RESEARCH PAPER

Novel Dicationic Imidazolium Based Ionic Liquid: Synthesis, Structural Characterization, and Electrochemical Analysis

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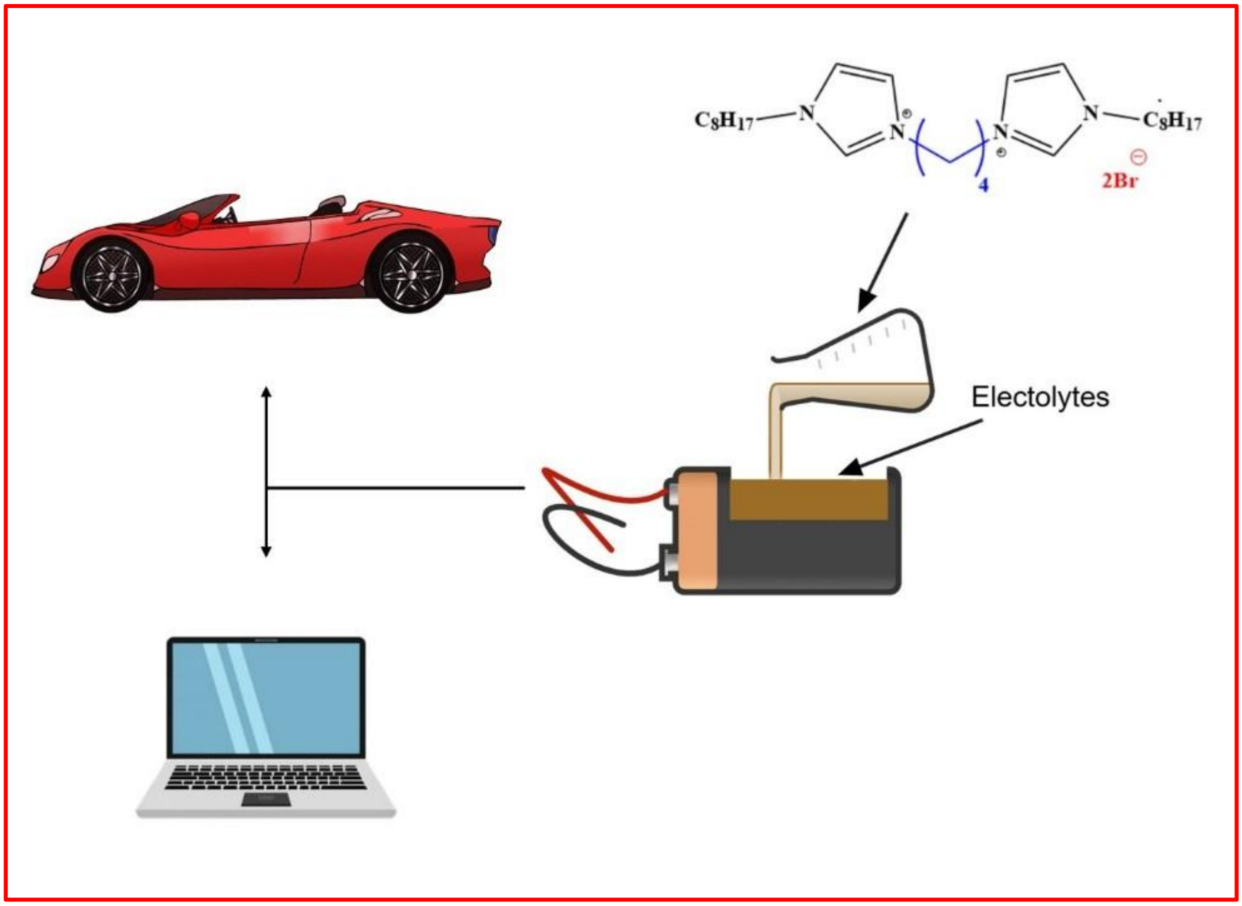
RESEARCH HIGHLIGHTS

- Synthesis of novel di-cationic ionic liquid 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide via quaternization of 1-octyl imidazole with butane-1,4-dibromide.
- The structure was confirmed by FT-IR, UV-Vis, ^{1}H NMR, and ^{13}C NMR spectroscopies.
- The electrochemical properties were evaluated by conductometry and cyclic voltammetry, highlighting the potential of the material as an electroactive compound.
- Thermal stability was confirmed by TGA.



ABSTRACT

In this study, a newly synthesized dicationic ionic liquid (DIL), 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide, was synthesized via a quaternization reaction between 1-octyl imidazole and butane-1,4-dibromide. The synthesized DIL was characterized using several analytical techniques, such as FT-IR, UV-visible spectroscopy, ^{1}H NMR, and ^{13}C NMR, to ensure the desired molecular structure. In addition, the conductivity of the synthesized DIL was determined, and the results indicated its potential use as an electrolyte. Cyclic voltammetry was performed to investigate the electrochemical behavior of the DIL. These findings may lead to the development of dicationic ionic liquids as potential electrolytes for various technological applications.

Keywords: Imidazolium, Di-cationic ionic liquid, FT-IR, Conductometry, Cyclic voltammetry

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Conflict of interest: None

1. Introduction

Ionic Liquids (ILs) are salts that remain in the liquid state at temperatures below 100 °C in their pure form [1–3]. These innovative compounds possess a unique combination of properties: they are highly hydrophilic, exhibit very low vapor pressure, are thermally stable, and have high ionic conductivity and low melting points [4,5]. Owing to these characteristics, ILs have garnered significant research interest in various industries. ILs can be categorized into several groups based on their cationic structure, including imidazolium-, pyridinium-, ammonium-, phosphonium-, and sulfonium-based ILs [1,6]. Among them, imidazolium-based ILs have been the most extensively studied because of their relatively low toxicity and environmental compatibility. ILs are increasingly used as safer alternatives to conventional volatile organic solvents. ILs have been employed in a wide range of applications owing to the tunability of their ionic components. These include their use as solvents [7], electrolytes [8], capacitors, catalysts [9], fuels, lubricants [10], high-temperature lubricant additives [11], and heat-transfer fluids [12–14].

A more specialized subclass of ILs, known as di-cationic ionic liquids (DILs), offers additional advantages over mono-cationic ILs. These benefits include higher viscosity, elevated melting point, improved thermal stability, and enhanced ionic conductivity [15–17]. Consequently, DILs are particularly well suited for advanced applications such as stationary phases in gas chromatography, high-temperature chemical processes, battery electrolytes, high-temperature lubricants, and dye-sensitized solar cells [18,19]. Imidazole ($C_3N_2H_4$) is a key precursor in the synthesis of many ILs because of its ability to form stable cationic structures that remain in the molten state, even at lower molar masses [20,21]. Imidazole-based ILs are particularly valued because of their thermal stability, persistent charge, and role as green solvents.

In the synthesis presented here, 1,4-dibromobutane was employed to link the two imidazole rings, while 1-bromooctane introduced an alkyl side chain. This approach led to the formation of a novel dicationic ionic liquid, 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide, synthesized via a quaternization reaction between 1-octylimidazole and butane-1,4-dibromide. The resulting DIL was thoroughly characterized by FT-IR, UV-Vis, ^{1}H NMR, and ^{13}C NMR spectroscopy to confirm its molecular structure. Ionic conductivity was measured, indicating its potential as an electrolyte. Cyclic voltammetry was conducted to assess the electrochemical behavior of the material, supporting its viability for application in electrochemical devices.

2. Materials and Methods

2.1. Materials

All chemicals used in this study were of ACS grade and used without further purification. However, the solvents were purified according to standard procedures. Imidazole,
1-bromooctane, 1,4-dibromobutane, NaOH, and magnesium sulphate were obtained from Sigma- Aldrich. Tetrahydrofuran (THF), Acetonitrile and Dichloromethane (DCM) were purchased from Hi-Media Chemicals.

2.2. Instruments employed

The electronic spectra of the compounds were recorded using a JASCO UV-Vis spectrophotometer at room temperature with DCM as the solvent in the wavelength range of 200–800 nm. Vibrational spectra were obtained using a JASCO ATR instrument with KBr discs. ^{1}H and ^{13}C NMR spectra were recorded on an Avance NEO 400 MHz instrument at room temperature (273 K), with CDCl3 as the solvent and tetramethylsilane as the internal standard. Cyclic voltammetry experiments were performed using a CHI6085C electrochemical analyzer. The DILs was then evaluated at 10mVs-1 scan rate in the potential range between -1.6 - 1.6 V in the intervals of 0.001V. Thermal analysis of the DILs was conducted using a NETZSCH thermogravimetric analyzer from room temperature up to 1300°C.

2.3. Synthesis of 1-octylimidazole

For the synthesis of 1-octylimidazole, 0.1 M of imidazole and 0.1 M of NaOH pellets were dissolved in 50 mL of THF. 0.1 M 1-bromooctane was added dropwise with stirring at room temperature. The reaction mixture was continuously stirred for 12 h. THF was then removed from the product under vacuum at 339.15 K. Excess starting material was removed five times with 20 mL of DCM. The resultant product was further dried in a vacuum oven for 6 hours at 313.15 K [22]. A schematic of the synthesis of 1-octyl imidazole is illustrated in Scheme 1.

2.4. Synthesis of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide

0.0125 M 1,4-dibromobutane was dissolved in acetonitrile (20 ml). To this end, 0.025 mol of 1-octyl-imidazole was added. The reaction mixture was refluxed for 12 hours at 353.15 K. Next, acetonitrile was removed from the product under vacuum at 356.15 K. Excess starting materials were removed five times with 20 mL of DCM. The resultant product was further dried in a vacuum oven for 6 hours at 313.15 K [23][24]. A schematic of the synthesis of 3,3'-(butane-1,4-dial)bis(1-octyl-1H-imidazol-3-ium) bromide is illustrated in Scheme 2.

3. Results and discussion

3.1. UV Spectroscopy

UV-visible (UV-Vis) spectroscopy was used to investigate the electronic transitions of the ionic liquids by measuring their absorbance. As shown in Figure 1, both compounds exhibit a strong absorption maximum at 225 nm. These absorption peaks arise from $\pi \rightarrow \pi^*$ transitions of π-electrons in the imidazole ring [25]. The higher absorption observed in the dicationic ionic liquid indicates a higher concentration of imidazole units than that of 1-octyl imidazole.

3.2. Infrared Spectroscopy

Fourier transform infrared (FT-IR) spectroscopy was used to identify the functional groups present in the compounds. The FT-IR spectra of 1-octyl imidazole and the dicationic ionic liquid are shown in Figures 2 and 3, respectively. Both compounds, containing the imidazole ring, exhibited characteristic bands at 629 and 752 cm^{-1}, which were attributed to ring deformation modes[26]. Additionally, 1-octyl imidazole displays a band at 1077 cm^{-1}, potentially indicating differences in the ring environment or substituent effects [27]. Aromatic C-N stretching vibrations were observed at 1228 and 1281 cm^{-1} in 1-octyl imidazole, whereas the dicationic ionic liquid exhibited a single corresponding peak at 1159 cm^{-1} [22][28]. This shift suggests variations in the electronic environment around the nitrogen atoms. Similarly, aromatic C-C stretching appeared at 1464 cm^{-1} in 1-octyl imidazole and at 1458 cm^{-1} in the dicationic ionic liquid. Side-chain vibrations also show a significant difference: C-H rocking occurs at 1375 cm^{-1} in 1-octyl imidazole and at 1367 cm^{-1} in dicationic ionic liquids. C-H stretching vibrations of both compounds appeared at approximately 2925, 2924, and 2855 cm^{-1}, consistent with the aliphatic chains of 1-octyl imidazole derivatives[29][30]. Notably, a broad absorption band at 3396 cm^{-1} in the dicationic ionic liquid, corresponding to O-H stretching, indicates the presence of water impurities. Overall, the spectral comparison confirmed the imidazole structures of both compounds. The FTIR spectral peak assignments for the synthesized 1-octyl imidazole and dicationic ionic liquids are tabulated in Tables 1 and 2, respectively.

3.3. NMR Spectroscopy

^{1}H and ^{13}C NMR spectroscopy further confirmed the structure of the di-cationic ionic liquid. In the ^{1}H NMR spectrum, as shown in Figure 4, signals observed in the region of 0.88-4.04 ppm correspond to the protons of the octyl side chains, indicating the presence of aliphatic methylene and terminal methyl groups [31]. Resonances appearing between 1.95-5.00 ppm are attributed to the protons associated with the bridging butane, confirming the bridging of the two imidazolium rings [32]. Peaks in the 7.00-9.00 ppm region are characteristic of aromatic protons from the imidazolium rings. The ^{13}C NMR spectrum further supports the ^{1}H NMR spectrum. Figure 5 shows that carbon resonances in the range of 14-50 ppm indicate aliphatic carbon atoms from the octyl chains [31]. Peaks between 25-80 ppm were assigned to the carbons of the butane bridge. The imidazolium ring carbons gave signals in the 120-140 ppm region, consistent with aromatic carbon environments [32].

3.4. Conductivity

The ionic conductivity of 3,3′-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide was measured using a digital conductivity meter equipped with a platinum black electrode (cell constant ≈ 1.0 cm^{-1}) at 25 ± 1 °C. The ionic liquid (0.5 g, 0.01 mmol) exhibited a conductivity of 4.36 mS cm^{-1}, demonstrating intrinsic charge mobility despite the relatively high viscosity typical of dicationic salts. Upon dissolution in 100 mL of distilled water, the conductivity increased to 7.94 mS cm^{-1}, indicating enhanced ionic dissociation and mobility in the aqueous medium. These results demonstrate that the synthesized dicationic ionic liquid possesses appreciable and competitive ionic conductivity, making it a promising candidate for electrochemical and ion-conducting applications.

3.5. Cyclic Voltammetry

Cyclic voltammetry (CV) of 3,3′-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide was carried out at 25 ± 1 °C in a three-electrode cell using a glassy carbon working electrode, Pt wire counter electrode, and Ag/AgCl reference. Two anodic peaks appeared at +0.859 V and +0.692 V, with corresponding cathodic peaks at –1.206 V and –1.388 V (vs. Ag/AgCl) (Fig. 5). The mean peak separations ($\Delta Ep \approx 90–110$ mV) exceeded the ideal value of 59 mV for a reversible one-electron process, indicating quasi-reversible behavior and moderate electron-transfer kinetics. The combined anodic/cathodic limits define an electrochemical stability window of ~2.2 V, comparable to other imidazolium-based dicationic ionic liquids and wider than many conventional aqueous electrolytes.

The two successive redox couples were tentatively assigned to single-electron reductions of the imidazolium C2 centers and possible participation of the butylene spacer, consistent with literature reports on bis-imidazolium systems[8]. While these data highlight a wide electrochemical window and reasonable stability, additional evaluation of the conductivity, viscosity, and cycling performance is required before confirming its suitability as an energy-storage electrolyte.

3.6. Thermal analysis

Thermal analysis is a crucial tool for evaluating the thermal stability and decomposition behavior of DILs. Figure 6 illustrates the thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) performed on 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) to determine its thermal properties under an inert atmosphere. The first region, approximately 50 -200°C shows only a slight mass decrease (5-7%) due to the evaporation of adsorbed and chemically bound water residues. The second transition occurs between 210-380°C due of the structural degradation of 3,3′-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide and accounted for 91.78% of the total weight loss. Third, the slow weight loss extends from ~400°C suggesting the complete degradation of the DILs [33][34]. For comparison, geminal dicationic imidazolium ILs reported in the literature show T_onset values of 223-294°C and complete decomposition near 900 °C. The synthesized DIL exhibited an onset temperature at the lower end of this range, but maintained a similar multi-stage degradation profile and near-complete mass loss, consistent with typical imidazolium-based dicationic ILs[35]. In addition, the exothermic peaks in DTA suggest a phase transition and decomposition process, rendering this material unsuitable for extremely high-temperature applications.

4. Conclusion

A novel dicationic ionic liquid (DIL), 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide, was synthesized and characterized. In addition, the conductivity of the synthesized DIL was determined, and the results indicated its potential use as an electrolyte. Cyclic voltammetry was performed to investigate the electrochemical behavior of the DIL. These findings may contribute to the development of dicationic ionic liquids as electrolytes for various technological applications. Overall, this study highlights the potential of this newly synthesized DIL for advanced electrochemical devices and energy storage systems.

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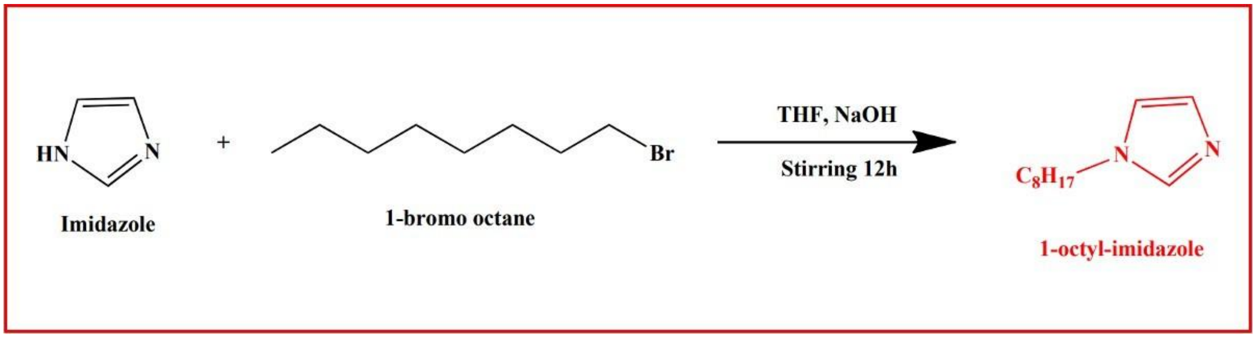
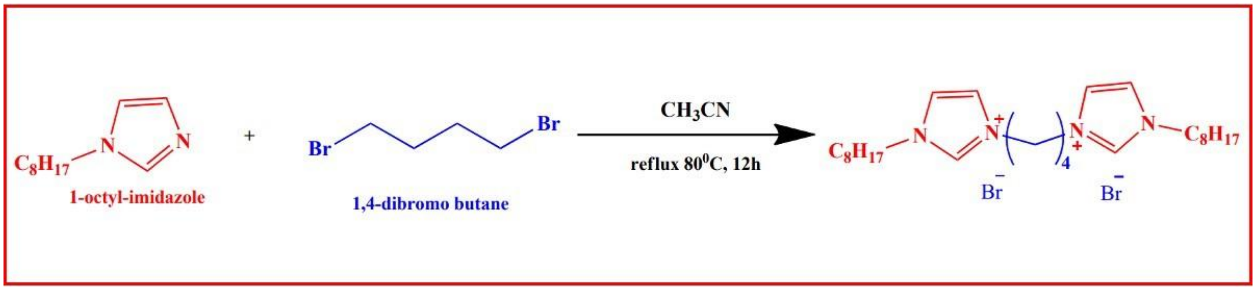
Figure captions

Scheme 1. Schematic route for the synthesis of 1-octyl imidazole.
Scheme 2. Schematic route for the synthesis 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide (DIL).
Figure 1. UV-Vis spectra of 1-octyl imidazole and 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.
Figure 2. FT-IR spectrum of 1-octyl imidazole and 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.
Figure 3. H^1 NMR spectrum of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.
Figure 4. C^{13} NMR spectrum of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.
Figure 5. CV graph of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide at $10mVs^{-1}$.
Figure 6. Thermal analysis of the synthesized dicationic ionic liquid, a) Differential Thermogravimetric (DTG) curve and b) Thermogravimetric Analysis (TGA) curve.

Scheme 1. Schematic route for the synthesis of 1-octyl imidazole





Scheme 2. Schematic route for the synthesis 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide (DIL).

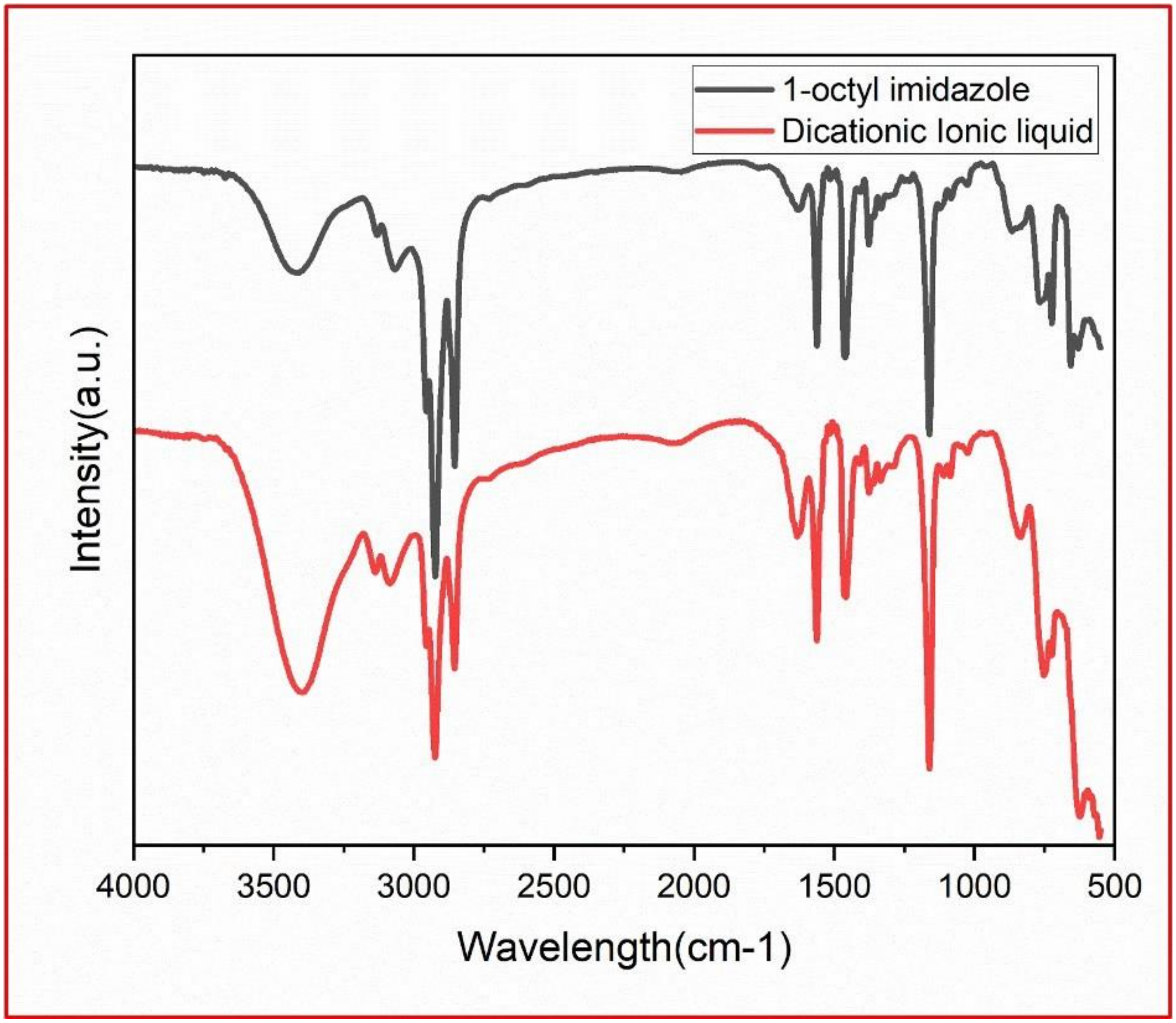


Figure 1. UV-Vis spectra of 1-octyl imidazole and 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.

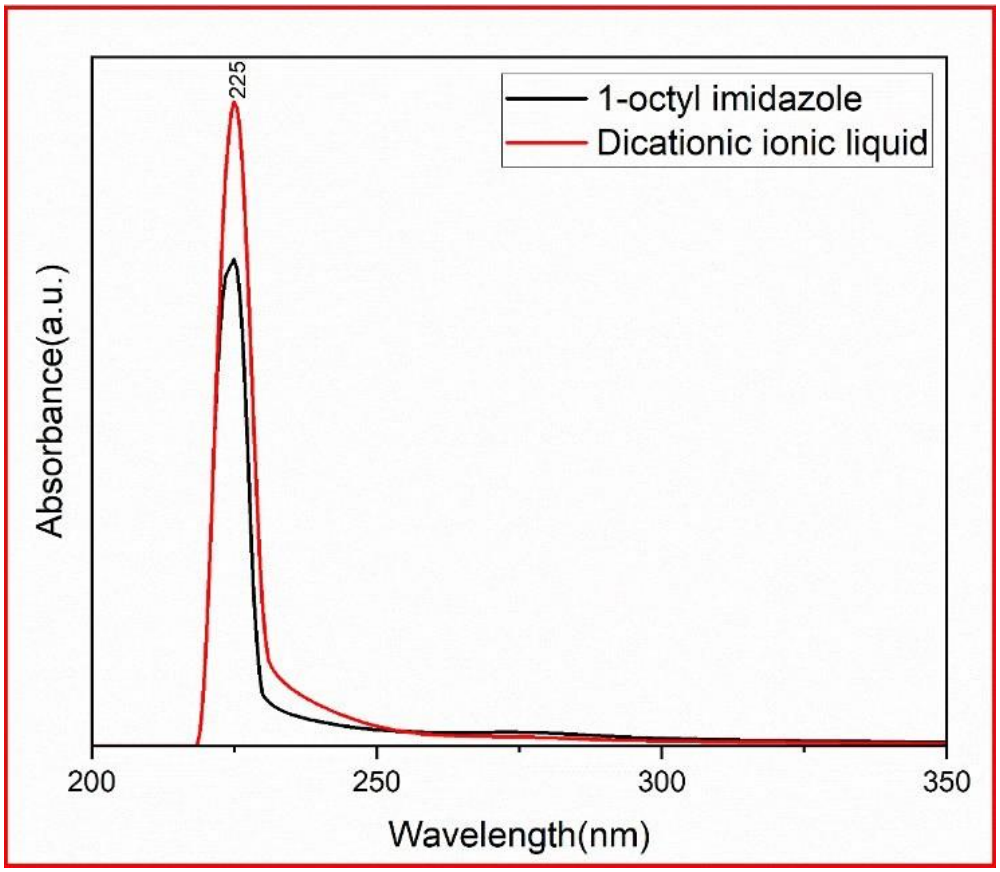


Figure 2. FT-IR spectrum of 1-octyl imidazole and 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.

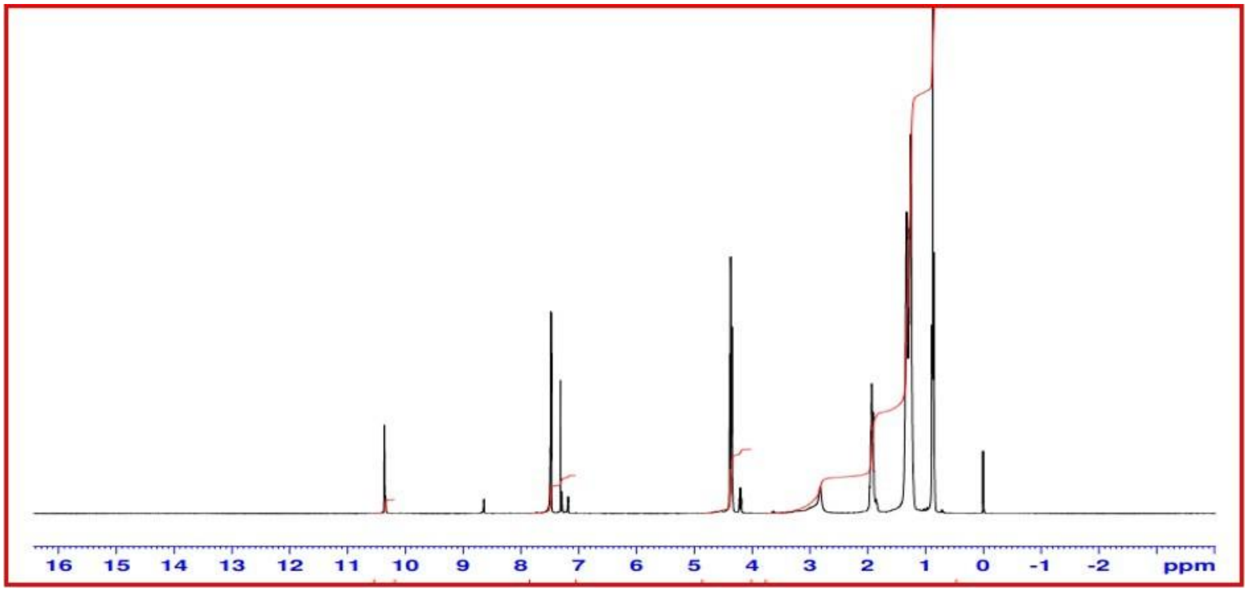


Figure 3. H^1 NMR spectrum of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.

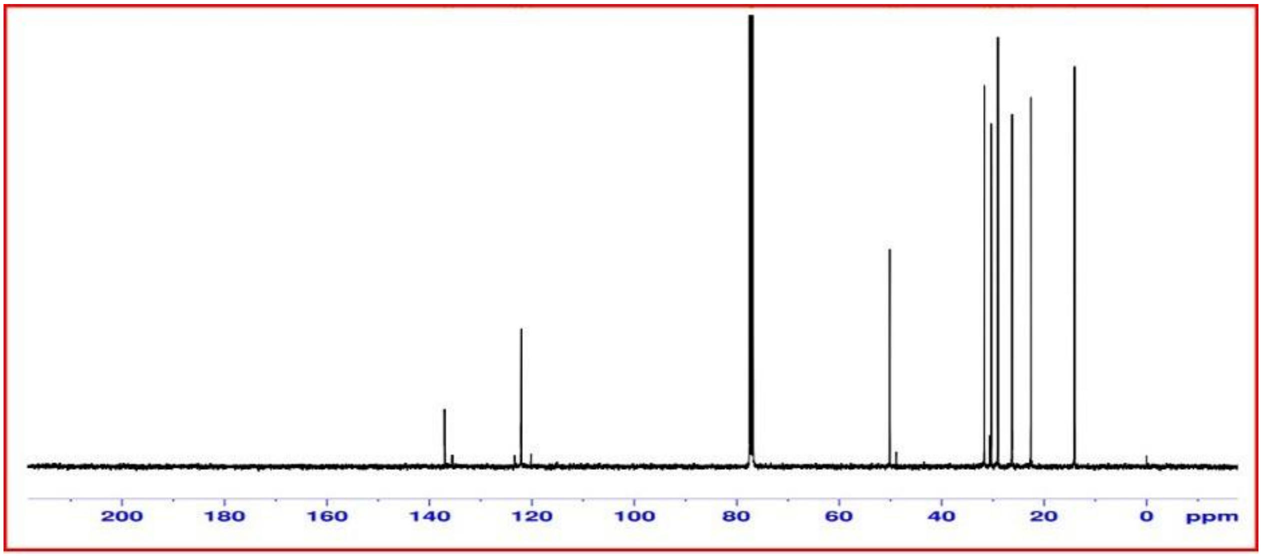


Figure 4. C^{13} NMR spectrum of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide.

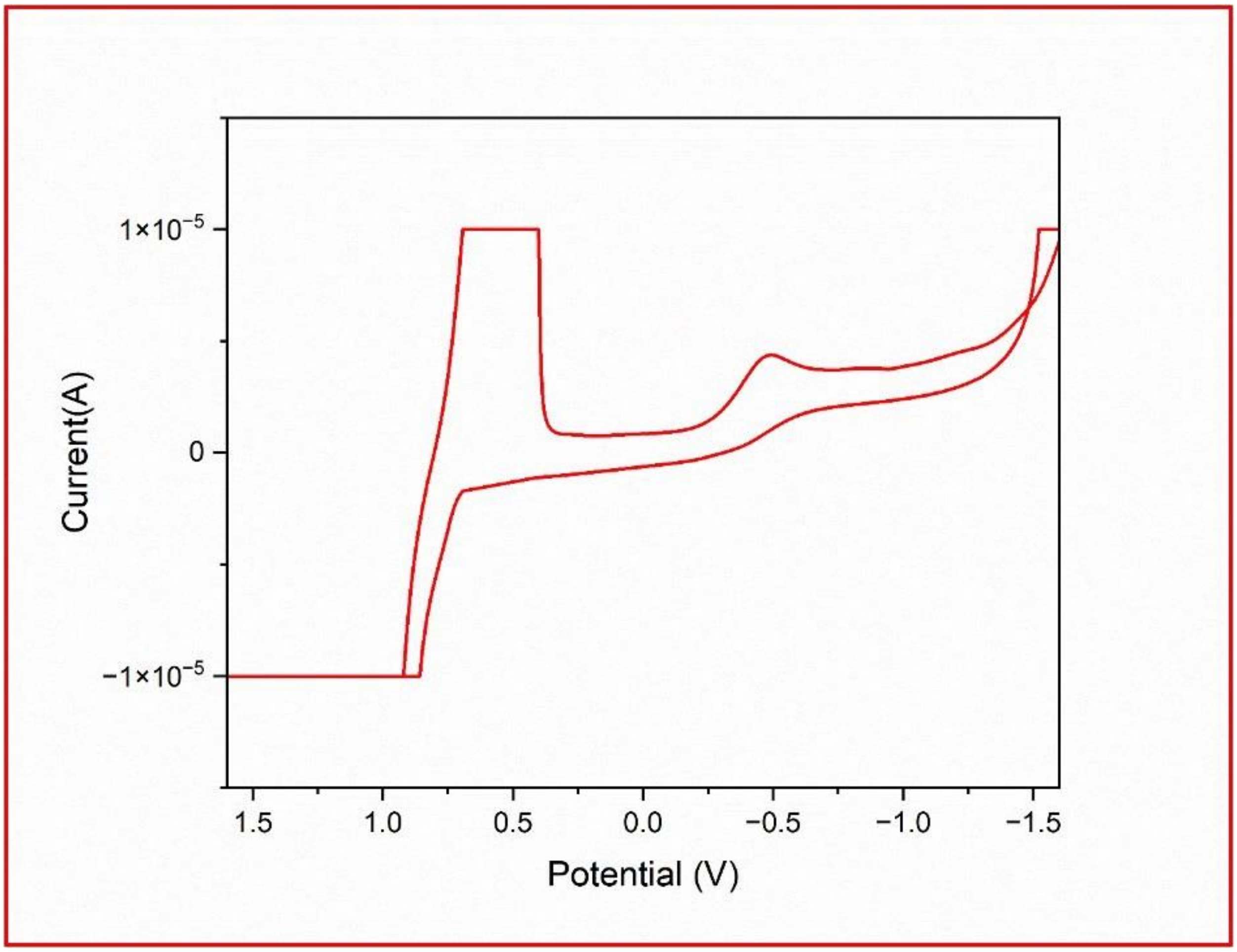


Figure 5. CV graph of 3,3'-(butane-1,4-diyl)bis(1-octyl-1H-imidazol-3-ium) bromide at $10mVs^{-1}$.

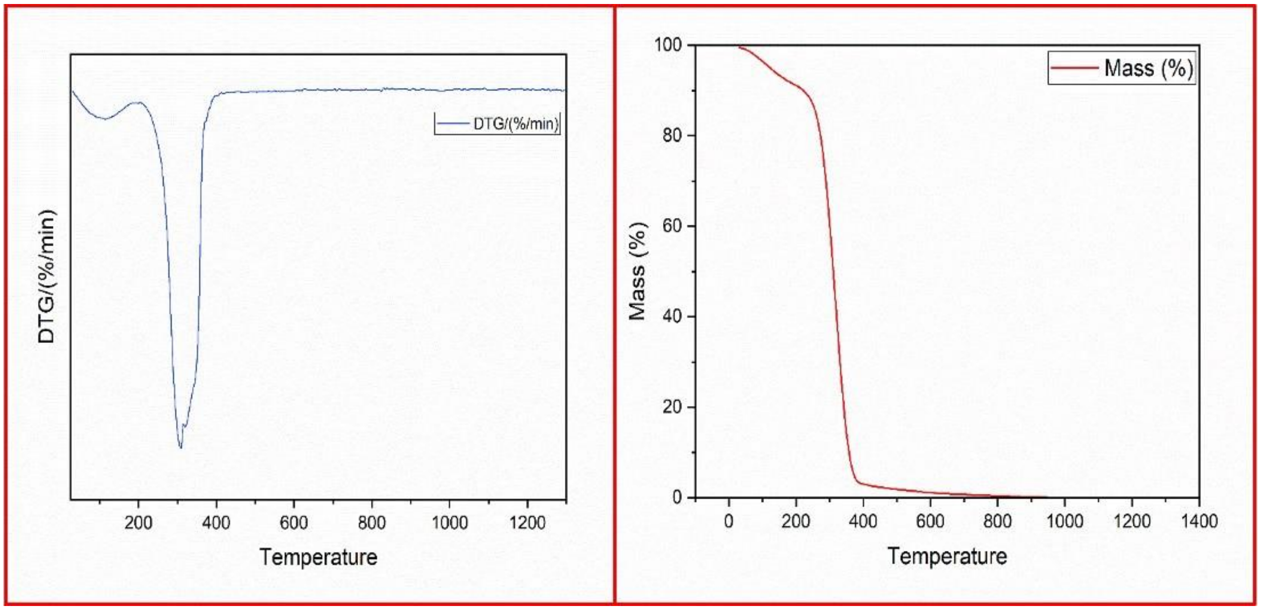


Figure 6. Thermal analysis of the synthesized dicationic ionic liquid, a) Differential Thermogravimetric (DTG) curve and b) Thermogravimetric Analysis (TGA) curve.

Table captions

Table 1. FTIR spectral peak assignments for the synthesized 1-octyl imidazole.

Table 2. FTIR spectral peak assignments for the synthesized dicationic ionic liquid.

Spectral peaks cm^{-1}	Assignment
663, 730 and 1077	CH_2-N bond stretching
1228, 1281	C-N aromatic bond stretching
1375	C-H bond rocking
1464	C-C aromatic stretching
1507	C=N aromatic stretching
2855, 2925	Aliphatic C-H stretching

Table 1. FTIR spectral peak assignments for the synthesized 1-octyl imidazole.

Spectral peaks cm^{-1}	Assignment
629,752	CH_2-N bond stretching
1159	C-N aromatic bond stretching
1458	C-C aromatic stretching
1562, 1632	C=N aromatic stretching
2924,2855	Aliphatic C-H stretching

Table 2. FTIR spectral peak assignments for the synthesized dicationic ionic liquid.