

Synthesis and Characterization of Imidazole-Based Cationic Surfactants as Disinfectants

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Abstract: Citric acid-based cationic surfactants using 1-methyl imidazolium as quaternisation agent have been synthesized and characterized. The cationic surfactants are produced in excellent yields (70%) and have been examined by TGA, FTIR, ¹H NMR and are found to have good surface-active properties. FTIR analysis showed the presence of ester confirmed by ¹H NMR (the chemical shift value of 3.7 ppm and 3.8 ppm is due to the presence of an ester) in the surfactant. The biodegradability was confirmed by BOD analysis. It is found that cationic imidazolium surfactants have surface tension of (12 mN/m) with chloride as a counter ion. The results show that cationic imidazolium surfactants with longer hydrophobic chains have a lower CMC value. TGA result shows good thermal properties of cationic surfactant. In vitro study of cationic surfactant over fungal cell indicates its antifungal activity (70% inhibition at 350 ppm) and its application as disinfectant.

Keywords: Cationic Surfactants; imidazolium; Citric acid, surface tension; disinfectant; In vitro.

INTRODUCTION

The focus of recent developments in the field of science is the production of renewable, environmentally friendly products¹⁻⁵. The ever increasing demand for surfactants has facilitated the development of such surfactants, which can be biodegraded after use^{6,7}. The raw materials used to manufacture such surfactants include oleo-chemicals,^{8,9} carbohydrates,^{10,11} amino acids^{12,13}, which are derived from natural sources only. These raw materials only give carbon dioxide as the end product after use as surfactant; hence, CO₂ can be eventually used by plants to manufacture food². Many surfactants have been developed on the lines of renewable products with sustainable biological and surface properties other than the conventionally used surfactants¹⁻³. Among the commercially used surfactants, Pyridinium and imidazolium attract attention due to their use in the cosmetic industry⁴⁻⁶. They also find their applications in textile and mineral processing⁹⁻¹¹, polymerization of emulsions, and curbing corrosion^{7,8}. The biological application of these surface-active agents includes antimicrobial activity and their specific role in drugs and gene delivery agents¹²⁻¹⁵. Every year, an increase in annual growth rate by 3-4% has been reported for the use of surfactants in the industry. However, petrochemical-based surfactants are still a feasible choice because of their low cost, easy manufacturing, and better physical and chemical properties¹.

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Biodegradable and renewable surfactants have found a wide range of applications in today's modern lifestyle. They are better suited for their eco-friendly nature compared to petrochemicals and commercially available surfactants. There are still certain issues which need to be resolved. These include ease of manufacturing, cost, and related environmental issues^{14,15}. Recent report shows the synthesis of new cationic surfactants based on oleic and stearic acid. Introduction of ester linkages into surfactants results in the biodegradation of surfactants^{15,16}.

This study reports the new citric acid-based cationic surfactant. Recent studies show the synthesis of new cationic surfactants based on oleic and stearic acid.^{15, 16-20} Renewable fatty acid-based surfactants, due to their biodegradability, natural abundance and non-toxicity, renewable fatty acid-based surfactants, are becoming popular with rising demands¹⁷⁻²⁵. The objective of this study was to develop a new surfactant based on natural products and minimize the number of steps to synthesize cationic surfactants using citric acid precursor. The synthesized surfactant possesses properties which can be altered by either changing the hydrophobic moiety or by changing the link between hydrophilic and hydrophobic parts. There are many methods which have been practiced for the synthesis of cationic-based surfactant²⁰⁻³⁰. However, the choice of a method is determined with the purpose of synthesized surfactant being biodegradable, cheap, and renewable. Cationic surfactants are the current research topic because of their broader applicability and synthesis process using renewable fatty acids. Cationic surfactants contain positively charged head parts. They are used in detergents and fabric softeners to provide softness to the fabric. Due to their good cleansing properties, they are also used in disinfectants and many household cleansers. Beside this, cationic surfactants were also synthesized by the use of different long chain fatty acids such as citric acid, Cinnamic acid, Tartaric acid, 2-Chlorobenzoic acid, with halo alcohol such as 2-chloroethanol and propanol and by adding few drops of sulphuric acid and then ester which is formed then reacted with 1-methyl imidazole. Their identifications are based on Infrared Spectroscopy (IR), Proton Nuclear Magnetic Resonance Spectroscopy (¹H NMR), and mass spectral studies. Surface tension measurement has also studied the surface activity of these surfactants in the presence of sodium halides (NaCl and NaBr).

MATERIAL AND METHOD

Chloroethanol (AR grade), bromoethanol (AR grade) and 1-methyl imidazole (AR grade) were purchased from Sigma Aldrich. Sulphuric acid (AR grade) was purchased from Merck, India. Deionized water from prepared using sigma Aldrich instrument. The citric acid (LR grade) was purchased from Merck, India.

CHARACTERIZATION

IR spectra were recorded as a thin film on KBr Pellet on a Shimadzu 8400s FT-IR (Kyoto, Japan) instrument.

¹³C NMR spectra were recorded on a JEOL AL-300 (JEOL, Japan) system as a solution in CDCl₃, using tetramethyl silane (TMS) as an internal standard.

The thermal stability of surfactant was measured by using PerkinElmer thermogravimetric analyzer (TGA) with a temperature ramping of 10 °C/min in an air atmosphere.

SYNTHESIS OF SURFACTANT (CITRIC ACID -2 CHLOROTKA-E):-

Synthesis of these ester-based cationic surfactants from fatty acids was reported previously [2, 3,15]. We report the preparation of Citric Acid -2 CHLOROTKA-E surfactant with excellent yield by using citric acid in place of fatty acid followed by quaternization with 1-methyl imidazole [2]. The citric acid (0.1mol)- (0.210g) was taken and added to the (0.1 mol) 2-chloroethanol with the addition of 2-3 drops of sulphuric acid as described in figure 1. Then stirred this content for 72 hours at 60-70 °C. Then washed the mixture with 50 ml of chloroform and water 2-3 times. Chloroform was removed from the mixture under reduced pressure in a rotatory flash evaporator at 40 °C. The product formed was then taken into the separating funnel and 10 ml of methanol was added with 2-3 drops of water which helps settle the product at the bottom in its purest form and collected gently. It was observed that almost 60% of the surfactant in the form of a semi-solid paste was recovered from the bottom of the column (separating funnel). The resulting ester which is formed was then reacted with 1-methyl imidazole in a 1:1 molar ratio (0.01 mol) at 80 °C for 2 hours. The resulting product was crystallized and re-crystallized with acetone to get pure surfactant which was characterized by IR, TGA, and NMR spectroscopy.

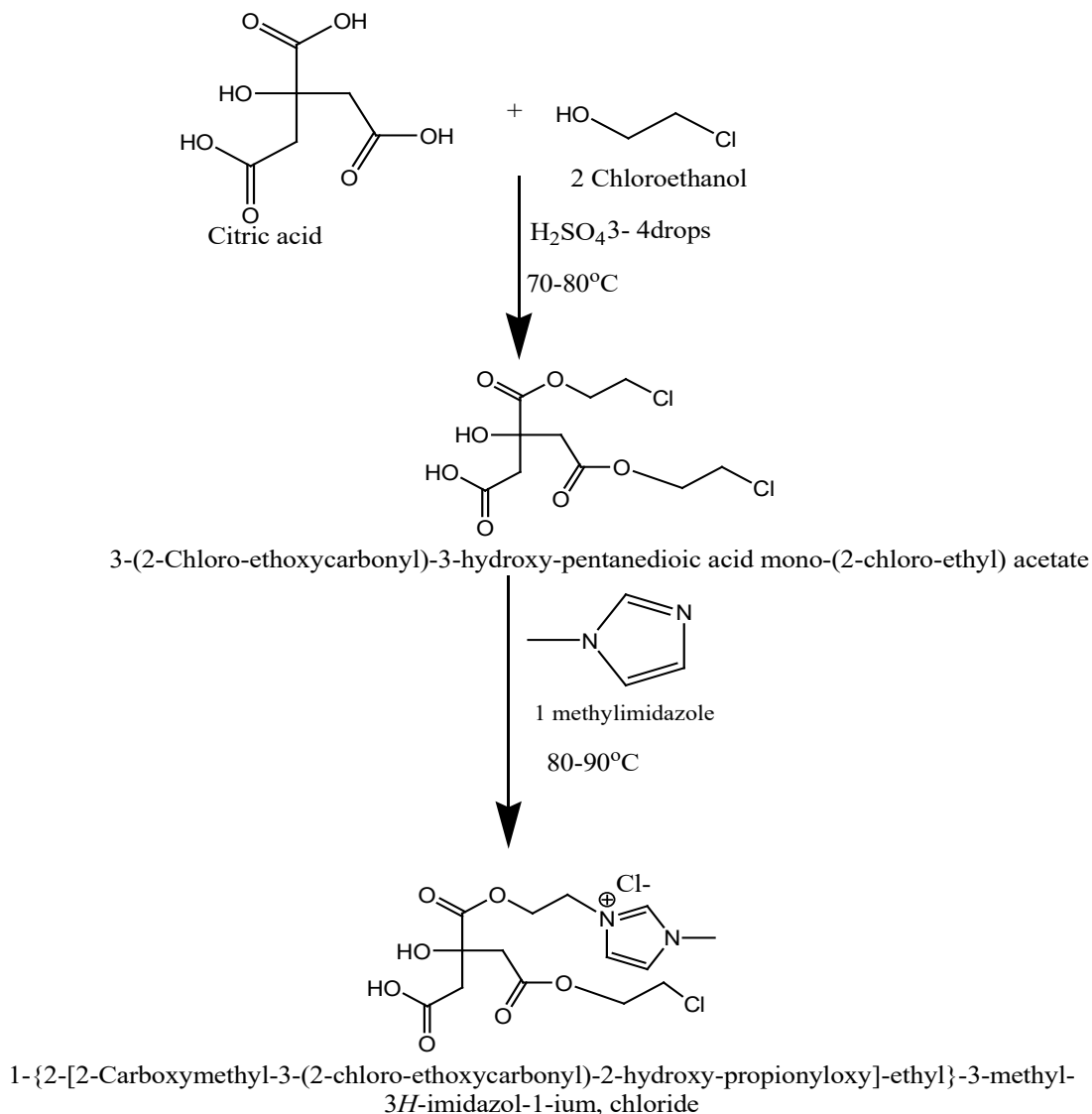


Figure 1. Synthesis of cationic surfactant from citric acid using chloroethanol and 1-methyl imidazole.

RESULTS AND DISCUSSION

TGA determined the thermal decomposition curve of synthesized Citric Acid -2 CHLOROTKA-E cationic surfactant in figure 2. Three endothermic peaks were observed at 110, 220 and 320 °C. We can also see the start and onset temperature in figure 2 above. TGA analysis shows that citric acid CHLOROTKA-E imidazole-based cationic surfactant is stable up to 400 °C. The start temperature (T start) of this surfactant is 200 °C, the temperature at which the sample decomposition begins.

(TG(mg) \approx T °C = 10 mg \approx 130 °C, 5 mg \approx 250 °C, 1.8 mg \approx 430 °C).

Thermal stability of Citric Acid -2 CHLOROTKA-E cationic surfactant was determined by Thermal Gravimetric analysis (TGA) in which a change in the weight of a cationic surfactant was recorded as a function of temperature as a derivative thermo gravimetric curve (DTG) where the first derivative of the TG curve was plotted concerning time as shown in figure 2. Three declined points (A, B, C) were observed at a temperature approximately equal to 130, 250 and 430 °C (TG(mg) \approx T °C = 10 mg \approx 130 °C, 5.5 mg \approx 250 °C, 1.8 mg \approx 430 °C). The curved portion AB and BC indicated that there are changes in the slope of the weight loss curve. The intermediate was formed at the region AB of

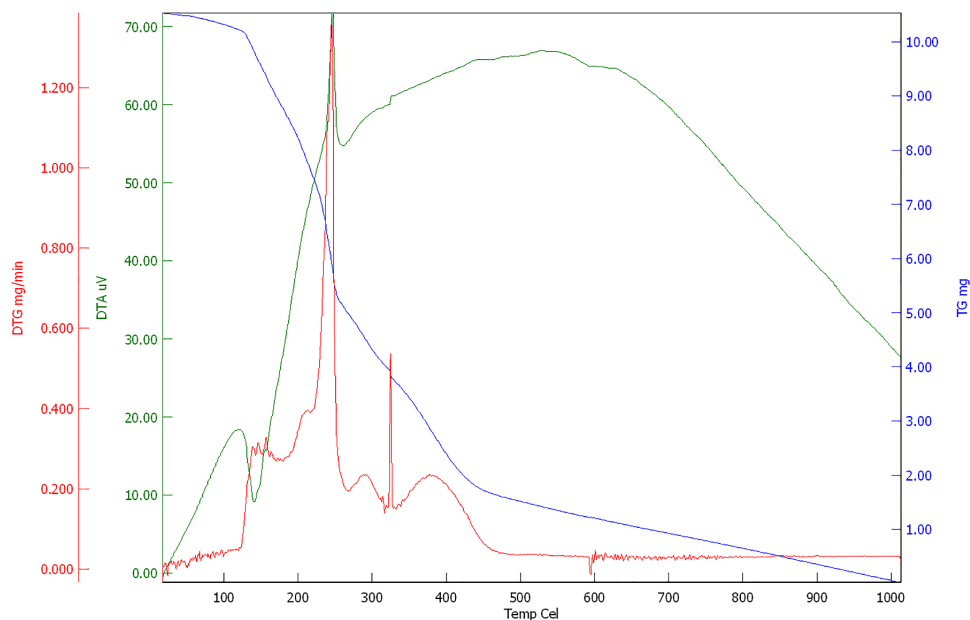


Figure 2. TGA and DTA analysis of Citric Acid (-2 CHLOROTKA-E) cationic surfactant using 1-methyl imidazole.

the derivative curve. We can also see the start and onset temperature in figure 2 above. TGA analysis shows that citric acid (-2 CHLOROTKA-E) imidazole-based cationic surfactant is stable after 430 °C. The start temperature (T_{start}) of this surfactant is 200 °C, the temperature at which the sample decomposition begins.

The rate of change of the DTG curve was obtained. In the DTG curve, the inflexion point was observed at a temperature corresponding to 250 °C, where the peak on the derivative curve corresponds to a maximum slope on the TG curve. i.e. $dW/dt = \text{maximum}$. When the temperature reaches 500 °C to 600 °C, the peak on the derivative curve corresponds to a zero slope. i.e. $dW/dt = 0$.

Proton NMR study helps us to understand the structure of synthesized imidazolium-based cationic surfactant. Proton (^1H) NMR is a powerful technique for studying the aggregation of surfactants. Figure 3 demonstrates the ^1H NMR spectra of Citric Acid (-2 CLOROKOTA-E) cationic surfactant using 1- methyl imidazole. Broad singlets are observed at 1.22-1.26 ppm, accountable for methylene protons of the chain. Another type of triplet is observed at 4.21 ppm due to a methylene proton of the imidazolium moiety. A triplet is observed at 5.67 due to two methylene protons of N^+CH_2 (imidazolium group).

In Figure 3, the chemical shift value of around 6.69 ppm is due to vinyl or conjugated proton of $\text{C}=\text{C}-\text{H}$. In Figure 4, the chemical shift value of 3.7

ppm and 3.8 ppm is due to the presence of an ester in the surfactant. In Figure 3, the chemical shift value of 2.5 ppm is due to the proton NMR signals of an acidic group-COOH present.

The FTIR spectrum of citric acid imidazolium is represented in figure 4. The peak around 2885 cm^{-1} corresponds to medium C-H stretching vibrations of alkyl groups present in an alkane. The N-H stretching vibrations are confirmed by the appearance of a peak around 2810 cm^{-1} , while 2221 cm^{-1} is due to C triple bond N ($\text{N}\equiv\text{C}$) stretching corresponding to the nitrile group. The carboxylic group is detected due to a peak around 1431 cm^{-1} due to medium O-H bending. Peak around 1356 cm^{-1} is due to medium O-H bending. The peak around 1185 cm^{-1} and 1066 cm^{-1} corresponds to Strong C-O stretching of an ester and primary alcohol, respectively. The region around 805 cm^{-1} corresponds to Medium C=C bending vibrations of alkene. The region around 400 cm^{-1} to 1500 cm^{-1} is the Fingerprint region.³⁵⁻⁴⁰ The fingerprint region is characterized by the presence of a large number of peaks which are unique and hence help in identifying the given compound from other compounds. The IR spectra of the cationic imidazolium surfactants showed the absorption bands in the region at 2885 cm^{-1} , indicating the presence of methylene groups. The absorptions at 1670 cm^{-1} indicate the presence of $\text{C}=\text{N}$. The band at 1570 cm^{-1} very well defines the presence of aromatic $\text{C}=\text{C}$ of all products.⁴⁷⁻⁵⁰

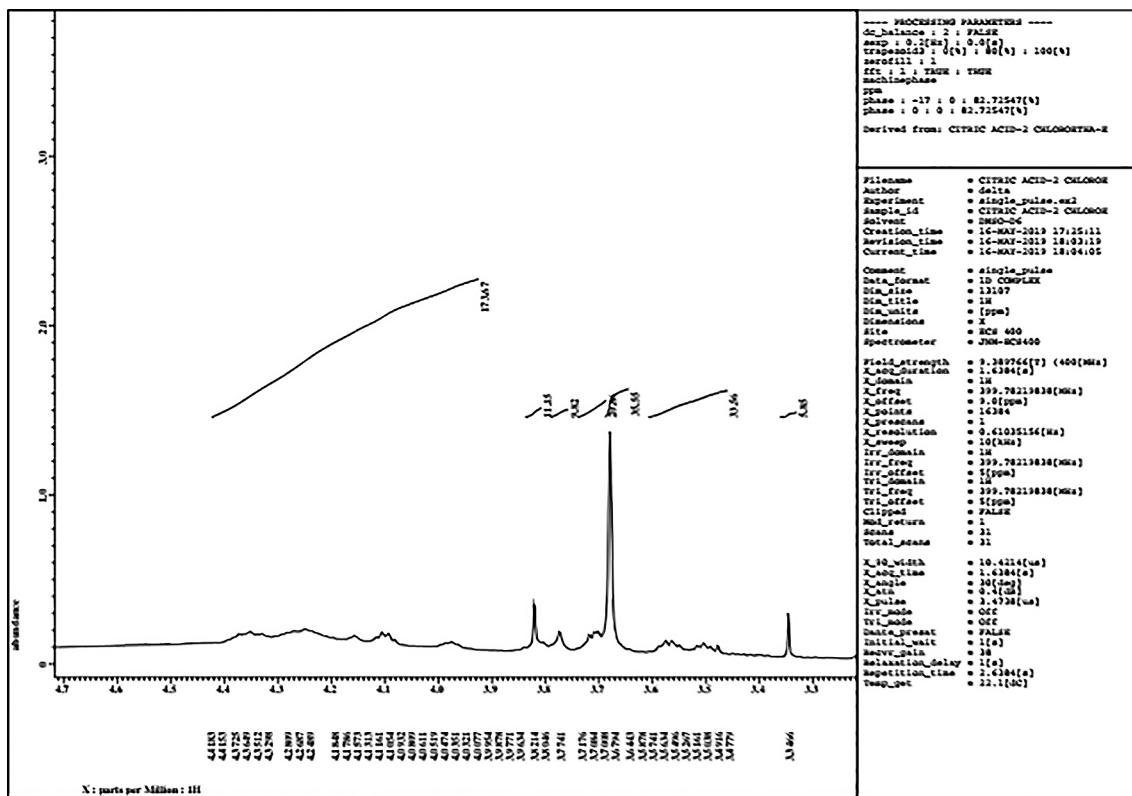
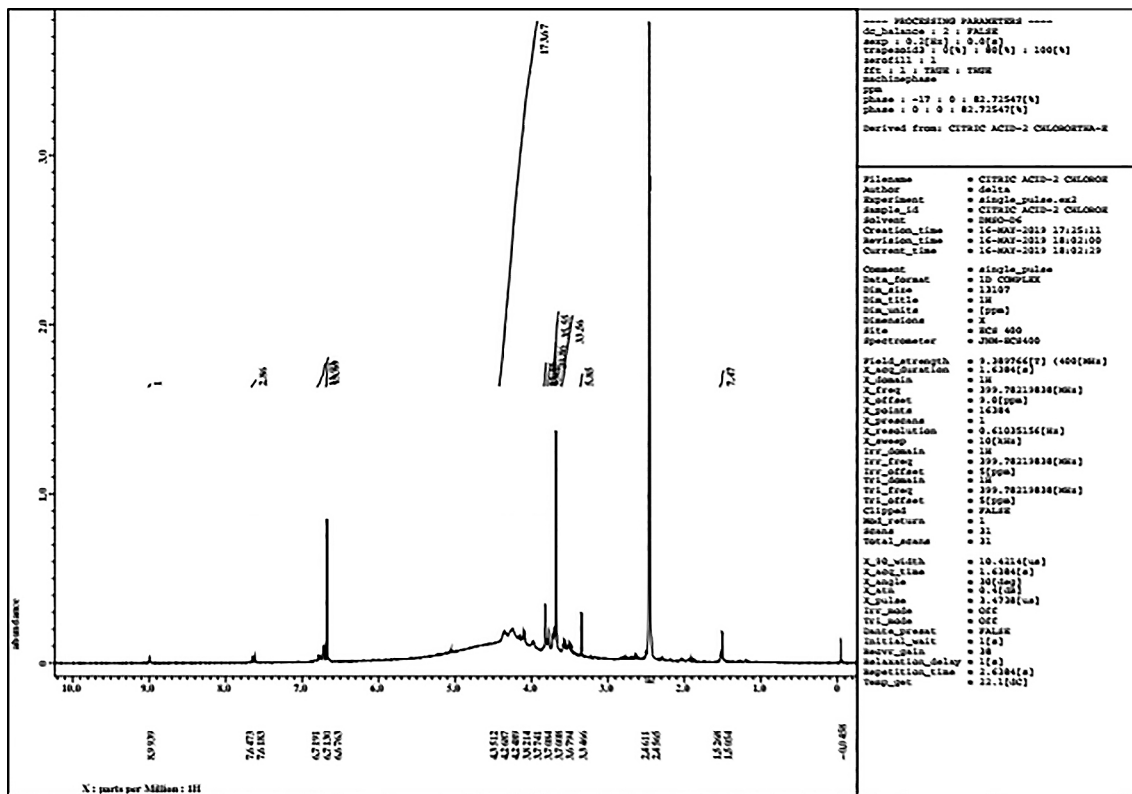


Figure 3. ¹H NMR spectra of synthesized Citric Acid -2 CHLOROTKA-E) cationic surfactant using 1-methyl imidazole.

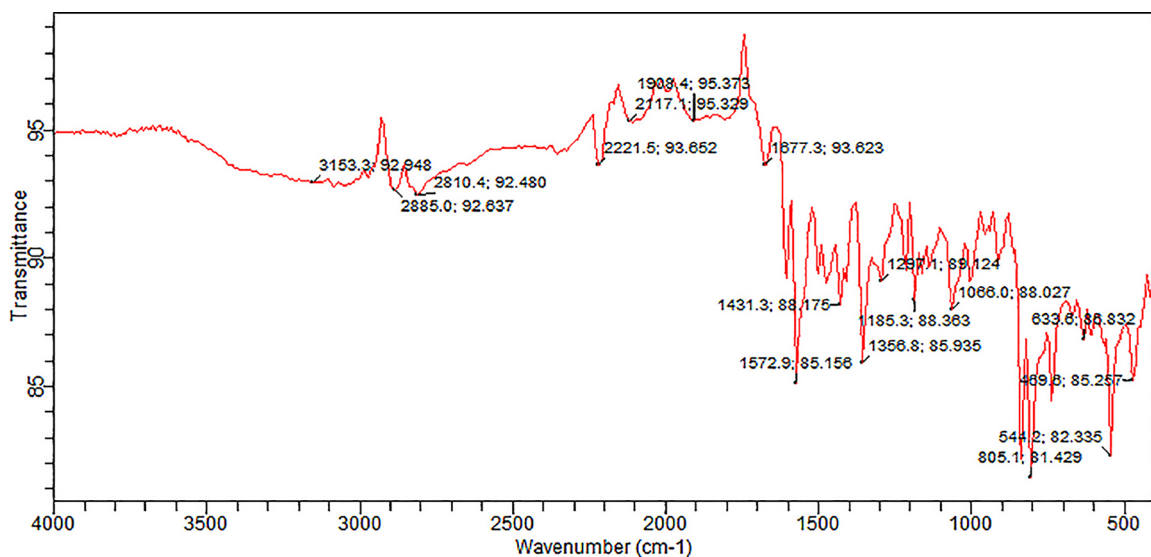


Figure 4. FTIR spectra of citric acid imidazolium based cationic surfactant.

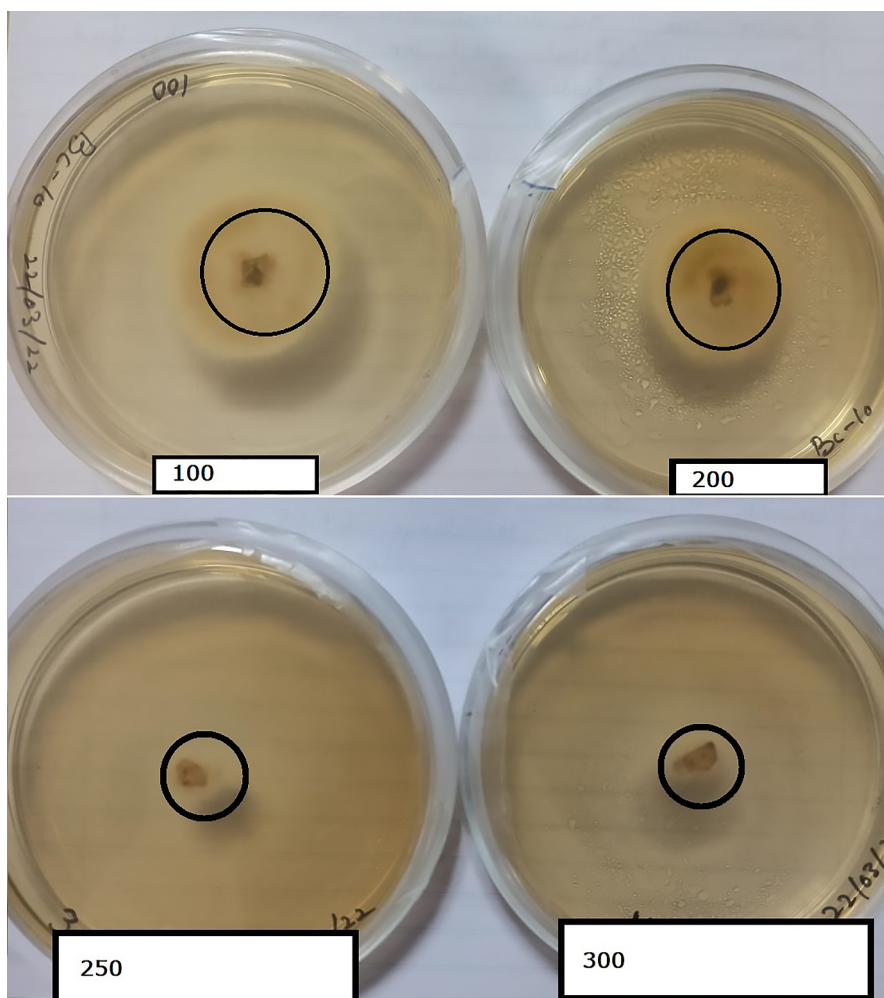


Figure 5. In vitro study of imidazole based citric acid cationic surfactant over bacteria (*Bacillus* cell) at 100, 200, 200, 350 ppm.

The Invitro study was conducted to understand the antibacterial activity of the synthesis surfactant using potassium dextrose agar media over Bacillus bacteria with different concentrations (100 to 350 ppm) of surfactant. The result (Figure 5) shows 35%

inhibition over a fungal cell of 100 ppm concentration and maximum inhibition (70%) using this cationic surfactant. These results confirm that the surfactant has antibacterial and can be used as a disinfectant for cleaning purposes.

Sample	Surface tension (mN/m)	Surface area (m ² /g)	Appearance	Density (g/cm ³) at 25°C
[C10MIm]Br ³⁰	14.51	29.30	Solid	1
[C12MIm]Br ³⁰	0.42	30.12	Solid	—
[C16MIm]Br ³⁰	0.77	34.30	Solid	2
[C10OCOCH ₂ MIm]Br ²⁶	0.76	26.12	Liquid	2.4
[C12OCOCH ₂ MIm]Br ²⁶	0.23	35.4	Liquid	2.4
CTAB (Commercial)	0.95	51	Solid	1
Citric Acid (-2 CHLOROTKA-E)	12	24	Semi-solid	1.4

Table 1. Surface properties of the synthesized imidazole-based cationic surfactant and their comparison with various reported imidazole-based surfactants.²⁶⁻³⁰

Table 1 shows two synthesized cationic surfactant surface properties using 1-methyl imidazole. Their properties compared with the reported imidazole-based surfactant such as commercial benchmark CTAB. The surface tension (12 mN/m for citric acid-based) of cationic synthesized surfactant is higher than the reported surfactant shown in table 1.

CONCLUSIONS

The citric acid, chloroethanol and 1-methyl imidazole were used as a precursor during surfactant synthesis at moderate temperature. The synthesis process followed the quaternization by 1-methyl imidazolium. The synthesized cationic surfactant showed biodegradability and thermal stability. The chemical and physical properties of synthesized cationic surfactants are comparable with the commercial cationic surfactants (CTAB). Therefore, it might be used as a soft template in heterogeneous catalysts synthesis. The synthesized surfactant is associated with water insolubility as a significant limitation. The natural fatty acid (castor oil) can be used as a precursor instead of citric acid for future research perspective as a cationic surfactant.

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