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FTIR Spectroscopic Studies and Structural Confirmations of Morpholinium Cation Based Ionic Liquids

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Abstract

A novel Series of morpholinium cation based ionic liquid was synthesized by the room temperature quaternization of morpholine with long chain alkyl bromides respectively. The synthesized ionic liquids were characterized by using Fourier Transform Infrared (FTIR) spectroscopic methods. The structures of all synthesized ionic liquids were in good agreement with obtained spectroscopic data which confirmed their formation. FTIR spectroscopy was used to analyse the functional groups and verify the structural features of the synthesized compound. The Fourier Transform Infrared Spectroscopy is widely employed for the identification of functional groups and confirmation of molecular structure in the chemical formulations. The Fourier Transform Infrared Spectroscopy gives information about molecular structure and functional groups which helps to confirm the compound identity. Fourier Transform Infrared spectroscopy was employed to characterize 4-butyl-4-hexyl morpholinium bromide. The spectrum exhibited characteristic absorption bands corresponding to C–H stretching, C–N^+ vibrations, and morpholine ring functionalities, confirming the successful formation of the target ionic liquid. These results provide qualitative evidence of the compound's structure and purity.

Keywords: FTIR, piperidinium, ionic liquid, functional group, synthesis and characterization.

Introduction –

Ionic liquids (ILs) have emerged as a revolutionary approach with distinct applications in both industry and academia [1]. ILs are molten state of solid having melting point below 100^0C [2].The ILs are molten salts or fused salts Some ionic liquids are liquid at room tempreture so they are called as the room tempreture ionic liquids (RTILs) [2].Ionic liquids are molten salts with tunable properties, widely used as environmentally friendly solvents and catalysts in modern chemical research[3].The ILs is also called as the "designer solvents". ILs are a class of organic salts consisting of bulky organic cations and inorganic or organic anions, which remain liquid at near-ambient temperatures. Their unique combination of negligible volatility, wide electrochemical window, and customizable properties makes them attractive as green solvents, catalysts, and functional materials in various chemical processes." The ILs is ionic species containing positively charged cation and negatively charged anion. ILs are molten state of solid that possesses compelling properties such as high thermal stability, lower vapor pressure, high liquidus range and promising electrochemical properties. The unique properties of ILs they are suitable for various field such as synthesis of nanoparticles, electrochemistry, space technology, biosensors, chemical engineering, enantioselective synthesis and drug delivery etc[5]. The ILs are also act as active pharmaceutical ingredients (APIs) they possess fascinating biological properties such as antimicrobial, antioxidants, acetylcholinesterase inhibition, serum albumin binding, antinociceptive, AMP deaminase inhibition and anticholirgenic activities etc[6].

Morpholine is a heterocyclic amine contains nitrogen and oxygen as a heteroatom having molecular formula (C_5H_9NO). Morpholine has unique structural and chemical properties that make it suitable as a **cation forming precursor.** *4-Butyl-4-hexyl morpholinium bromide is an example of a morpholinium-based ionic liquid, a class of salts that remain liquid at low temperatures[4]. These ionic liquids combine low volatility, high thermal stability, and tunable physicochemical properties, making them promising for applications in synthesis, catalysis, and electrochemical processes[7]. The structural features and functional groups of this ionic*

liquid were further confirmed by FTIR spectroscopy, supporting its successful synthesis and purity[5].

2) Experimental –

2.1) Materials -

The all chemicals and solvents are purchased from the commercial sources and used as without any purification. The completion of reaction is confirmed by analytical technique thin layer chromatography (TLC) was carried out on precoated aluminium sheet (Silica gel 60 F 254) and spot were visualized under ultraviolet light. The formed compound is characterized by Faurier Transform Infrared Spectroscopy was measured with a Bruker Alpha II Spectrophotometer. The samples were examined as KBr discs. The melting point was determined by melting point apparatus.4-Butyl-4-hexyl morpholinium bromide was synthesized according to literature procedure.

2.2) Synthesis

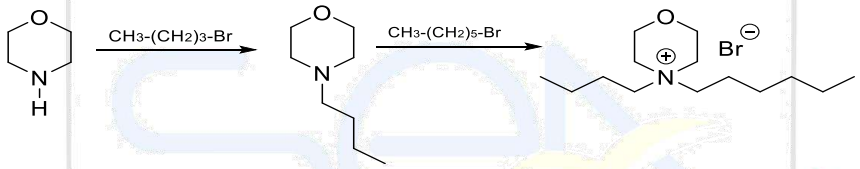
2.2.1) Preparation of 4-Butyl morpholine

Morpholine (5 mmol) was dissolved in toluene (100 ml) and stirred. Add stoichiometric amount of 1-bromo butane (5 mmol) and aqueous solution of sodium bicarbonate (2N). The reaction mixture was heated to the boiling point under reflux condition for 24 hr. The product solid NaBr was filtered and washed with toluene. Then the toluene from organic phase was removed by evaporation get 4-butyl morpholine.

2.2.2) Preparation of 4-butyl-4-hexyl morpholinium bromide –

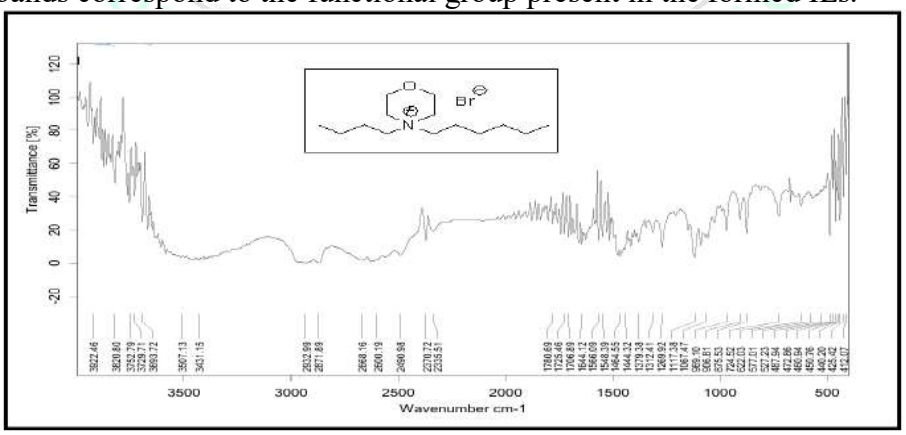
In a round bottom flasks added 50 ml acetonitrile and 4-butyl morpholine (3 mmol) was added and vigorously stirred then add hexyl bromide (3 mmol).The reaction mixture was stirred at its boiling point under reflux condenser for 24 hr. The solvent acetonitrile is removed under reduced pressure. The formed precipitate was filtered, washed with ethyl acetate and dried at 60^0C under reduced pressure.

3) Reaction Scheme –



4) Analysis –

The synthesized 4-butyl-4-hexyl morpholinium bromide is characterized by FTIR spectral analysis. The FTIR spectroscopic analysis was carried out in Bruker Spectrophotometer. The spectrum was recorded transmittance verses wave number having range 3500 – 500 cm^{-1} under ambient condition[8]. The obtained FTIR spectrum was analyzed to identify the characteristics absorption bands correspond to the functional group present in the formed ILs.



The Fourier Transform Infrared (FTIR) spectrum of the synthesized ionic liquid shows several characteristic absorption bands confirming the presence of expected functional groups. A broad

absorption band observed in the region **3500–3400 cm⁻¹** is attributed to **O–H/N–H stretching vibrations**, which may arise from hydrogen bonding or trace moisture due to the hygroscopic nature of ionic liquids. The strong peaks at approximately **2922 cm⁻¹ and 2871 cm⁻¹** correspond to **aliphatic C–H stretching vibrations** of $–CH_2$ and $–CH_3$ groups, indicating the presence of alkyl chains within the cation structure.

The absorption bands appearing near **1725 cm⁻¹** suggest **C=O stretching**, while the band around **1664 cm⁻¹** can be assigned to **C=C stretching or amide-related vibrations**. Peaks in the region of **1568–1434 cm⁻¹** are associated with **N–H bending and CH_2 deformation**, supporting the existence of nitrogen-containing cations. Furthermore, the bands detected between **1312 and 1267 cm⁻¹** are characteristic of **C–N stretching**, which is a key indicator of ammonium or morpholinium-based ionic liquids.

The strong absorptions observed in the range of **1187–1068 cm⁻¹** are attributed to **C–O–C stretching vibrations**, confirming the presence of an ether linkage consistent with the morpholine ring structure. Additional peaks below **1000 cm⁻¹** correspond to **C–H out-of-plane bending and ring deformation modes**. The bands located in the lower frequency region (**600–400 cm⁻¹**) are typically related to skeletal vibrations and possible anion-related stretching modes.

Overall, the FTIR spectrum verifies the formation of the ionic liquid through the presence of diagnostic bands corresponding to alkyl groups, C–N bonds, and ether functionalities. The observed vibrational features are in good agreement with previously reported spectra of morpholinium-based ionic liquids, thereby supporting the successful synthesis of the targeted compound.

Conclusion –

The FTIR analysis confirmed the successful formation of morpholine cation based ionic liquids through the identification of characteristic vibrational bands corresponding to the morpholine ring and associated functional group. The presence of strong absorption peaks related to C-N, C-O-C and aliphatic C-H stretching indicates the structural integrity of the cation, while the distinct bands of the counter anions further support effective ionic interaction. Overall, the FTIR results validate the proposed molecular structures and confirm that the adopted synthesis method is reliable for producing stable morpholine based ionic liquids. These findings highlight their potential suitability for advanced applications in electrochemistry, catalysis and green solvents systems.

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