

Synthesis and Characterization of Caprolactam-based Ionic Liquids as Green Solvents

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Authors' contributions

This work was carried out in collaboration among all authors. Authors RAN and FOK designed the study. Author RAN collected the samples and performed the analyses and the literature searches. Authors FOK, KHK and YAR supervised the study and provided technical support. Author RAN wrote the first draft of the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Aims: To synthesize and characterize six caprolactam-based ionic liquids (CPILs) by combination of caprolactam with different organic and inorganic Brønsted acids that can be utilized for lipid extraction from microalgae.

Study design: Experimental design include quantitative and qualitative.

Place and duration of study: The study was done at Department of Chemistry & Biochemistry, School of Sciences and Aerospace Studies, Moi University (Kenya) between November 2020 and May 2021.

Methodology: Six CPILs were prepared through a simple neutralization reaction between Caprolactam and Brønsted acids such as Hydrochloric acid (HCl), Methane sulphonic acid

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(CH₃SO₃H), Trifluoromethanesulphonic acid (CF₃SO₃H), Acetic acid (CH₃CO₂H), Trifluoroacetic acid (CF₃CO₂H), and Sulfuric acid (H₂SO₄). The first three acids were used in the synthesis of CPILs for the first time. The chemical structures of the synthesized CPILs were characterized by Fourier transform infrared and Raman spectroscopy. The densities and viscosities were measured at 20 °C using the weight (pycnometer) and capillary viscometer (Oswald) methods, respectively.

Results: All the CPILs were insoluble in hexane and had high miscibility with water and methanol. Fourier transform infrared and Raman spectra of the CPILs were compared with that of free Caprolactam. The characteristic absorption bands of the synthesized compounds showed a big shift in position and/or intensity (compared to caprolactam), indicating the formation of the CPILs. The results showed that both the density and viscosity increased with the molecular weight of the anion - except in Caprolactamium hydrogen sulphate (CPSA)- which could be due to the strong interactions between the cation and anion resulting from the dimerization between hydrogen sulphate anions.

Conclusion: The hydrophilic nature of the CPILs indicated by high miscibility with polar solvents (water and methanol) indicates that they are suitable for the dissolution of cellulose of microalgae cell wall and thus could result in high lipid extraction efficiency. Further studies should therefore utilize the synthesized CPILs in lipid extraction from microalgae.

Keywords: Caprolactam; ionic liquids; green solvents; FTIR; raman; viscosity; density.

1. INTRODUCTION

Ionic liquids (ILs) are a group of compounds that have been known for more than a century and have attracted substantial attention in science and technology in the last two decades. Ionic liquids are organic salts that melt below 100°C (some of them are liquids at room temperature). Ionic liquids are composed entirely of ions [1]: a cation which is normally a bulky organic structure with low symmetry (such as imidazolium, pyridinium, ammonium and phosphonium), and an anion which could be organic or inorganic (such as a halogen, tetrafluoroborate and hexafluorophosphate). The forces operating between the cation and anion of an ionic liquid are hydrogen bonds, coulombic interactions, and weak dispersion forces [2]. There are three basic chemical reactions for synthesis of ILs, namely metathesis reaction, reaction of imidazole carbenes and Acid-base neutralization, which is very simple and inexpensive [3]. Ionic liquids possess several superior properties including extremely low vapor pressure, non-flammability, excellent thermal and chemical stabilities, catalytic properties, good solvating potential for a wide range of substrates, and potential recoverability [4].

Ionic liquids are also referred to as “designer solvents” because their polarity and hydrophilicity/hydrophobicity can be tuned by an appropriate combination of cation and anion [5]. The anion is currently utilized to control the water solubility, but the cation can also influence the hydrophobicity or hydrogen bonding ability [6].

Therefore, ILs are utilized in a variety of applications such as extractions, separation processes [7,8] and organic synthesis [9]. Additionally, ILs have found useful applications in sensors, solar cells, solid state photocells, batteries, and as thermal fluids, lubricants, hydraulic fluids, ionogels, fuels and electrolytes in thermoelectrochemical cells due to their wide electrochemical windows and good conductivities [3,10-15].

The two forms of ILs: aprotic ionic liquids (APILs) and protic ionic liquids (PILs) have gained growing interest in science and technology. The PILs are normally simpler and less costly to prepare than AILs - and other classes of ILs - due to the absence of byproducts [16]. Therefore, the use of PILs as novel green solvents for lipid extraction that can replace flammable and highly toxic volatile solvents (such as hexane and chloroform) has attracted significant attention. Lactams, particularly caprolactam (CP) and butyrolactam (BT) are a type of amine derivatives that could be quaternized to form new types of PIL cations (CPILs and BTILs). Compared to imidazolium-based ILs and organic solvents, they are relatively cheaper, have lower intrinsic toxicity (hence safer to use), and CP is available in large quantities from industry [16]. Among the many current and future applications of ILs, it is worth noting their role in lipid extraction from different biomasses. For example, the CPILs with carboxylate anions, specifically formate showed high efficiency in lipid extraction processes from microalgae biomasses [17]. In fact, the anionic

structure of ILs influences the extraction effectiveness of lipids. For example, hydrophobic and water immiscible ILs have a poor extraction efficiency, whereas hydrophilic and water miscible ILs have a high extraction efficiency [16,18]. Furthermore, distinct microalgal cell wall structures (cellulose, glycoprotein, silica, and peptidoglycan) may play a key role in ILs' species selection. In spite of the many potential advantages that CPILs could confer, they are rarely synthesized and hence their application remains limited. Therefore, the current focus of ILs research is on developing new types of CPILs that can act as green solvents, and thereby improve the long-term viability and sustainability of lipid extraction procedures. Six CPILs namely: Caprolactam chloride, Caprolactam methyl sulphonate, Caprolactam trifluoromethane sulfonate, Caprolactam acetate, Caprolactam hydrogen sulphate and Caprolactam trifluoromethane-acetate were synthesized by combination of caprolactam with different organic and inorganic Brønsted acids. Of these, the first three are novel ILs.

2. MATERIALS AND METHODS

2.1 Chemicals and Reagents

Caprolactam (CP 99%), Methane sulphonic acid ($\text{CH}_3\text{SO}_3\text{H}$, 99%), Trifluoromethanesulphonic acid ($\text{CF}_3\text{SO}_3\text{H}$, 99%) were supplied by Sigma-Aldrich (Germany). Hydrochloric acid (HCl, 37%), Sulfuric acid (H_2SO_4 , 98%), Trifluoroacetic acid ($\text{CF}_3\text{CO}_2\text{H}$, 98%) were supplied by Labo Chem PVT (India), Toluene (99.6%) was supplied by VWR (China) and Acetic acid (CH_3COOH , 99.6%) was obtained from M&B (England).

2.2 Synthesis of Ionic Liquids

Six CPILs (Table 1), namely, Caprolactam chloride (CPHA), Caprolactam methyl sulphonate (CPMS), Caprolactam acetate (CPAA), Caprolactam hydrogen sulphate (CPSA),

Caprolactam trifluoromethane- acetate (CPTFA), and Caprolactam trifluoromethane sulfonate (CPTFS) were prepared using the method reported by Huang et al. [19]. The synthesis of (CPHA) was used as an example in a typical experiment, and others were similar.

Water (10 mL) was added to a 100 mL flask containing 11.32 g of Caprolactam (0.1 mol) and stirred. Then 3.65 g of hydrochloric acid (0.1 mol) was added slowly into the flask, within 30 min in an ice bath. The flasks were left to stand for 24 hours at room temperature. Thereafter, water was removed by vacuum distillation and the mixture was washed with toluene, followed by drying at 80°C in a vacuum oven, and the resultant percentage yield was calculated. Then water content and the pH of the synthesized CPILs were measured and reported (Table 1). For pH determination, CPILs were prepared with concentration (0.01mol/L).

The preparation of lactam-based ILs is illustrated in Fig 1.

2.3 Characterization of the Synthesized Ionic Liquids

The chemical structures of the synthesized CPILs were characterized using Fourier transform infrared (FT-IR) spectroscopy and Raman spectroscopy. The FT-IR spectra were recorded using a Shimadzu IRTracer –100 spectrometer (Japan), through the use of a KBr liquid cell or by dilution with thoroughly dried KBr followed by pressing into pellets, in the range of 500 to 4000 cm^{-1} . Raman spectra were obtained using a Raman Spectrometer (CBEx 2.0, USA) at room temperature in small glass tubes by using laser light of 785 nm in the range of 400 to 2200 cm^{-1} . The density and viscosity were measured at 20°C by common methods such as weight method (pycnometer) and capillary viscometer (Oswald), respectively [20,21].

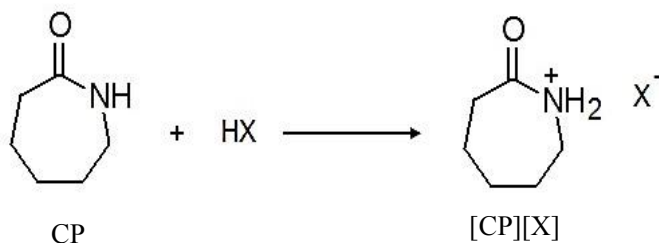
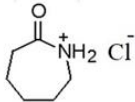
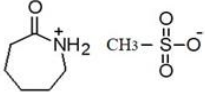
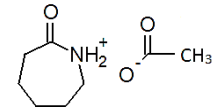
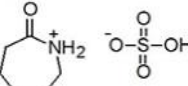
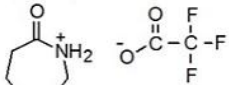
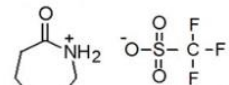


Fig. 1. X= HSO_4 , CPSA; X= Cl, CPHA; X= CH_3SO_3 , CPMS; X= CH_3COO , CPAA; X = CF_3CO_2 , CPTFA & X = CF_3SO_3 , CPTFS

Table 1. Structural formula, molecular formulae and water content of the six synthesized ILs

Compound	Structural formula	Molecular formula	Water content (w/w%)	pH
CPHA		C ₆ H ₁₂ ClNO	0.74	2.66
CPMS		C ₇ H ₁₅ NO ₄ S	1.85	2.36
CPAA		C ₈ H ₁₅ NO ₃	4.52	3.01
CPSA		C ₆ H ₁₃ NO ₅ S	1.21	1.08
CPTFA		C ₈ H ₁₂ F ₃ NO ₃	0.65	1.24
CPTFS		C ₇ H ₁₂ F ₃ NO ₄ S	1.53	1.28

3. RESULTS AND DISCUSSION

3.1 Physical Properties of Synthesized Ionic Liquids

All the CPILs were prepared with high yields of 80.7 - 99.6 %. Among them, CPAA, CPSA, CPTFA, and CPTFS were colorless with excellent fluidity at room temperature, whereas

CPMS and CPHA were white solids (Fig. 2). They are moisture stable. All the ILs were insoluble in hexane and revealed high miscibility with water and methanol, confirming that they are hydrophilic. This may provide evidence that these ILs are suitable for the dissolution of cellulose that lead to easy release of lipids from microalgae biomass [16].

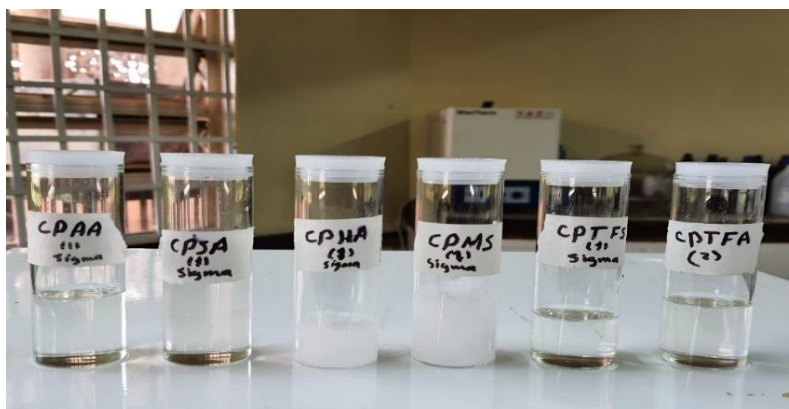


Fig 2. The synthesized ionic liquids from left to right: CPAA, CPSA, CPHA, CPMS, CPTFS and CPTFA

3.2 Characterization of the Synthesized Ionic Liquids

3.2.1 FTIR analysis of caprolactam

Garbuzova & Lokshin [22] reported that solid caprolactam is present in dimeric hydrogen-bonded form as shown in Fig. 3a. Therefore, the peaks at 3296.12, 3209.33 and 3074.32 cm^{-1} correspond to N-H in hydrogen bonding as shown in Fig. 3b. The observed peaks at 2929.67 and 2856.38 cm^{-1} were assigned to the vibration of asymmetrical and symmetrical stretching, respectively, of the $-\text{CH}_2-$ group from cyclic lactam. The peak at 1487 cm^{-1} corresponds to a hydrogen bond formed between C=O and N-H. The band at 1440.73 cm^{-1} relates to the scissoring vibration in the $-\text{CH}_2-$ group and the band located at 1417.58 cm^{-1} was assigned to the bending vibrations in the same group. The bands at 1365.51, 1290.29, 1257.50, and 1197.71 cm^{-1} correspond to the amide group bonded with the alkyl group ($-\text{NH}-\text{CH}_2-$). The C=O bond, related to lactam was observed at 1658.67 cm^{-1} and was confirmed by the bands at 865.98 and 690.47 cm^{-1} . The band located at

823.55 cm^{-1} relates to out of plane bending vibration of the NH bond. Both bands at 582.46 and 505.31 cm^{-1} correspond to the amide group of lactam ring [22,23]. Moreover, according to a study reported by Huang et al. [19], the characteristic peaks of caprolactam-based ionic liquids at 1441-1445 cm^{-1} and 2930-2941 cm^{-1} may relate to N-H and N-C-H, respectively.

3.2.2 FTIR analysis of the synthesized caprolactam-based ILs

The characteristic IR absorption peaks of the ILs are shown in Fig. 4. A significant change was observed in the caprolactam IR spectrum after reaction with HCl, $\text{CH}_3\text{SO}_3\text{H}$, $\text{CF}_3\text{SO}_3\text{H}$, $\text{CH}_3\text{CO}_2\text{H}$, $\text{CF}_3\text{CO}_2\text{H}$, and H_2SO_4 acids to produce ionic liquids. This modification results in the loss of three N-H hydrogen-bonded peaks, and new peaks at different frequencies appear due to the interaction between the cation and the anion. Furthermore, the decrease in intensities of the C=O peak observed in most spectra is attributed to the loss of double bond character in the caprolactam units due to the formation of hydrogen bonds [24].

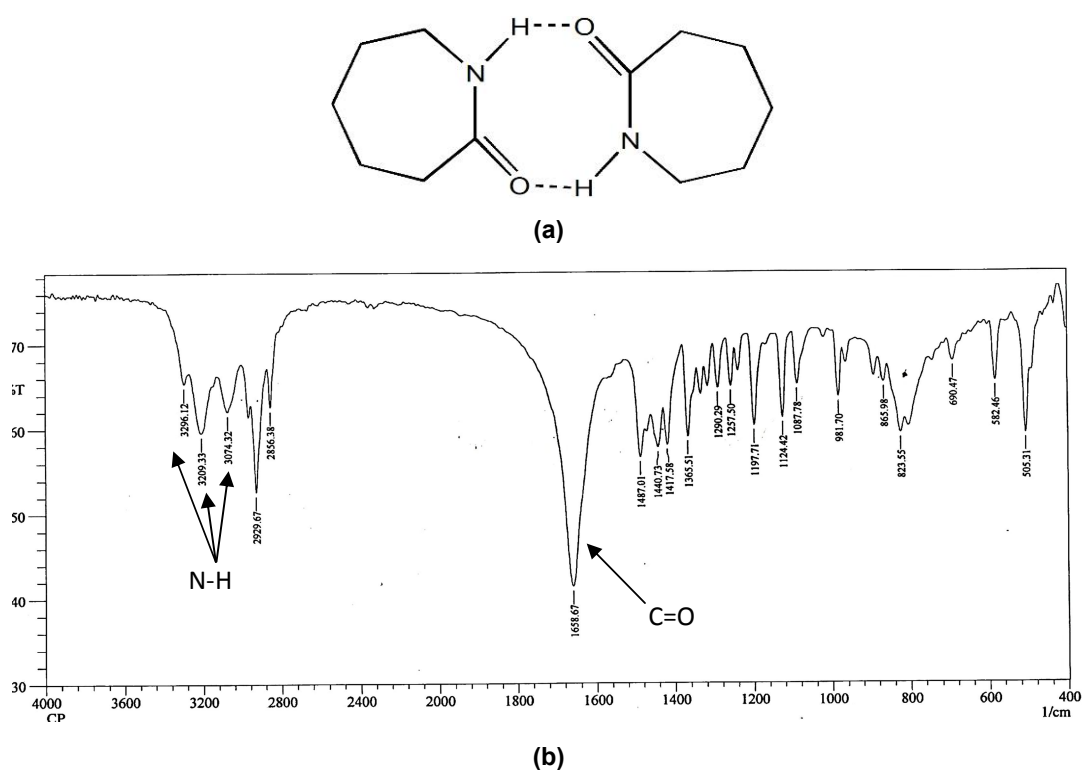
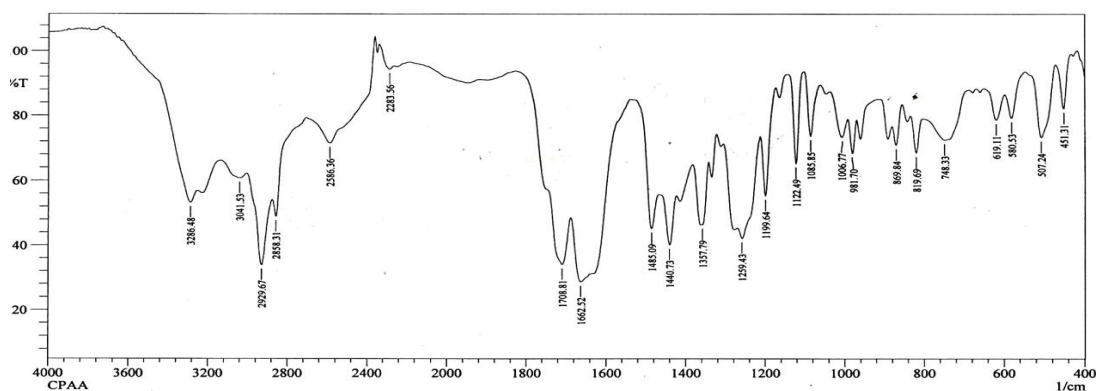


Fig. 3. (a) Solid caprolactam dimer showing hydrogen bonding between two amide groups, (b) FTIR spectrum of Caprolactam

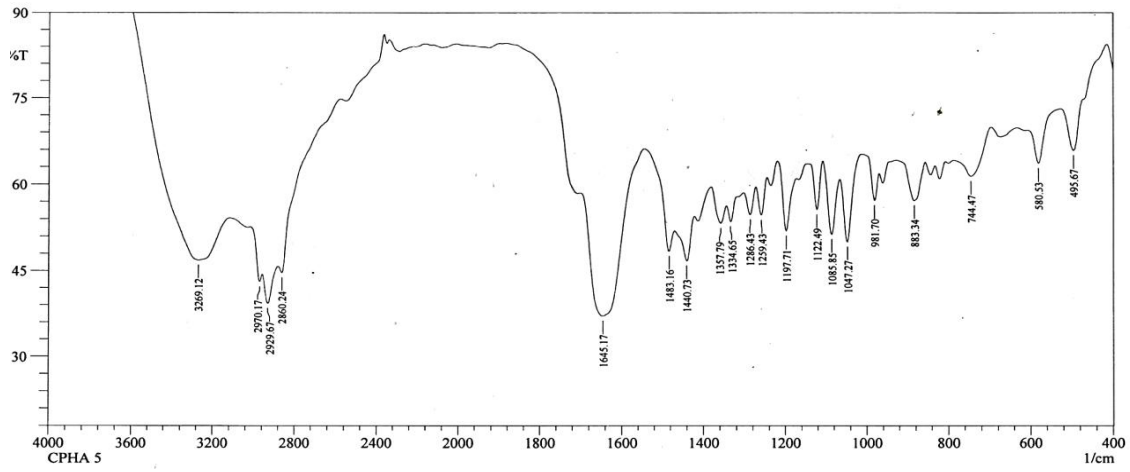
Functional groups were assigned to the characteristic IR absorption peaks for each synthesized IL (Fig. 4a – 4f). CPAA (Fig. 4a): N-H₂⁺ (3286.48 cm⁻¹); CH₃ asymmetric stretch (3041.53 cm⁻¹) related to acetate ion; CH stretching (2929.67, 2858.31 cm⁻¹); C=O (1708.81, 1662.52 cm⁻¹); CO...H (1485.09 cm⁻¹); CH bending vibration (1440.73, 1357.79 cm⁻¹). CPHA (Fig. 4b): N-H₂⁺ (2970.17 cm⁻¹); asymmetric and symmetric CH stretching (2929.67 and 2860.24 cm⁻¹), respectively; C=O (1645.17 cm⁻¹); CO...H (1483.16 cm⁻¹); CH scissor vibration (1440.73, 1357.79 cm⁻¹). CPMS (Fig. 4c): N-H₂⁺ (3404.13); CH₃ asymmetric stretch (3049.25 cm⁻¹) related to methanesulphonate ion; CH asymmetrical stretching (2937.38 cm⁻¹); C=O (1704.96, 1623.95 cm⁻¹); CO...H (1502.44 cm⁻¹); CH bending vibrations (1413.72 cm⁻¹); -NH-CH₂- (1332.72 cm⁻¹); the broad and sharp peak located at 1191.93 cm⁻¹ may be due to overlapping of the symmetric stretch SO₃ and (-NH-CH₂-), whereas the peak at (1043.42 cm⁻¹) may correspond to S-O-H bend or SO₃ symmetric stretch (associated). The peaks at 781.12, 599.82 and 557.39 cm⁻¹ may indicate free and associated stretch (C-S cm⁻¹) and 536.17 cm⁻¹ may be related to SO₃ symmetric bend [25,26]. CPSA (Fig 4d): N-H₂⁺ (3045.39 cm⁻¹); CH asymmetrical stretching (2935.46 cm⁻¹); C=O (1681.81, 1633.59 cm⁻¹); the band at (1512.09 cm⁻¹) may be related to CO...H or scissor H-C-H); CH bending vibrations (1438.80 cm⁻¹); overlapping of symmetric stretch SO₃ and -NH-CH₂- at (1193.85 cm⁻¹). The peak that may be related to the S-O-H bend or SO₃ symmetric stretch (associated) is located at (1054.99 cm⁻¹). The two peaks at (879.48 and 852.48 cm⁻¹) may indicate an associated and monomeric stretch of (S-OH) [26]; stretch C-S at (781.12 cm⁻¹); SO₃ symmetric bend (580.53 cm⁻¹). CPTFA (Fig. 4e): N-H₂⁺ (3047.32 cm⁻¹); CH stretching (2939.31,

2866.02 cm⁻¹); C=O (1676.03, 1627.81 cm⁻¹); CH scissor vibrations (1438.80 cm⁻¹) or may also be related to symmetric vibration in CO₂, and the one at (1336.58 cm⁻¹) associated to -NH-CH₂- or may also attributed to symmetric vibration in CF₃. Broadband at (1178.43 cm⁻¹) may indicate vibrational in F-CF₂ and symmetrical CF₃ stretch. The peaks located at (798.47, 721.33, 601.75, and 514.96 cm⁻¹) may be associated to symmetric CO₂ scissor and umbrella, bending and asymmetric vibrations in CF₃, respectively [27]. CPTFS (Fig. 4f): N-H₂⁺ (3242.12 cm⁻¹); CH asymmetrical stretching (2941.24 cm⁻¹); C=O (1638.74 cm⁻¹); CO...H or scissor H-C-H at (1514.02 cm⁻¹); CH bending vibrations (1450.37 cm⁻¹). A broad overlapping band at (1251.72 and 1170.71 cm⁻¹) indicates -NH-CH₂-, asymmetrical SO₃ stretching and symmetrical CF₃ stretch [2]. The peak located at (761.83 cm⁻¹) may be associated to symmetric FCF scissor and stretch C-S. The bands observed at 638.39, 578.60 and 518.82 cm⁻¹ may be attributed to SO bond oscillation, symmetric (OSO) scissor & (FCF) scissor and asymmetric (OSO) scissor & (FCF) scissor, respectively [2].

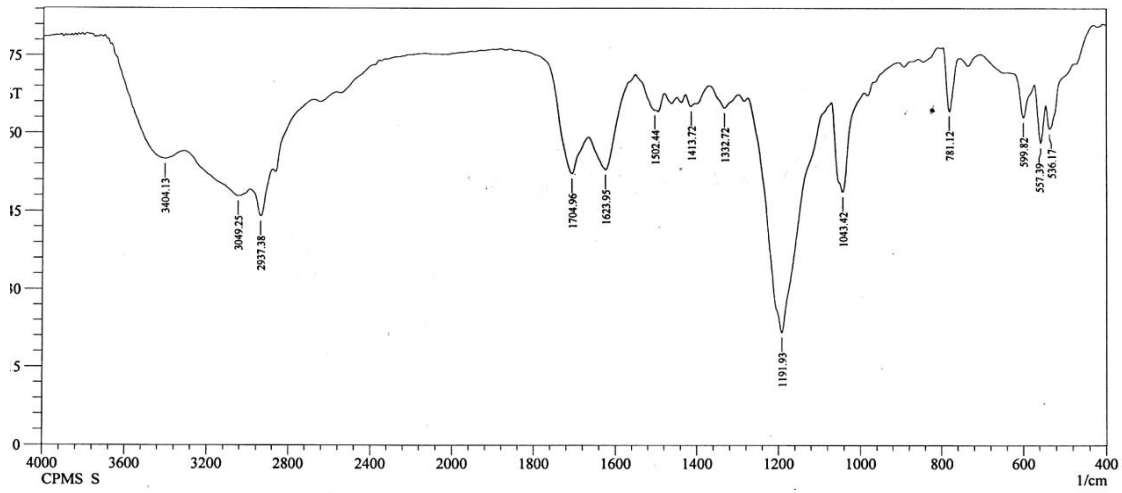
The observable split carbonyl band in CPAA and CPTFA are related to carbonyl groups in acetate and Caprolactamium ions at the higher and lower frequencies, respectively. Nevertheless, the same split exists in each of CPSA, CPMS, and CPTFS, and this phenomenon was explained by Winston and Kemper [28], as resulting from coupled stretching vibrations of the O=C-C group in acyclic structures, through symmetric asymmetric modes. Furthermore, there is a significant change in the fingerprint regions in IR spectra of all ILs except CPHA, which showed slight differences compared to caprolactam spectrum. This may infer the presence of more interactions between Caprolactamium cation and the anions.



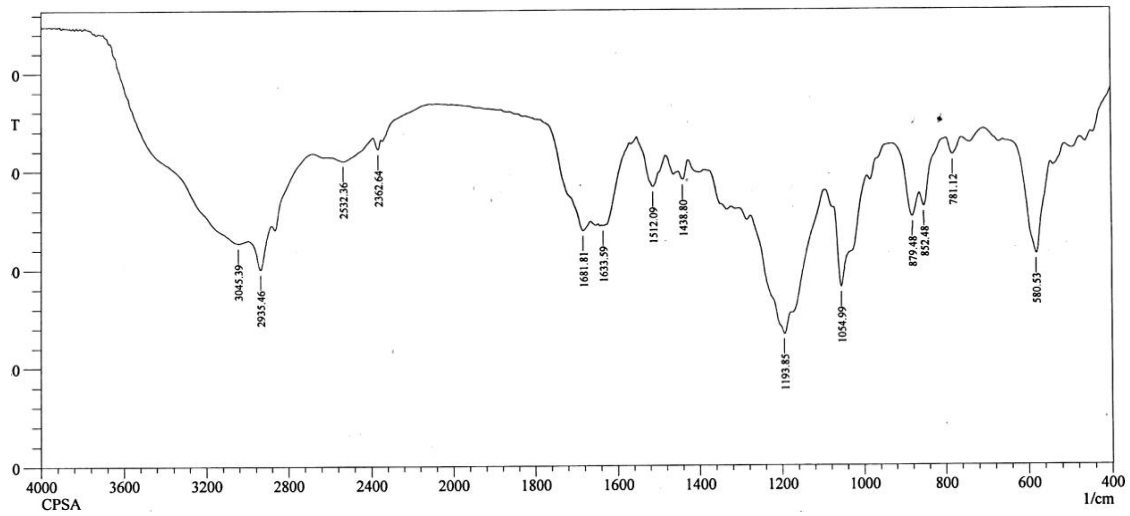
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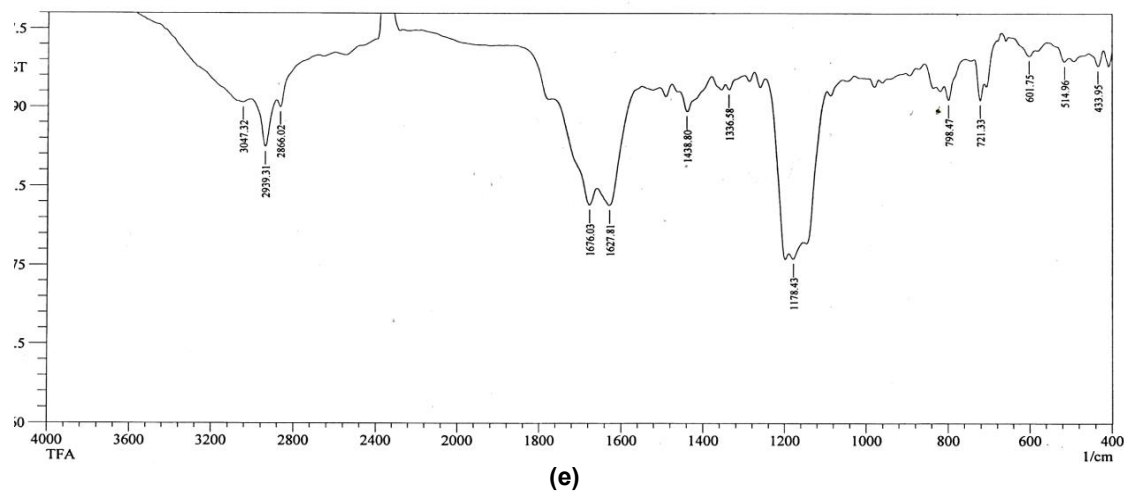
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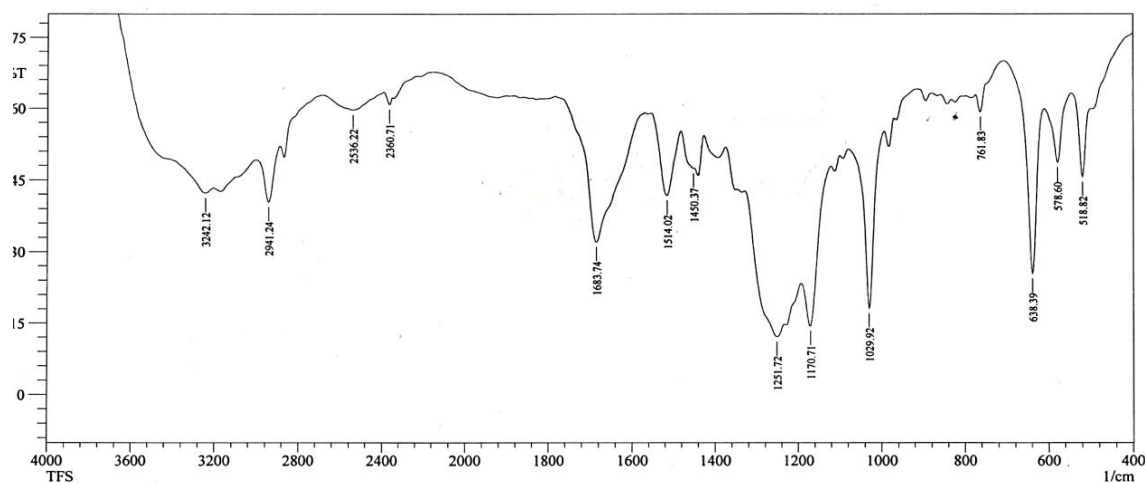
(c)



(d)



(e)



(f)

Fig. 4. FTIR spectra of (a) CPAA, (b) CPHA (c) CPMS, (d) CPSA, (e) CPTFA and (f) CPTFS

3.2.3 Raman analysis

Fig. 5 shows Raman spectra of the original caprolactam and its ionic liquids [ILs] in the region $400\text{--}2200\text{ cm}^{-1}$. The caprolactam (CP) spectrum shows two main absorption peaks. Compared to a previous study by Triggs et al. [29], the two peaks correspond to the vibrations of the C=O stretch between ($1600\text{--}1700\text{ cm}^{-1}$) and C-N stretch between ($1480\text{--}1580\text{ cm}^{-1}$). Other peaks appeared in the spectrum for the bending vibration of C=O ($579.75, 865.56\text{ cm}^{-1}$), and at 696.59 cm^{-1} which may be assigned to vibration of (N-H). In the ($739.59\text{--}1438.98\text{ cm}^{-1}$) region of the (CP) Raman spectra, the absorptions may be attributed to methylene sequences (CH₂ rock, wag, twist, and scissor) [30].

Similar absorption peaks were observed in CPAA and CPHA with a slight shift in position and/or a difference in band areas and intensities. In particular, the C=O stretch on CPAA was located at 1618.73 cm^{-1} (for Caprolactamium cation) and 1708.61 cm^{-1} (for acetate anion), whereas on CPHA was located at 1625.92 cm^{-1} (for Caprolactamium cation). The lower frequencies of C=O bend on the two ILs appeared at ($579.7, 619.3$ and 577.96 cm^{-1}), respectively. The observable change in absorption peak intensities occurred in the region of C-N stretch and N-H vibration, which provide evidence for the generation of new interactions due to the formation of the ILs. Moreover, there is a band that appears at 879.94 cm^{-1} , which may relate to symmetric C-C stretch in the acetate ion [31].

In CPSA, CPMS, and CPTFS spectra, a sharp and intense peak occurred between (1000-1100 cm^{-1}) and was assigned to asymmetric vibration of SO_4 and symmetric SO_3 stretch [2,25,32] whereas the strong peak at 742 cm^{-1} in the CPTFA spectrum may be attributed to the presence of a C-F bond [33], which may also be overlapped by the vibration of CH_2 rock. Moreover, a significant change was revealed by the intensities and positions as well as the

disappearance of the main characteristic peaks such as C=O, C-N, and N-H. Specifically, the missing carbonyl band in each of CPTFA and CPTFS spectra, may be explained by the presence of fluorine groups, which may generate more interactions with caprolactamium cations through H-bond due to the protonation of the carbonyl group as illustrated in Fig. 6 [34]. The summary of all absorption bands is presented in Table 2.

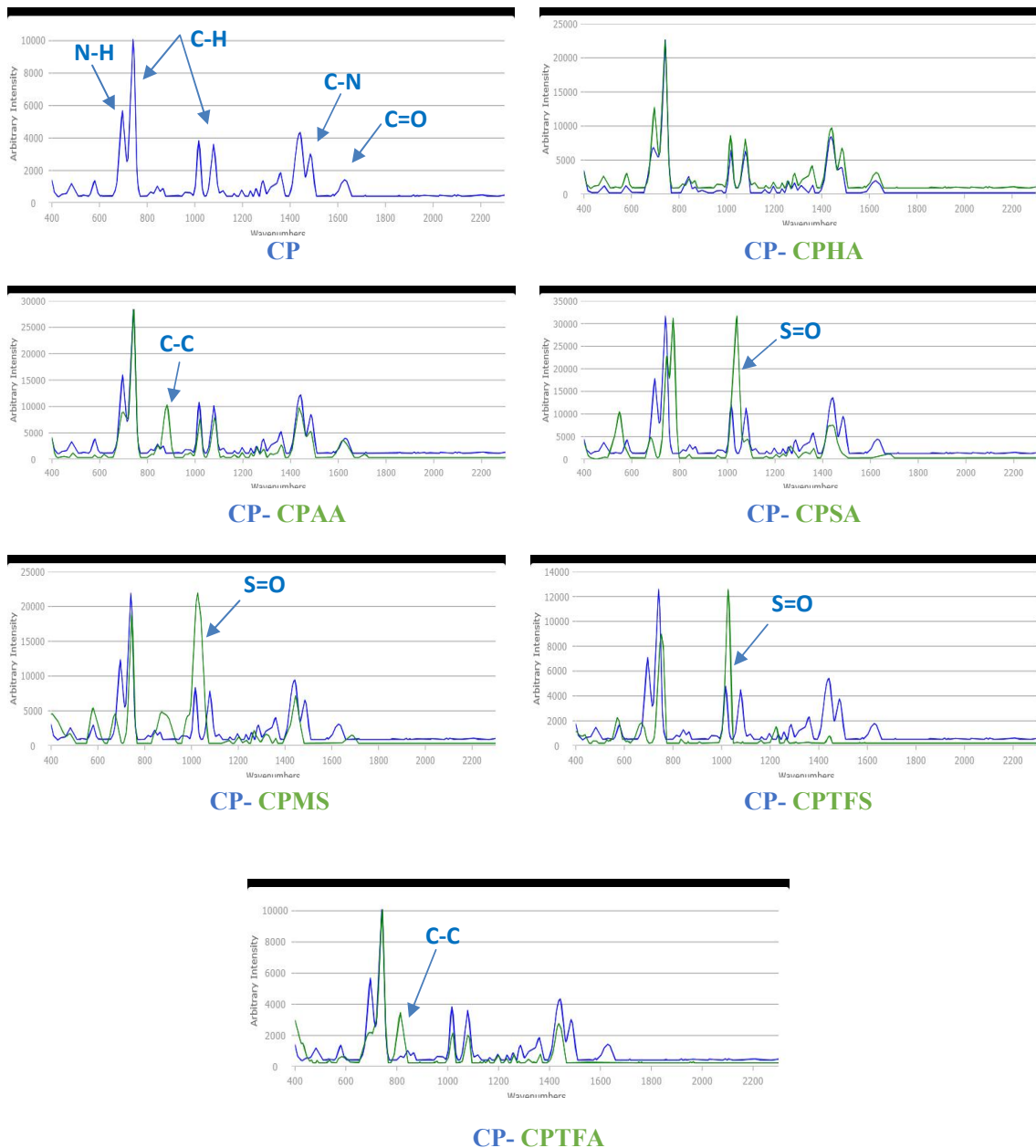


Fig 5. Comparison of Raman spectra of caprolactam (CP, blue) and its ionic liquids (green)

Table 2. Vibrational wavenumbers and mode assignments of caprolactam ionic liquids (in cm⁻¹)

ILs	ν_s C=O	ν C-N	ν N-H	δ C=O	Methylene sequences CH2	ν_s C-C	ν_{as} SO4	ν_s, δ SO3	ν_s S-OH
CP	1627.53	1485.71	696.59	577.96	1016&1077,r 1284.31& 1358.09,w& tw 1438.98 sci				
CPAA	1618.73, 1708.61	1482.12	696.59	579.75	1018 & 1081r 1359.89,w& tw 1437.16 sci	879.94			
CPHA	1625.92	1487.52	694.8	574.36	1016.56,1075.88r 1358.09 w & tw, 1438.98 sci				
CPMS	1689.79	-	745.13	-	1305.52, w & tw 1431.84 sci 942.6, CH3 r			1035.11,s 544.1, δ	
CPSA	1685.24	-	671.43	579.75	1268.21& 1320.34, w & tw, 1446.17 sci		1025.54		878.15
CPTFA	-	-	693 sho	588.74	1016.56, 1079r 1261.02& 1363.48, w & tw 1437.18 sci	813.43			
CPTFS	-	-	667.83	572.56	1221.48, w & tw 1442.27sci			1027.34	

ν_s =stretch; δ = Asymmetric bend; r = rock, w=wag; tw= twist; sci= scissor; sho= shoulder

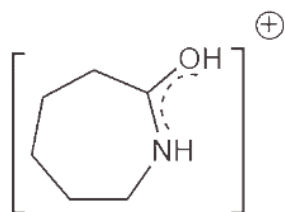


Fig 6. Protonation of ϵ -caprolactam

3.3 Density and Viscosity

The densities (ρ) and dynamic viscosities (η) of CPAA, CPSA, CPTFA, and CPTFS ILs were measured at 20 °C without further drying and the results are presented in Table 3. The η and ρ magnitudes of CPAA were the lowest. These values are different from η and ρ at 20 °C (39.81 mPa.s and 1.06 g/ml), respectively, reported by Chhotaray et al. [35] with water content of 3872 $\mu\text{g/g}$. The viscosity and density of CPTFA (Table 3) are almost similar in magnitude to the values of 28 mPa.s and 1.24 g/ml reported at 25°C by Du et al. [20]. The CPSA exhibited a higher viscosity value (3549.63 mPa.s), and this is in stark contrast to the findings of Fábos et al. [31] who reported that the concentration was below detection limit. The viscosity value of CPTFS has not yet been reported in literature. In contrast, the density value of CPSA (1.358g/ml) is lower than the density value of CPTFS (1.375g/ml). Fábos et al. [31] also reported that the density value of CPSA was (1.372g/ml) at 50°C. The

differences in the viscosities and densities from literature can be explained by the presence of water impurities [36]. Moreover, given that viscosity and density depend on molecular weight (Mw) of the anions - as in the case of CPAA, CPTFA, and CPTFS - then it follows that the two properties increase with increase in Mw [37]. However, CPSA is structurally dependent on density and viscosity, findings that have been reported for imidazolium-based ILs by Ullah et al. [38], who demonstrated that the phenomena is due to strong interaction and less steric hindrance of cationic and anionic structures. We propose that the interaction between $[\text{HSO}_4]^-$ and Caprolactamium cation results in hydrogen bonding between the $[\text{HSO}_4]^-$ ion dimers and the acidity hydrogen of the amide group on the caprolactam (Fig. 7). Dronskowski [39], investigated and showed the bridging of two $[\text{HSO}_4]^-$ anions by two short hydrogen bonds to form dimers, as well as the formation of hydrogen bonding between the dimer and the acidity hydrogen on the imidazolium ring.

Table 3. Density and viscosity of lactam-based ILs at 20°C

Ionic liquid	Density (g/ml)	Viscosity (mPa.s)
CPAA	1.042	13.69
CPAS	1.358	3,549.63
CPTFA	1.249	24.81
CPTFS	1.375	144.76

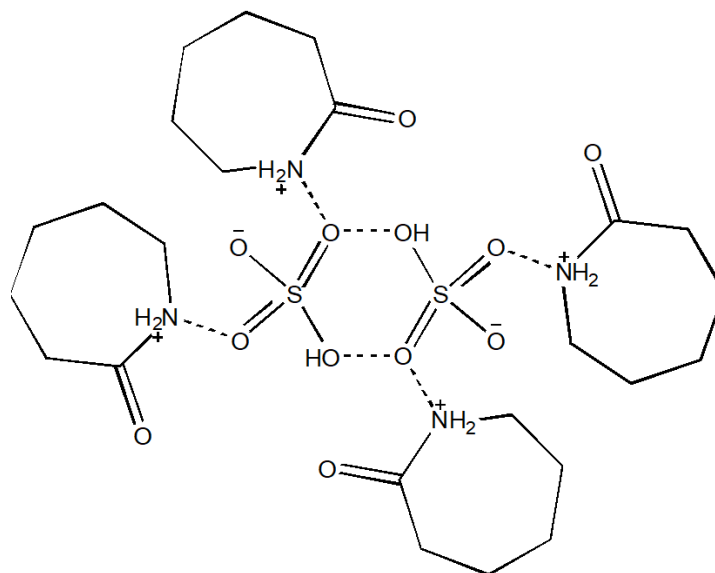


Fig 7. Proposed hydrogen bond interaction in CPSA

4. CONCLUSION

The ILs were synthesized through a simple neutralization reaction, characterized via FTIR and Raman spectroscopy as well as density and viscosity. Considering the FTIR and Raman spectra, the characteristic absorption bands of the synthesized compounds showed a big shift in position and/or intensity (compared to caprolactam), indicating the formation of ionic liquids. All the ILs were insoluble in hexane and showed high miscibility with water and methanol, indicating their hydrophilic nature, and thus their ability to dissolve the cellulose of microalgae cell wall for easy release of lipids. The results showed that both density and viscosity values increase with molecular weight of the anion – except for Caprolactamium hydrogen sulphate (CPSA) - and this is because of interactions between cations and anions resulting from formation of dimers between the hydrogen sulphate anions. Three novel compounds (Caprolactam chloride, Caprolactam methyl sulphonate, Caprolactam trifluoromethane sulfonate,) with potential applications as green extraction solvents were synthesized. Further studies should determine the fusion temperature of the synthesized compounds that are solid at ambient temperature.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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