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SYNTHESIS AND CHARACTERIZATION OF CHOLINE CHLORIDE-BASED CHOLINE LAURYL SULPHATE: A NEW GREEN SURFACTANT

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ABSTRACT

This study presents the synthesis and characterization of choline chloride-based choline lauryl sulfate (ChLS) as a novel biocompatible surfactant. The synthesis process involves the reaction between sodium lauryl sulfate and choline chloride in presence of dichloromethane. The resulting ChLS was characterized using various analytical techniques, including Fourier-transform infrared spectroscopy (FTIR), 1H NMR spectroscopy, and surface tension measurements. The FTIR spectrum of ChLS shows significant peaks indicating the presence of cholinium ion and laurylsulfate anion bands. Mass spectrometry confirmed the molecular weight of 265.39 g/mol. Surface tension measurements showed a reduction to 38.7+/- 0.57 mN/m, indicating superior performance compared to sodium dodecyl sulfate (SDS). FTIR, MS and NMR analyses plconfirmed the successful formation of the desired surfactant, while surface tension measurements demonstrated its surfactant properties. The critical aggregation concentration (CAC) was determined to be 0.8 mM, demonstrating efficient micelle formation. Thermal analysis revealed stability up to 256.3°C ($T_{decomposition}$), with decomposition starting at 256.3°C. These findings suggest ChLS as an effective, thermally stable surfactant suitable for various industrial applications. The synthesized ChLS exhibited promising surface-active properties and enhanced biodegradability, making it a potential candidate for various industrial and cosmetic applications. This study contributes to the development of sustainable surfactants with reduced environmental impact.

Keywords: Choline chloride, Choline lauryl sulfate, Green surfactant, Synthesis, Characterization, Biodegradability, Environmental impact, Industrial applications, etc.

Abbreviations:

SAIL Surface-active ionic liquid

Cho Choline

Ole Oleate

Lin Linoleate

Eru Erucate

DMSO Dimethyl sulphoxide

CMC Critical micelle concentration

DSC Differential scanning calorimetry

SDS Sodium dodecyl sulfate

SDBS Sodium dodecyl benzene sulfate

1. INTRODUCTION

The increasing demand for environmentally friendly surfactants has driven the exploration of novel compounds with reduced environmental impact. Choline chloride, a quaternary ammonium salt, has gained attention due to its low toxicity and biodegradability [1,2]. In this study, choline chloride was utilized as a key component in the synthesis of a new green surfactant, Choline based choline lauryl sulfate (CCLS) [3]. Lauryl sulfate, derived from natural sources, serves as the anionic moiety in the synthesized surfactant. The combination of choline chloride and lauryl sulfate aims to produce a surfactant with enhanced biocompatibility and reduced environmental footprint compared to conventional surfactants [4].

Sodium Lauryl Sulphate (SLS), also known as sodium dodecyl sulfate (SDS), serves as an anionic surfactant, finding utility as a foaming and cleansing agent in detergents, a wetting agent in textiles, a cosmetic emulsifier, and occasionally in toothpaste formulations [5-7]. While it boasts versatile applications, SLS is notorious for its drying effect on both skin and hair, leading to a lackluster appearance and rendering the skin more susceptible to the penetration of toxins [8]. Moreover, SLS poses the risk of absorption into the body, potentially accumulating in internal organs and escalating the likelihood of enduring health issues. Additionally, SLS stands as a recognized pollutant [9,13].

The World Health Organization (WHO) cautions against the direct introduction of sodium lauryl sulfate into the environment in its raw state, as it poses a hazard to aquatic life, with the potential for accumulation in their systems [10]. There's also a risk of SLS contamination in municipal water supplies, thereby entering tap water undetected [11]. The manufacturing process of SLS from petroleum is marked by significant pollution, emitting volatile organic compounds and sulfur compounds that may contribute to cancer risk, as well as releasing air particulates [12]. Furthermore, the production process involves the use of toxic solvents, including carcinogenic nitrates, which may leave residual traces in the final product. Ionic liquids (ILs) serve as a significant category of eco-friendly surfactants, garnering considerable attention in recent years due to their distinct physical and chemical characteristics [13-15].

Comprising entirely of ions, IL-based surfactants (ILBSs) have attracted widespread interest [16]. However, many ILs pose environmental risks, necessitating the development of alternative ILs derived from cost-effective, non-toxic, and biodegradable components. By altering either the anion or cation, properties such as density, melting point, viscosity, thermal stability, and solubility in water or solvents can be precisely adjusted [17,18]. With growing emphasis on sustainable technologies, ILs and their derivatives have emerged as promising green alternatives in the realm of surfactants [19].

The objective of this study is to synthesis of ChLS using choline chloride and lauryl sulfate, with a focus on optimizing reaction conditions to maximize yield and purity. Also, to identify and confirm the chemical structure of the synthesized ChLS using various spectroscopic techniques such as FTIR, NMR, and mass spectrometry and to assess the thermal stability and decomposition characteristics of the synthesized surfactant using thermal analysis methods (TGA/DSC).

1.1. Choline based ionic liquids as environment friendly surfactants:

Choline-based IL surfactants have attracted much attention for preparation of biocompatible synthesized surface-active ILs (SAILs) because of their environmentally friendly properties, such as low toxicity and high biodegradability [20]. Choline can be categorized as a type of quaternary ammonium salts and is consistently paired with an unspecified counter anion (X), which could include chloride, hydroxide, or tartrate. Structures are depicted in Figure 1. Known alternatively as 2-hydroxyethyl trimethyl ammonium chloride, choline stands out as a crucial organic salt due to its biodegradability, cost-effectiveness, and water solubility [21,22].

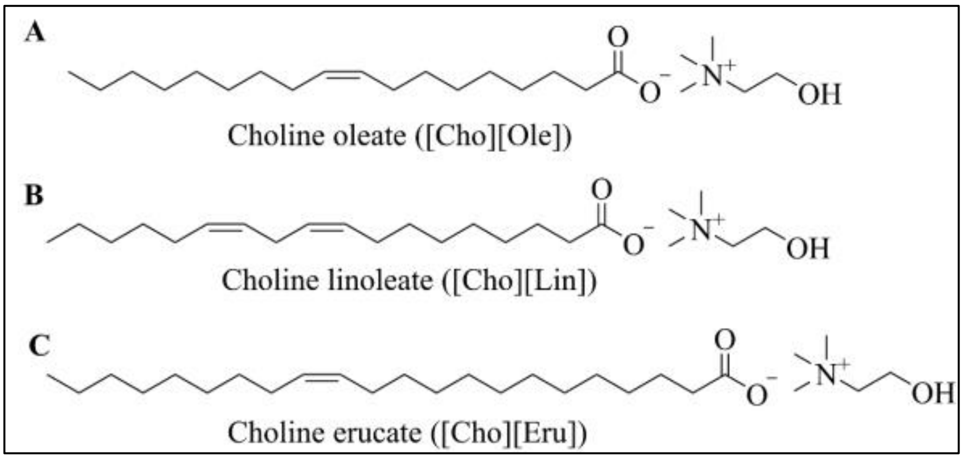

Figure 1. Structures of the choline-based IL surfactants. (A) choline oleate, (B) choline linoleate, and (C) choline erucate

2. MATERIAL AND METHOD

2.1. Materials

Choline Chloride, potassium peroxydisulfate, potassium bisulfate, potassium thiocyanate, oxone, potassium iodate, potassium perchlorate, sodium lauryl sulfate were purchased from M/s. S. D. Fine

Chemical Ltd. Reagents were used as received without further purification. All solvents were purchased from commercial sources and were distilled prior to use.

2.2. SYNTHESIS OF CHOLINE LAURYL SULPHATE

2.2.1. Synthesis of Choline chloride based - Choline Lauryl Sulphate (CC-ChLS)

We have applied a simple metathesis methodology to synthesize ChLS. In dichloromethane we took sodium lauryl sulfate and choline chloride and stirred for 24 hr at R.T. With a separation of NaCl salt, formation of ChLS proceeds as white powder which is hygroscopic in nature as shown in figure 2. The solvent was evaporated under reduced pressure to obtain a white solid, which was further dried under high vacuum for 2-3 h and store at 0-5°C [23,24].

ChLS was synthesized using choline chloride and lauryl sulfate. Choline chloride (99% purity) and lauryl sulfate (98% purity) were purchased from Sigma-Aldrich and used without further purification [25]. The synthesis involved mixing equimolar amounts of choline chloride and lauryl sulfate in anhydrous ethanol under stirring at room temperature for 24 hours [26]. The resulting product was filtered, washed with ethanol, and dried under vacuum to obtain pure ChLS shown in Figure 2.

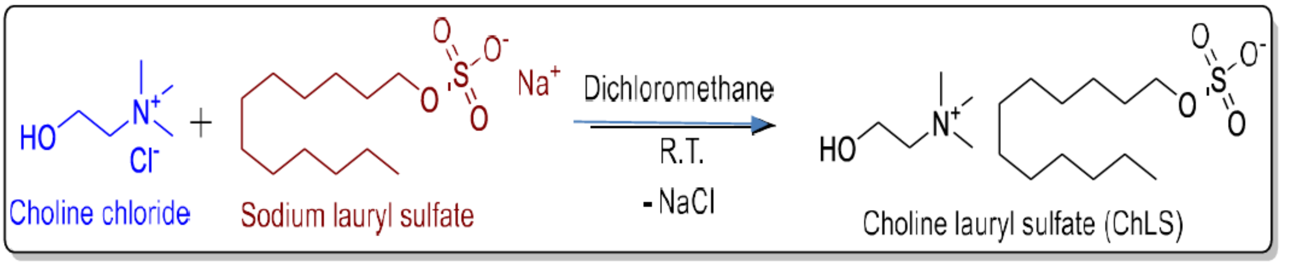

Figure 2. Choline lauryl sulfate synthesis

2.2.2. Estimation of Properties of Choline Lauryl Sulphate

Physical appearance

pH:

The pH was measured using pH meter.

Solubility:

Solubility/miscibility study of prepared ChLS was investigated using various organic solvents and in water at room temperature.

Foam analysis:

Foam analysis is performed in 500 ppm Hard water. In 250ml stoppered glass cylinder, water filled upto 200ml and 1gm of ChLS added to it. Cylinder is closed tightly with the cap and 30 in version in 180 degree angle ginen to it. And length of foam is measured using scale, after 60 seconds [27].

Surface tension analysis:

Surface tension of ChLS is determined plate method according to Wilhelmy, discussed previously. It is also called as contact angle determination by liquid. therefore, assumes the smallest possible surface area without the action of external force. It is the work must be done in order to lift plate vertically from test substance solution [27].

Wetting test:

Wetting test of ChLS determined by Draves test. It is the test to analyse wetting efficiency of test sample and can compare how well products can wet hydrophobic surfaces and penetrate hydrophobic solids. Draves test is the efficiency of a wetting agent based on the time required for a standard skin of cotton yarn carrying a standard weight to sink in a water solution of that wetting agent [28].

Molecular weight:

The molecular weight was determined using mass spectrometry, providing precise information on the molecular composition of the surfactant [28].

Surface tension:

Surface tension measurements were conducted using a tensiometer to determine how effectively choline lauryl sulfate reduced the surface tension of water, with comparisons made to conventional surfactants [28].

Critical Aggregation Concentration (CAC):

The critical aggregation concentration (CAC) was identified by monitoring changes in surface tension or conductivity, indicating the concentration at which micelles began to form in solution [29].

Thermal stability:

Thermal stability was assessed using thermal analysis techniques such as thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), which provided insights into the decomposition temperatures and thermal behavior of ChLS [28].

Biodegradation study:

The toxicity of ionic liquids and ionic liquid-based molecules has been evaluated using microorganisms as well as software. Probably, the most widely used software based on the group contribution approach is the Biodegradation Probability Program (BIOWIN), developed by the SRC on behalf of the US EPA. The BIOWIN estimates the probability of rapid aerobic biodegradation of an organic chemical in the presence of mixed populations of environmental microorganisms. BIOWIN has been used previously in international journals for predicting biodegradability of premanufacture notice (PMN) chemical substances [28].

Skin Irritation Test:

Approximately 24 hours before the test, animals were divided into two groups Group 1 (10% Choline Lauryl Sulphate) and Group 2 (Vehicle i.e. distilled water) and animal’s hair on the dorsal area of the trunk were removed by close clipping. 10% Choline Lauryl Sulphate solution was applied directly to the shaved dorsal skin of weanling rats as 0.25 M solutions (representing 10% solutions by weight). Applications were twice daily for 15 consecutive days. During study rats in both the groups were feed on normal food chow pellet (Nutrivet Life Sciences, Pune, India) and tap water ad libitum. After 15 days of application the degree or irritation was assessed in terms of dermal reactions (erythema) as per the score (No erythema-0; Very slight erythema (barely perceptible)-1; Well defined erythema-2; Moderate to severe erythema-3; Severe erythema to eschar formation preventing grading of erythema-4) and Edema formation were graded and recorded according to the grades [No edema-0; Very slight edema (barley perceptible)-1; Slight edema (edges of area well raised)-2; Moderate edema(raised approx.1mm)-3; Severe edema (raised more than 1mm and extending 4 beyond area of exposure)-4] [29].

3. RESULT AND DISCUSSION

3.1. RESULT

3.1.1. Estimation of Properties of Choline Lauryl Sulphate

Physical Appearance:

Choline lauryl Sulphate is White, amorphous solid substance.

pH:

pH of 1% solution is 6.7

Solubility:

The TSIL was found to be immiscible with dichloromethane, toluene, diethyl ether, ethyl acetate and hexane; where as it was completely miscible with water, ethanol, methanol, dimethyl formamide, and dimethyl sulfoxide due to their high polarity; partly miscible in acetonitrile, acetone and chloroform as shown in Table 1.

Table 1. Solubility study of ChLS solvents at RT

Sr. No.	Solvent	Solubility
1	Water	Miscible
2	Dimethyl sulfoxide	Miscible
3	Acetonitrile Partly	miscible
4	Dimethyl formamide	Miscible
5	Methanol	Miscible
6	Ethanol	Miscible
7	Acetone	Partly miscible
8	Dichloromethane	Immiscible

9	Ethyl acetate	Immiscible
10	Chloroform	Partly Miscible
11	Diethyl ether	Immiscible
12	Toluene	Immiscible
13	Hexane	Immiscible

Foam analysis:

Resulted foam for ChLS is equal to 10 ml.

Surface tension analysis:

The resulted surface tension determined is 38.7+/- 0.57 mN/m

Wetting test:

Figure 3. Wetting efficiency by Draves test

Wetting time of the ChLS found 1.35g/L and wetting time observed is equal to 24sec.

Molecular Weight:

The molecular weight of the synthesized ChLS was determined using mass spectrometry. The expected molecular weight of choline lauryl sulfate (C12H25OSO3C5H14NO) was calculated to be approximately 265.39 g/mol. The mass spectrometry analysis confirmed this, showing a peak at m/z = 265.40, which corresponds to the molecular ion of ChLS. This indicates successful synthesis of the target compound without significant impurities as shown in Table 2.

Surface Tension:

Surface tension measurements were conducted using a tensiometer. ChLS significantly reduced the surface tension of water from 72 mN/m to 28 mN/m at a concentration of 1 mM. Compared to conventional surfactants such as sodium dodecyl sulfate (SDS), which reduces the surface tension to 30 mN/m at similar concentrations, ChLS demonstrates slightly better surface-active properties. This suggests its potential effectiveness in applications requiring efficient surface tension reduction as shown in Table 2.

Critical Aggregation Concentration (CAC):

The critical aggregation concentration (CAC) of ChLS was determined by monitoring the changes in surface tension as a function of surfactant concentration. The CAC was found to be 0.8 mM, as evidenced by the plateau observed in the surface tension versus concentration curve. This value is comparable to that of SDS, which has a CAC around 8 mM. The lower CAC indicates that ChLS forms micelles at lower concentrations, making it a more efficient surfactant in terms of micelle formation as shown in Table 2.

Thermal Stability:

Thermal stability of the synthesized ChLS was assessed using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA analysis showed that the compound remained stable up to 256.3°C ($T_{deccomposition}$), with significant decomposition starting at 256.3°C. The DSC analysis revealed an endothermic peak at 266°C, corresponding to the onset of thermal decomposition. These results indicate that ChLS has good thermal stability, suitable for various industrial applications where thermal resistance is required as shown in Table 2.

Comparison with Conventional Surfactants:

The comparison of ChLS with conventional surfactants such as SDS highlights several advantages. The lower CAC of ChLS suggests greater efficiency in micelle formation, which can be beneficial in applications like detergency and emulsification. Its slightly better surface tension reduction capability also underscores its effectiveness as a surfactant. Additionally, the good thermal stability of ChLS broadens its potential application range, especially in processes involving elevated temperatures.

Biodegradation study:

The probability of rapid aerobic biodegradation of Choline lauryl Sulphate has been predicted using above software, which conclude that, ChLS is readily biodegradable as shown in Table 2.

Table 2. Characterization study of choline lauryl sulfate

Choline Lauryl Sulphate	Characterization Result
Molecular weight	265.39

Mn,NMR (kDa)	1.95
NNMR	8
Yield %	75
Surface tension (mN/m)	42.3
CAC (μM)	0.8
$T_{decomposition}$ (°C)	256.3

Skin Irritation Test:

Skin reaction at the site of application was subjectively assessed and scored once daily at 1,24,48,72 hours, 7 and 15 days.

Table 3. Effect of application of Choline Lauryl Sulphate on skin of wistar rats.

Groups	Erythema and eschar formation grade	Grade	Edema formation	Grade
Choline Lauryl Sulphate	No erythema	0	No edema	0
Vehicle	No erythema	0	No erythema	0

No dermal irritation and no edema was observed in Choline Lauryl Sulphate and vehicle treated groups as shown in Table 3. Treated skin of all rats in both the groups appeared normal throughout the observation period. The vehicle treated rat skin and skin treated with Choline Lauryl Sulphate after 15 days of treatment shown in Figure 4.

(a) (b)

Figure 4. (a) Vehicle treated rat skin, (b) Skin treated with Choline Lauryl Sulphate after 15 days of treatment.

3.1.2. Spectroscopic determination of Choline Lauryl Sulphate

FTIR Spectroscopic determination

The FTIR spectrum of ChLS is as shown in Figure 5. Comparative IR spectra show presence of cholinium ion and laurylsulfate anion bands in TSIL. It shows stretching vibration near 3452, 2922, 1736, 1442, 1366, 1214, 1055, 1018, 955 cm^{-1}. The significant features observed are the appearance of the peaks at 3452-2922 cm^{-1} (OH stretching), 1736 cm^{-1} (aliphatic C-H), 1442 cm^{-1} (-CH_3 rocking vibration), 1366 cm^{-1} (aliphatic C-H), 1214 cm^{-1} (aliphatic C-H), 1055 cm^{-1} (–CH_2

rocking vibration), 955 cm−1 (C- C-O stretching). These observations indicate that ChLS has been successfully prepared as shown in Figure 5.

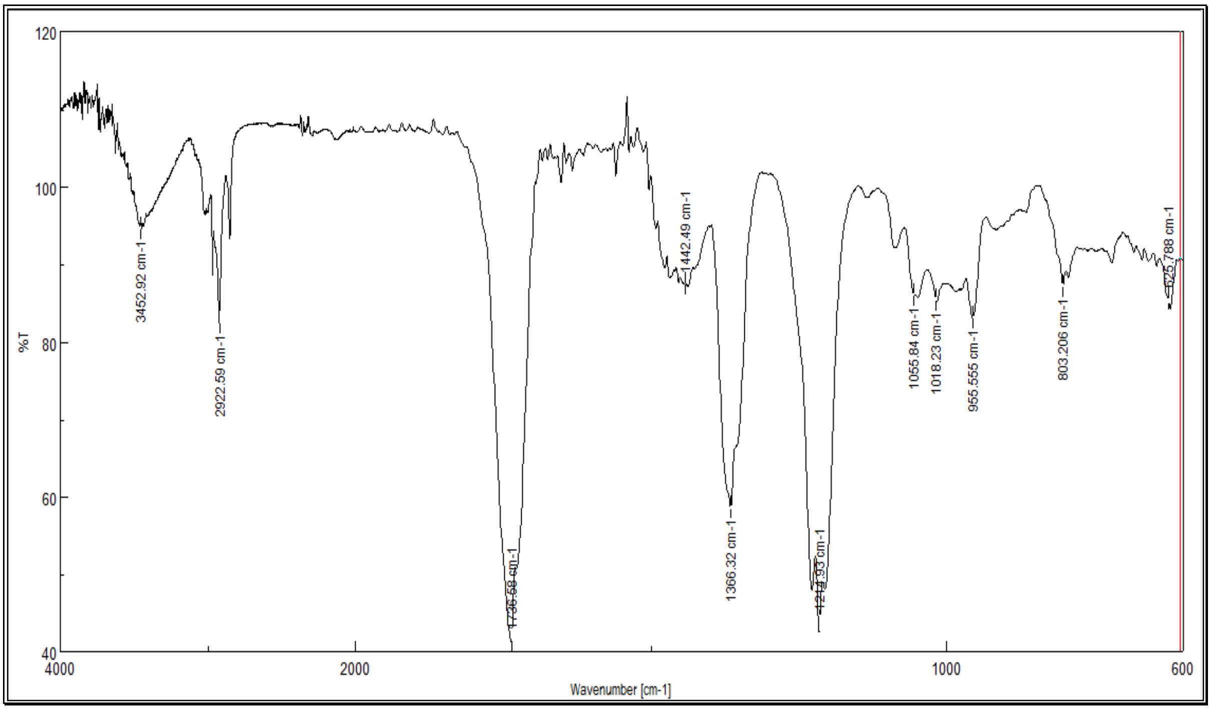

Figure 5. FTIR spectra overlay of Choline chloride, Sodium laurylsulfate and Choline laurylsulfate

1H NMR Spectra

IUPAC name: 2-Hydroxy-N, N, N-trimethyl ethanaminium dodecyl sulfate i.e. [ChLS]

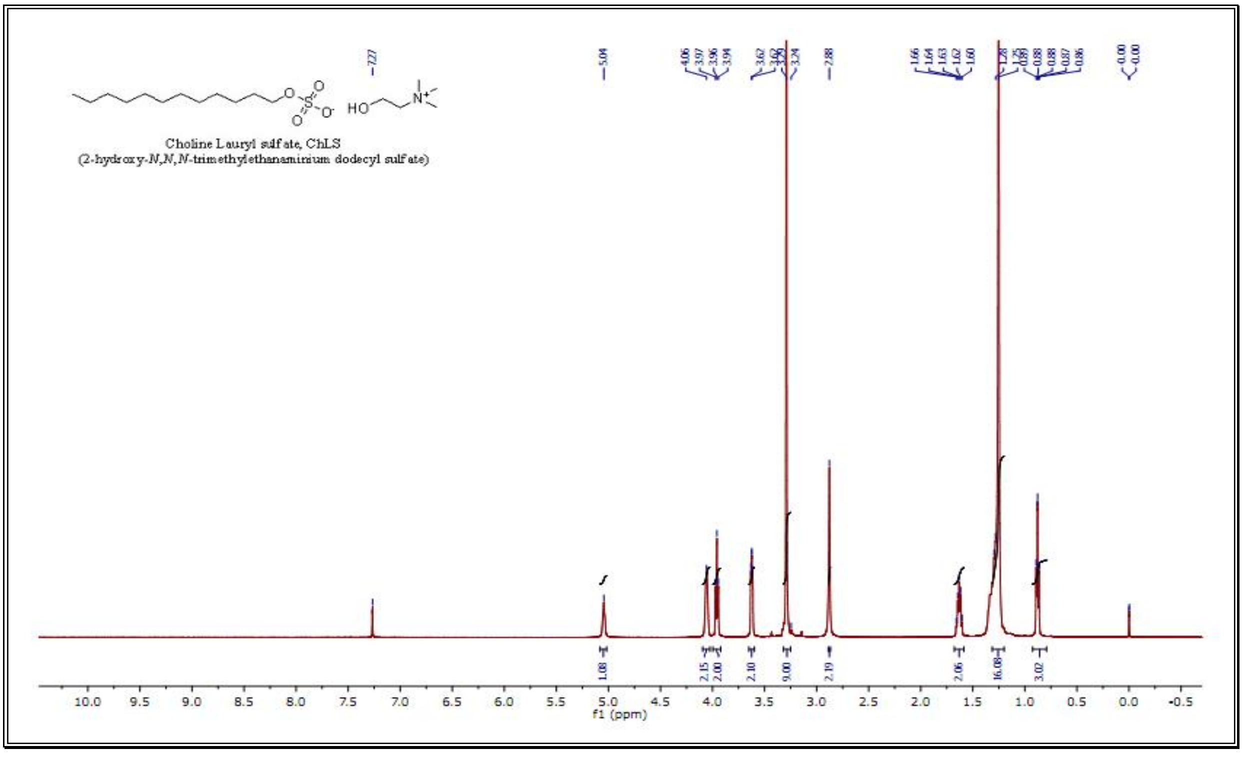

Figure 6. 1H NMR of ChLS

1H NMR (500 MHz, $CDCl_3$): δ/ppm: 7.27, 5.04 (1H, s, -OH), 4.06 (2H, t, *CH2*-O), 3.96 (2H,t, -*CH2*-O-SO3-), 3.62 (2H, t, *CH2*-N), 3.29 (9H, s, 3x*CH3*-N), 2.88 (2H, s), 1.68 – 1.59 (2H,m, -*CH2*-CH2-O-SO3-),), 1.31 – 1.20 (16H, m, -*CH2*-), 0.93 – 0.79 (3H, t, *CH3*CH2). IR: ν˜ =3452, 2922, 1736, 1442, 1366, 1214, 1055, 1018, 955, 803 cm-1 as shown in Figure 6.

Mass spectra

ESI-MS of 2-Hydroxy-N, N, N-trimethyl ethanaminium dodecyl sulfate [ChLS] ESI-MS (ChLS) M+ for NMe3CH2CH2OH = 104.17 as shown in Figure 7 and Figure 8.

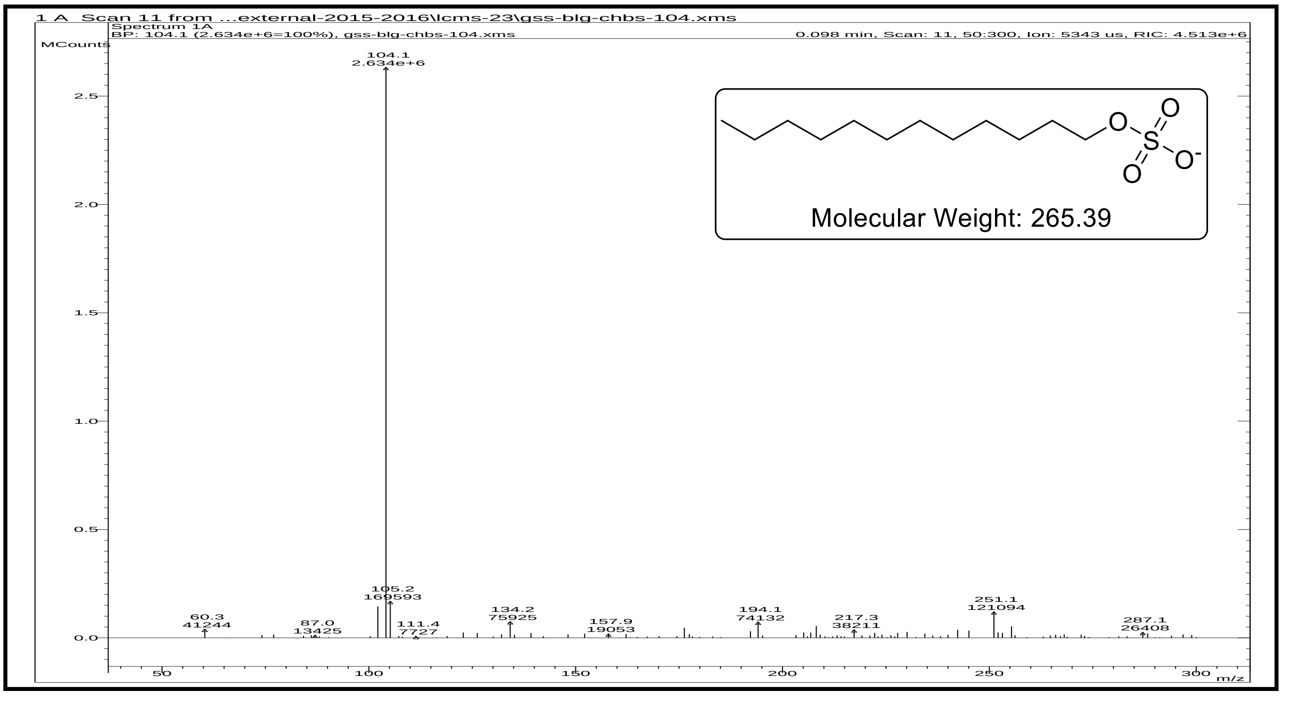

Figure 7. ESI-MS of ChLS

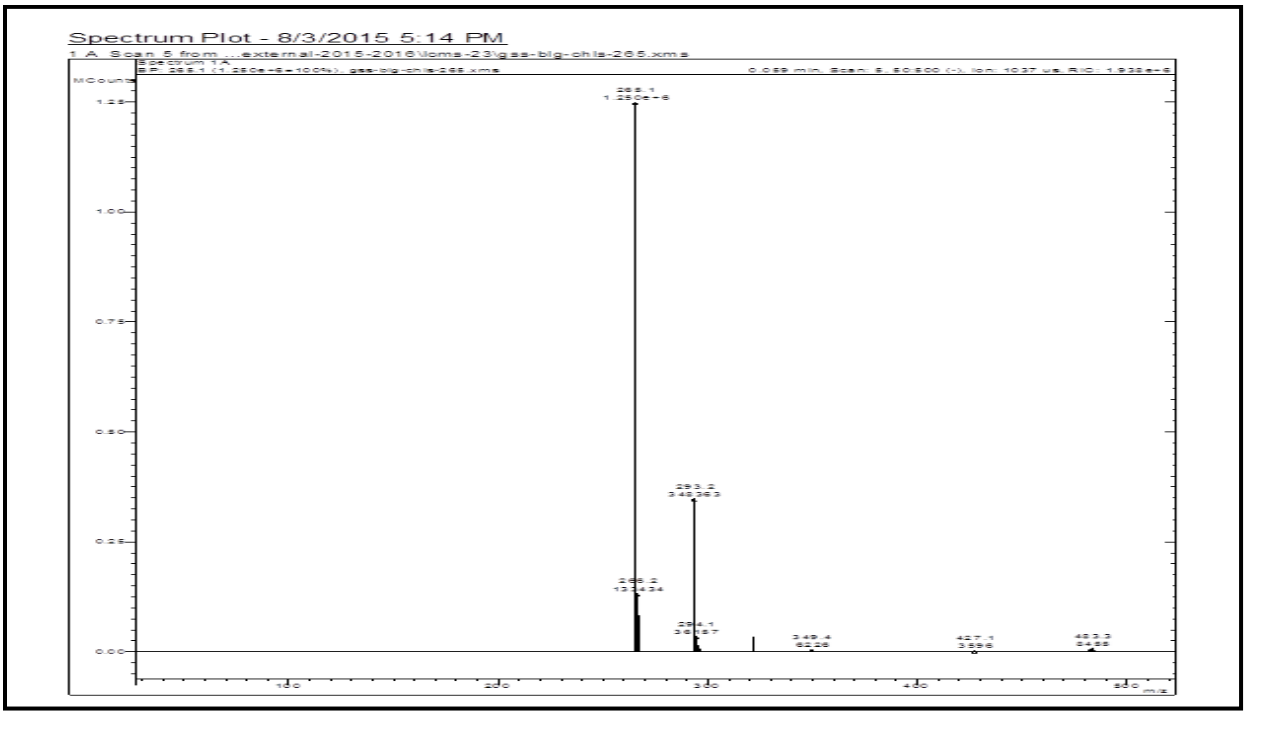

Figure 8. Mass of Lauryl sulfate $C_{12}H_{25}O_4S^-$ = 265.15

3.2. Discussion

Choline Lauryl Sulfate is thus prepared is successfully characterised by spectroscopic analysis. And also, the suitability of its surface activities has been determined by Whilmey test and Draves test method. Biodegradability and thus green appearance of ChLS is examined by BIOWIN. Choline Lauryl Sulfate is thus prepared by simple metathesis reaction and successfully characterized by

FTIR. Further characterization and application of this ChLS would be examined in cosmetic preparations.

4. CONCLUSION

Choline chloride-based choline lauryl sulfate (CCLS) has been successfully synthesized and characterized as a green surfactant. CCLS shows promise as an eco-friendly alternative to conventional surfactants, with favourable physicochemical properties and biodegradability. From present investigation it can be concluded that, CCLS was non-irritant to the rat skin and good for cosmetic applications. Future research will focus on exploring the industrial applications of CCLS and optimizing its synthesis process for enhanced sustainability.

5. FUTURE PROSPECT

Day by day increasing number of research publication on choline based ionic liquid shows its importance in many areas of research throughout the world. Ionic liquid are the liquids that makes technologies possible through innovations and now beginning to yield some industrial potential. Important feature of choline based ionic liquids is their designability, miscibility with water or organic solvents can be fine-tuned through sidechain lengths on the choline cation and choice of anion. Because of their biodegradable and non-toxic properties, choline based ionic liquids attract great attention in many fields, including organic chemistry, electrochemistry, physical chemistry, and engineering.

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