

**MICROWAVE-ASSISTED SYNTHESSES,
CHARACTERIZATION AND SOME
UTILIZATION OF ROOM-TEMPERATURE
IONIC LIQUIDS**

Ph D (DISSERTATION)

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JUNE, 2005

ABSTRACT

Room Temperature Ionic Liquids (RTILs) are salts, consisting solely of ions and existing as liquid over a wide range of temperatures, including room temperature. The six types of room-temperature ionic liquids: moisture-sensitive 1-butylpyridinium chloroaluminate (BPC- AlCl_3), 1-butyl-3-methylimidazolium chloroaluminate (BMIMC- AlCl_3) and moisture-stable 1-butylpyridinium tetrafluoroborate (BPC- BF_4) (hydrophilic), 1-butyl-3-methylimidazolium tetrafluoroborate (BMIMC- BF_4) (hydrophilic), 1-butylpyridinium hexafluorophosphate (BPC- PF_6) (hydrophobic), 1-butyl-3-methylimidazolium hexafluorophosphate (BMIMC- PF_6) (hydrophobic), were synthesized via quaternary salts [1-butylpyridinium chloride (BPC) and 1-butyl-3-methylimidazolium chloride (BMIMC)]. BPC and BMIMC, two precursors of some room-temperature ionic liquids, were synthesized under microwave radiation in a closed vessel. The optimum conditions of BPC using microwave digester Q15 were found to be 300 W, 15 bar, 200 °C and 60 min and (300-400) W, 75 bar, 200 °C and 25 min using microwave digester An. Paar. The optimum conditions of BMIMC were found to be 300 W, 4 bar (57 psi), 150 °C and 25 min with Q15. Compared to the conventional method, the microwave technique indicates two main advantages: shortening the reaction time and an enhancement in yield with minimum impurities. Using the microwave method, the reaction time of BPC was drastically reduced from 72 hrs to 1 hr and substantial yield of (65%) was obtained. Similarly, the reaction time of BMIMC was drastically reduced

from 48 hrs to 25 min and very significant yield of (91%) was obtained. These two precursors were characterized by using ^1H NMR, ^{13}C NMR, FT-IR and UV methods. Room-temperature ionic liquids were also characterized by using EDXRF, FT-IR and UV methods. Then physicochemical properties (melting point, density, viscosity, conductivity and solubility) of these ionic liquids were also investigated. The temperature range of these ionic liquids were found to be 19-240 °C (BPC- AlCl_3), >-20-250 °C (BPC- BF_4), 76-270 °C (BPC- PF_6), -80-263 °C (BMIMC- AlCl_3), >-20->300 °C (BMIMC - BF_4) and 13->300 °C (BMIMC- PF_6). Generally, the density of produced ionic liquids was found to be between 1.2 to 1.5 g cm^{-3} at ambient temperature. Moreover, they are excellent solvents for a wide variety of organic and inorganic species. The catalytic activity and reusability of synthesized ionic liquids (BPC- BF_4 , BPC- PF_6 , BMIMC- BF_4 , BMIMC- PF_6) were studied in model chemical reactions. They were used as catalysts and/or solvents in esterification and lipase-catalyzed transesterification reactions. In esterification reaction, the respective yields of ester 95%, 93%, 91% and 90% were achieved within (6-7) hrs using various ionic liquids. Ester was identified by using GC-MS and FT-IR methods. In lipase catalyzed transesterification reaction, BMIMC- BF_4 and BMIMC- PF_6 ionic liquids can be reused (3) times, giving 44%, 35%, 27% and 47%, 38%, 28% yields of ester respectively.

Keywords: *Room-Temperature Ionic Liquids, Microwave, Esterification, Transesterification,*

Lipase