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Microwave Assisted Synthesis of Ionic Liquids with its Applications as CO_2 Capture & Antimicrobial Activity

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Abstract:

Synthesis of ionic liquids by using microwave irradiation shows application of green chemistry principles for the development of cleaner processes. Microwave irradiation is rapid, safe, highly efficient, solvent free process to obtain various ionic liquids. By using cations like (N-heterocycles) imidazole, triazole, pyridine, piperidine, pyrrolidine and guanidine with fluoroboric acid different ionic liquids are prepared. In this work Ionic liquids were synthesized and characterized by using FTIR & 1HNMR spectra. These Ionic liquids further utilized for carbon dioxide capture and also studies for its antimicrobial activity against *Escherichia coli* (Gram-negative) and *Staphylococcus aureus* (Gram Positive) pathogenic bacteria using disk diffusion assay (Modified Kirby- Bauer Test). AD5: n -hydrogen pyrolidinium tetrafluroborate shows very good activity of capture for CO_2 absorption.

Keywords: Ionic Liquids, microwave irradiation, fluoroboric acid, Antimicrobial activity, N-heterocycles.

Introduction:

Ionic liquids are considered as good molecular and green solvent in the replacement to commonly used volatile organic solvents (1). They have been utilized for both academic and industrial applications associated with specific biological, chemical and physical properties. Normally the ionic liquids are accepted in the ionic form. However, it is also distinguished in classical form as fused salts, molten salts, liquids, organic salts and many more (2-3).

Ionic liquids are usually defined as organic and inorganic salts with very low melting point (lower than 100 degree Celsius) , comprising cations and anions. The

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introduction of novel ionic liquids shows characteristic features over traditional ones. Ionic liquids are used as the best reaction solvents for various reactions such as alkylation, acidic hydrolysis, Beckmann rearrangement and polymerization (4-5).

The ionic liquids as green solvents or catalysts mostly useful for various synthesized products. Ionic liquids have negligible vapor pressure, low viscosity, long range thermal stability, non-flammability and very low corrosivity related to mineral acids and base (6).Development of economical and eco-friendly techniques not only improves the yields but also decreases the generation of waste to minimize the pollution .(7). With respect to cationic composition for e.g.: ammonium, imidazolium, morpholinium, phosphonium, piperidinium, pyridinium, pyrrolidinium and sulphonium salts reactions can be tailored.

Martyl J Earle mentioned first time ionic liquid as a green solvent. Apart from electro chemistry ionic liquids can be used for various chemical reactions due to their exceptional properties (8). The ethyl ammonium nitrate is the first ionic liquid introduced in the year 1914 having melting point 12 degree Celsius. Considering numerous properties of ionic liquids attention has been drawn in the bio-medical research and in drug formulations (9). Several years ago ionic liquid salts were active pharmaceutical ingredients which were proposed to become an alternative to common crystalline salt. Ionic liquids are employed in the synthesis of heterocyclic molecules such as imidazole's, furanynes, oxazole's, quinolines and others which are exploited in biology and medicine. Recent Research focuses on the tuning of the biological properties to design novel ionic liquids based anti-microbial materials (10-11).

Ionic liquids and ionic liquid-based materials are reviewed with focus on anti-microbial properties applied to water treatment, air filtration, food packaging and anti-corrosion (11-12). Due to unique properties of ionic liquids attention has been focused on their application deal with the challenge of bacterial resistance (13). Economical and environmental aspects are the main motivation for rsearch on energy efficient processes and search for environment friendly materials for CO_2 capture(14,16-17)

Synthesis of Ionic liquids (ILs)

Before describing potential applications of ILs, their synthesis and purification needs to understood . ILs are synthesized by using many method like Liquid precipitation, ultrasonic synthesis, hydrothermal method, heating method,microemulsion method, microwave assisted method etc.

We have adopted microwave assisted method for preparation of ionic liquid of fluroborate. This method have shown several advanteges compared to convential synthetic procedure. Microwave irradiation is rapid, safe,highly efficient, solvent free process to obtain various ionic liquids. By using cations like imidazole,triazole,pyridine,piperidine, pyrrolidene and guanidine with fluroboric acid different ionic liquids are prepared.

CO_2 capture procedure

IL of fixed weight and volume was purged with CO_2 gas for 10 minutes. By using gravimetric method difference in weight

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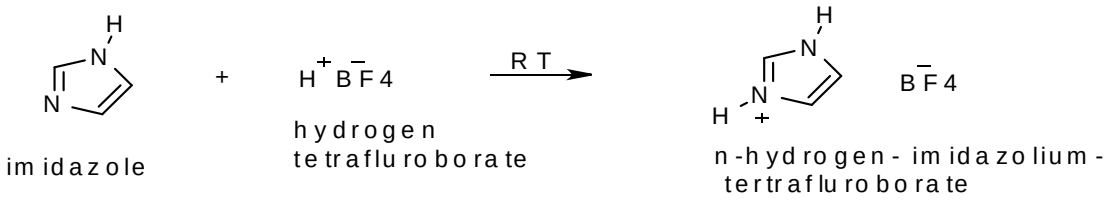
was observed . It also gives change in structure in NMR data .

Antimicrobial activity

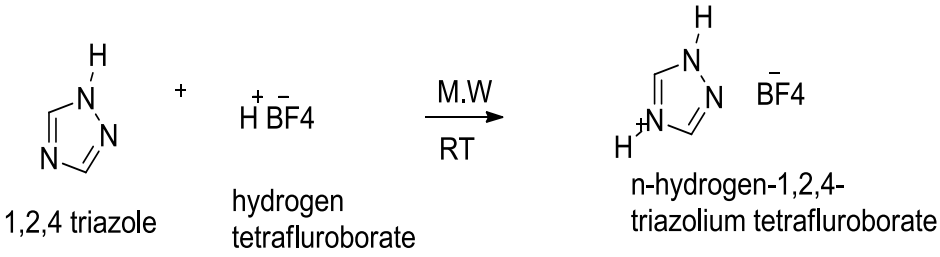
Theantibacteria activity of the ionic liquids (AD3, AD4 and AD5) was examined against *Escherchia coli* (Gram-negative) and *Staphylococcus aureus*(Gram Positive) pathogenic bacteria using disk diffusion assay (Modified Kirby- Bauer Test) (Reference 1). In this aaasy approximately 20 mL of sterile molten nutrient agar was poured into the petri dishes and cooled and solidified. The 0.1 ml of $O.D_{600nm}$ bacterial suspension was spread over the solidified medium and the disks soaked with suitable concentration of ionic liquids were placed on the plate using sterile forecep aseptically.(15)The sterile distilled water soaked disk was was used as negative control. The plates were then incubated at 37^0C for 24 hr in incubator. The inhibition zone formed round each disk was measured in millimeter.

Reactions are shown as follows.

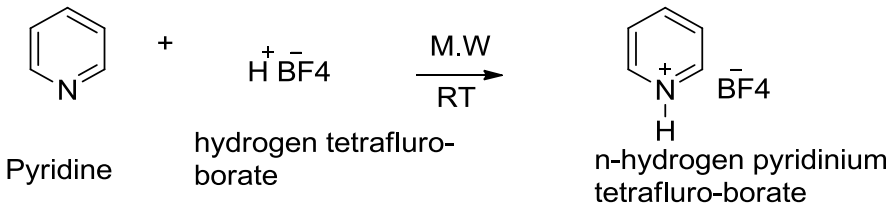
Reaction 1 : Formation of n hydrogenimidazoliumtetrafluroborate



Reaction 2 : Formation of n hydrogen triazoliumtetrafluroborate



Reaction 3 : Formation of n hydrogen pyridinium tetrafluroboric acid



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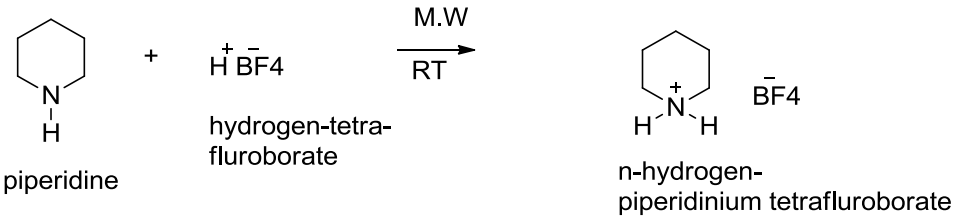
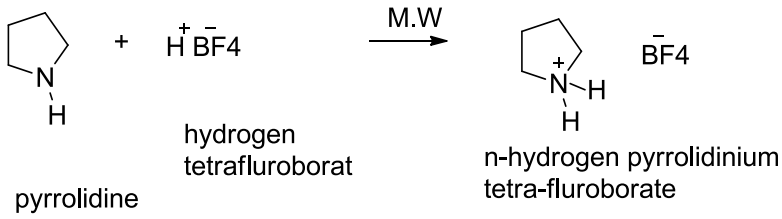
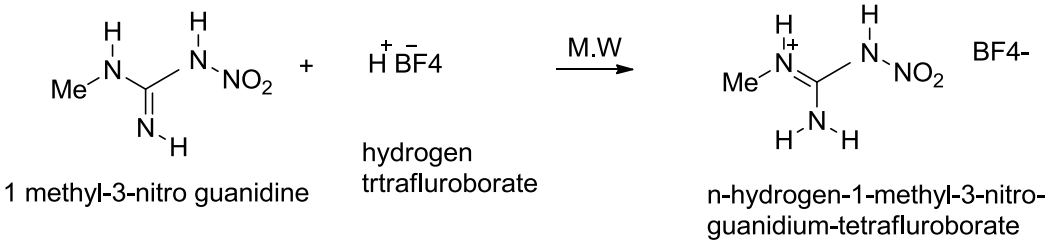
Reaction 4 : Formation of n hydrogen piperidinium tetrafluroborate



Reaction 5 : n hydrogen pyrolidinium tetrafluroborate



Reaction 6 :n hydrogen 1 methyl 3 nitro guandiniumtetrafluroborate



RESULTS:

IR , NMR DATA AND PERCENTAGE YIELD FOR AD1 to AD6

AD1 : n hydrogen imidazolium tetrafluroborate

IR cm $^{-1}$ 3571 ,3162 ,1446

NMR ; 1H NMR CDC l_3 , δ ppm 7 ,7.1 ,7.5 ,7.7

yield - 92%

AD2: n hydrogen triazolium tetrafluroborate

IR cm $^{-1}$ 3593 ,3156 ,1631

NMR ; 1H NMR CDC l_3 , δ ppm 1.3, 8.2

yield - 90 %

AD3: n hydrogen pyridinium tetrafluroborate

IR cm $^{-1}$ 3394 ,1633

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NMR ; 1H NMR CDC l_3 , δ ppm 7.2, 7.3 ,7.7, 8.6

yield - 90 %

AD4: n hydrogen piperidinium tetrafluroborate

IR cm $^{-1}$ 3601

NMR ; 1H NMR CDC l_3 , δ ppm 1.5,2.2,3.0

yield - 92 %

AD5: n hydrogen pyrolidinium tetrafluroborate

IR cm $^{-1}$ 3397

NMR ; 1H NMR CDC l_3 , δ ppm 2.0,3.3, 7.2

yield - 92 %

AD6: n hydrogen 1 methyl ,3 nitro guanidinium tetrafluroborate

IR cm $^{-1}$ 3306 ,1567

NMR ; 1H NMR CDC l_3 , δ ppm 1.1 ,3.2 ,3.3 ,6.2 , yield - 80 %

DISCUSSION

REACTION 1 : Explains formation of n-hydrogen Imidazoliumtetrafluroborate which is supported by the values given in table no .1 ^{1} H NMR data gives peak at 7,7.1,7.5 and 7.7 which confirms the structure of the above mentioned compound named as **AD1.** Tables of FTIR , NMR and plots are listed in supporting data.

REACTION 2 Explains formation of n hydrogen Triazoliumtetrafluroborate which is supported by the values given in the table no . 2 **.** ^{1} H NMR data gives peak at 1.3 and 8.2 which confirms the structure of the above mentioned compound named as **AD2**

Similar fitting of NMR peaks for all other ionic liquids was done to verify their structures and named as **AD3** (n hydrogen pyridiniumtetrafluroboric acid) , **AD4** (n hydrogen piperidiniumtetrafluroborate) , **AD5**(n hydrogen Pyrolidiniumtetrafluroborate) and **AD6** (n hydrogen 1 methyl , 3 nitro guanidiniumtetrafluroborate) respectively .

^{1} H NMR data gives additional peak at 1.6,1.8,3.2, and 4.3 after purging the CO_2 gas in the **AD4** ionic liquid . which confirms that there is a change in the structure of the above mentioned compound due to the reaction between CO_2 and ionic liquid under observation . This could be one of the important application as CO_2 capture in current scenario .

^{1} H NMR data gives additional peak at 1.2, 1.8 ,1.9, 2.2 ,2.3 , 2.6, 3.3 3.4 and 3.5. after purging the CO_2 gas in the **AD5** ionic liquid . which confirms that there is a change in the structure of the above mentioned compound due to the reaction between CO_2 and ionic liquid under observation . This could be one of the important application as CO_2 capture in current scenario . (Supporting data)

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IR OF IONIC LIQUIDS

From FTIR figure no 10(supporting data) , frequencies obtained at 3162 corresponds to NH streching ,3195 and 1446 cm $^{-1}$ which explainspresence of , C -H and C-N (C ≡N) bond which confirms structure of formation of **AD1** (n hydrogen Imidazoliumtetrafluroborate) .From FTIR figure no 11 , frequencies obtained at 3593 corresponds to NH streching , 3156 and 1631 cm $^{-1}$ which explains presence of , C -H and C-N (C ≡N) bond which confirms structure of formation of **AD2** (n hydrogen Triazoliumtetrafluroborate)

From FTIR figure no 12 , frequencies obtained at 3394 corresponds to NH streching and 1633 cm $^{-1}$ which explains presence of C-N (C ≡N) bond Which reveals structure of formation of **AD3** (n hydrogen pyridiniumtetrafluroborate)

From FTIR figure no 13 , frequencies obtained at 3601cm $^{-1}$corresponds to NH streching bond Which reveals structure of formation of **AD4** (n hydrogen piperidiniumtetrafluroborate) From FTIR figure no 14 , frequencies obtained at 3397cm $^{-1}$ corresponds to NH streching bond .Which reveals structure of formation of **AD5** (n hydrogen pyrolidiniumtetrafluroborate) From FTIR figure no 15 , frequencies obtained at 3306 corresponds to NH streching and 1567 cm $^{-1}$ which gives idea about presence of NO_2 group. Which reveals structure of formation of **AD6** (n hydrogen 1 methyl 3 nitro guanidiumtetrafluroborate)

ANTI BACTERIAL ACTIVITY:

Anti-bacterial activity against e.coli and staphylococcus aureus. Using agar diffusion array(disc diameter- 6 mm)

Table 1. Antibacterial activity of ionic liquid using disc diffusion assay against *E.coli* and *Staphylococcus aureus.*

Sample code	Average Zone Diameter against *E.coli* (mm)	Average Zone Diameter *against Staphylococcus aureus* (mm)
AD3	15	16
AD4	13	17
AD5	17	16

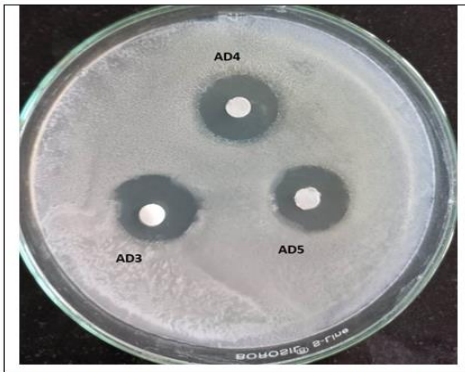

Fig.1 Inhibition zones of ionic liquid (AD3, Ad4 and AD5) against *Staphylococcus aureus* (Gram-positive) bacteria

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Antimicrobial Acivity results

The Table 1 and figure 1 shows the antibacterial activity of ionic liquid tested in this study.

The antibacterial activity of all three ionic liquid shows similar activity hence it can be concluded that they are active against both the Gram positive and Gram negative pathogenic bacteria selected. This is advantageous as drugs made using these liquid may show broad spectrum of activity. Result mentioned in above table gives information about efficiency of ionic liquid against microorganisms tested .

AD3 andAD4 give good result for s.cocci while **AD5** is actingas potential drug against antimicrobial activity for both e. coli and s. cocci .

Conflict of Interest: Authors declares no conflict of interest

Authors Contributions: Each and every author had contributed to the manuscript.

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