity was measured.

4. Results and Discussion

Table 1: Physico-chemical characterization of sulfosuccinate monoester:

Name of Product	Acid Value Before	After	Sap.Value Before	After	H.V.	pH	% active matter	% bisulfide content	Cloud point $0^0C(1\%)$	% solid	% yield
DSOSS	60.49	1.43	148.50	95.97	00	7.1	38.44	6.79	8	95.96	78.58

The purified disodium oleyl sulfosuccinate monoester surfactant was characterized by analytical and instrumental method. The AV and SV of DSOSS shows very high changes, before reaction, the AV, SV is very high and after reaction AV, SV was decreases. The free fatty acid is converted in to ester, hydroxyl value is zero that means alcohol is converted in to ester. pH of DSOSS shows neutral, % active matter found very high percentage. In DSOSS % bisulfate found very low, at very low temperature it forms cloudy. Yield of DSOSS becomes very high with out use any catalyst during synthesis.

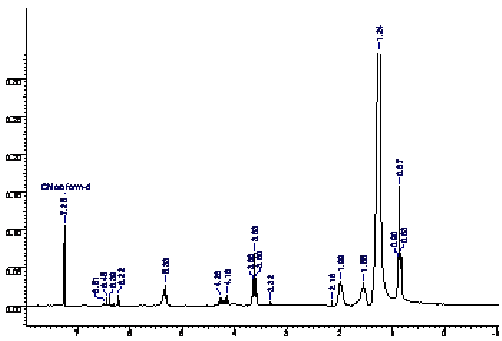

Fig. 1: FTIR of Disodium Oleyl Sulfosuccinates

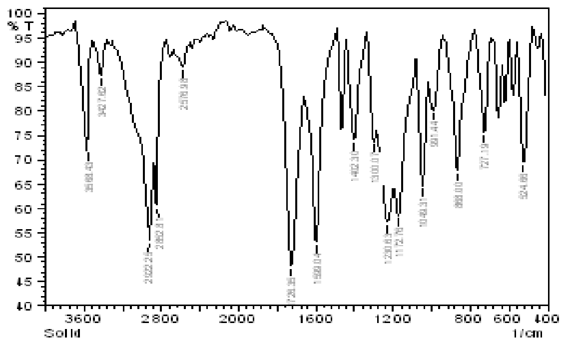

Fig. 2: ^{1}HNMR of Disodium Oleyl Sulfosuccinates

Fig. 1 shows FTIR spectrum of DSOSS was performed to characteristic absorption band at 1726 cm^{-1} for ester sulfosuccinate. The stretching vibration band of C=S Symmetric and S=O symmetric Stretching appears at 1402,1049 $cm^{-}1$ and Fig.2 confirms the surfactant structure from the ^{1}H-NMR spectra

The surface tension of DS-OSS decreases significantly from 26.602 mN/m to 25.230 mN/m for the surfactant concentration of 0.02% to 0.14% respectively. It shows slight reduction in S.T. because of it has one double bond of unsaturation with higher C-18 chain length. The CMC of DS-OSS was determined from intersection point in the surface tension Vs. log concentration value. It is obtained 0.046 mg/lit.

Table 2: Surface Properties of DSOSS:

Name of Product	Surface tension	CMC (mg/l)	Surface excess (mol/mm^2)	Surface area/molecule (mm^2)	Gibbs F. E. of adsorpt-ion(J/mol)	Gibbs F. E. micellization (J/mol)	Foam stability (%)
DSOSS	25.230	0.046	2.0768	7.9957	50200.28	-07644.08	72.50

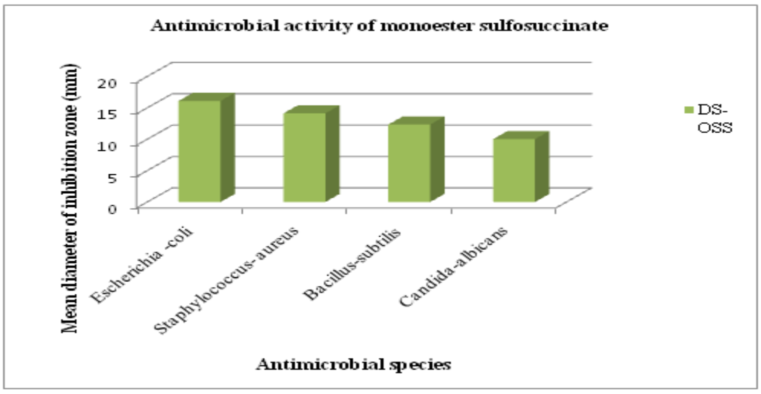

Fig. 3: Antimicrobial activity of DSOSS.

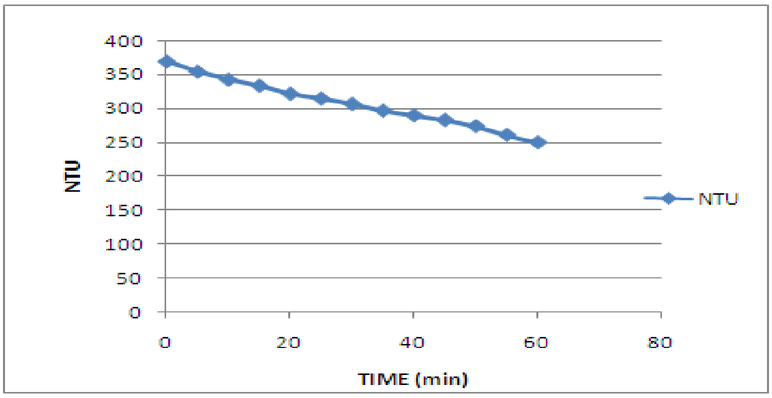

Fig. 4: Solubilization behavior of DSOSS.

Fig. 3 Shows antimicrobial activity of DSOSS was assayed with four different microbes. The organisms used in the assay were bacterium Staphylococcus-aureus, Bacillus-subtilis, and Escherichia-coli.The bioactivity of surfactants is an increased activity against all microorganisms; this is probably due to the increase in liposolubility which favours its permeation through the lipid layer of the microorganism.

Fig.-4 Shows the solubilizing behavior for the surfactant/ paraffin oil system of DSOSS, this can be attributed to the fact that the amount of material solubilized increased with an increase in time. As the paraffin oil was dispersed in surfactant solution to increase in time decreases the CMC.

5. Conclusion

The synthesized sulfosuccinate monoester surfactants have better profile for mildness for skin and eyes with surface properties viz. cleansing, emulsifying, dispersion and foaming properties. Due to their antimicrobial activity, they have applicability in personnel care product. The oleyl sulfosuccinate surfactant was synthesized without any phase catalyst gives very high yield. These synthesized route are ecofriendly and reduces the environmental problem. The surface properties and biological properties of synthesized surfactant found very good.

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