



# Synthesis of silver nanoparticle in imidazolium and pyrrolidinium based ionic liquid reverse micelles: A step forward in nanostructure inorganic material in room temperature ionic liquid field

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## ABSTRACT

Two silver salts, silver tetrafluoroborate and silver trifluoromethanesulfonate were dissolved in two different room temperature ionic liquids (RTILs), 1-Butyl-3 methyl imidazolium tetrafluoroborate ([Bmim][BF<sub>4</sub>]) and 1-Butyl-1 methyl pyrrolidinium trifluoromethanesulfonate ([Bmpy][Tfms]). Triton x-100 (TX-100) surfactant and cyclohexane as nonpolar medium are used to dissolve these RTILs to create reverse micellar system. Pure reverse micellar system is characterized by dynamic light scattering (DLS) measurement. These pure RTIL reverse micellar systems are used to prepare stable silver nanoparticle solution without using any other auxiliary solvent in the whole process.

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## 1. Introduction

Room temperature ionic liquids [RTILs] nowadays are very fascinating and most interesting in the field of solvent chemistry. They are solely composed of ions and are liquids at room temperature (temperature below 100 °C). The reason behind this uncommon behavior is the presence of the sterically mismatched ions [1,2] that resist solid formation. They are normally tunable, designer, nontoxic or very less toxic and nonflammable with essentially zero volatility. They have very long liquid range. For these reasons, they are considered as “green” solvent [3–6]. It has been used in synthesis, catalysis, solar cell, desulfurization of fuel and in many other applications [7]. Despite these rich features, sometimes, chemists have to face a general problem of solubility of the reagents during its application as a substitute of the conventional solvent in its pure state. In the organic chemistry field (where most of the solutes are apolar or very less polar), the problem has been solved by preparing specific ionic liquid with desired solubility criteria or by using the concept of microheterogeneous media which creates a hydrocarbon region by creating normal micellar surfactant assemblies or by creating an ionic liquid-in-oil microemulsion [8,9]. Recently this concept has also been extended to the super critical carbon dioxide continuous media where both the solvents used are considered to be as attractive “green”

solvents [10]. But such application of RTILs in inorganic material chemistry is very rare. Inorganic materials and salt are mostly ionic in nature. Any type of ionic compound cannot be dissolved in any type of ionic liquid. In many cases, we will get almost insoluble nature or very less solubility which will be insufficient for the practical application purpose. Some efforts have been made by few groups from the beginning of this century in this new direction. But they mainly devoted their work in creating new system by applying new RTILs in different media as a new component, showing their phase behavior or simply characterized their properties by applying different photo-physical studies [11–15]. In very recent times, a few attempts have been made to extend the RTIL field to inorganic and nanomaterial chemistry [16]. Zhao et al. [17] reported the synthesis of nickel oxide nanoparticle in bmimBF<sub>4</sub> ionic liquid but they used water to make both the reactant precursor solution (nickel salt and ammonia) and added that solution in the RTIL. Dupont et al. [18] and Wang et al. [19] have applied some better concept, they used transition metal complex with organic ligand and transition metal salt with crystal water as the precursors. Still they have to add dichloromethane as their cosolvent solvent to dissolve the precursor. More over they had to use very drastic conditions like high temperature, pressure and vigorous stirring for long time to disperse the precursor solution in the media. Another drawback of this procedure has already been mentioned by Fonseca et al. that ionic liquid decomposition occurs in the presence of both water and transition metal complex precursor [20]. Tsuda et al. [21] used somewhat different approach from the others for their reduction procedure, they used radiation irradiation

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method but in this procedure reduction was occurring with the decomposition of RTIL by radiation used. All these works contributed a lot in this field but can't solve the problem impeccably because they all have to compromise with the system purity (used other solvent with RTIL) and correctness to achieve the goal.

In this communication we are going to report a method which is simple, does not require high pressure, temperature or vigorous stirring for very long hours or any specialized equipments and more importantly in this procedure, one doesn't have to achieve the goal at the cost of his system's correctness and purity.

## 2. Materials and methods

### 2.1. Solubilization of metal precursor in the respective RTIL and preparation of stock solution

In this work, we used two RTILs. They belong to two different categories and have different anionic counterpart. The first one belongs to imidazolium based RTIL having tetrafluoroborate as its anion counterpart [1-Butyl-3 methyl imidazolium tetrafluoroborate ([Bmim][BF<sub>4</sub>])], and the other one belongs to pyrrolidinium based RTIL with trifluoromethanesulfonate anion counterpart [1-Butyl-1 methyl pyrrolidinium trifluoromethanesulfonate ([Bmpy][Tfms])]. For reverse micelle preparation, we used TX-100 as the surfactant in cyclohexane, which is an extensively studied and a well-known surfactant for its capability of forming RTIL microemulsion [12c,22]. As the buck stops with the solubility problem of the metal precursor salt in RTIL, we are clarifying that concept first. Generally metal salt solubilization process in polar solvent is achieved by separating solute particle from its solid state. The positive ion (metal ion) of the solute is attracted by the negative end of the solvent molecules and dissolution takes place when the solvent molecules are able to pull the metal ion out of their solid structure. This dissolution process was not occurring in RTIL. The reason may be the different nature of RTIL from the general nature of the dipolar solvent molecules, its independent ionic character or the larger size of the ion counterpart or both. The only way to solve this problem was to enhance the force of attraction exerted by the solvent molecules on the particle of the solid solute. For that purpose we

selected the silver salt with tetrafluoroborate anion (AgBF<sub>4</sub>) and checked its solubility in [Bmim][BF<sub>4</sub>]. We got good results, sufficient solubility for the application purpose. For further verification, we extended the concept in [Bmpy][Tfms] and used silver trifluoromethanesulfonate (AgTfms) as precursor salt. In that case also, we got very good solubility. The idea behind the use of metal ion precursor having the same counteranion as that of the RTIL was that, in that situation not only the anion part of the solvent exerts attractive force for the metal ion but also the positive part of the solvent also exerts attractive force on the anion part of the metal precursor. Combination of both the force will be much more helpful to achieve the goal because ultimate purpose of both the force will be the dissolution of the solid precursor in the ionic liquid. Using this method we prepared two stock solutions of silver ion in two different RTIL, [Bmim][BF<sub>4</sub>] and [Bmpy][Tfms] having strength 0.18(M) and 0.1(M) respectively.

### 2.2. Selection of reducing agent

Finally we trickily solved the problem of selecting the suitable reducing agent for the reduction of silver ion by selecting molecular hydrogen as the reducing agent because recently molecular hydrogen has successfully been used as an environmentally benign reducing agent [23].

### 2.3. Reverse micellar solution preparation

Reverse micellar solutions were prepared by weight fraction. [Bmim][BF<sub>4</sub>]/TX-100/cyclohexane system was created using 5, 60 and 35 weight percentages respectively. In [Bmpy][Tfms]/TX-100/cyclohexane system, the respective weight percentages were 8, 58 and 34. Details of the reagents and their purifications are mentioned in the supporting information. We prepared 5 g reverse micellar solution for each type of reverse micellar system. At the first step, we weighted the required amount of TX-100 surfactant in a 10 ml volumetric flask then we added the required amount of cyclohexane by direct weighing. In the next step we homogenized the mixture by simple hand shaking. Then we calculated the required volume of RTIL from its density and added that volume cautiously by a microliter syringe to achieve the

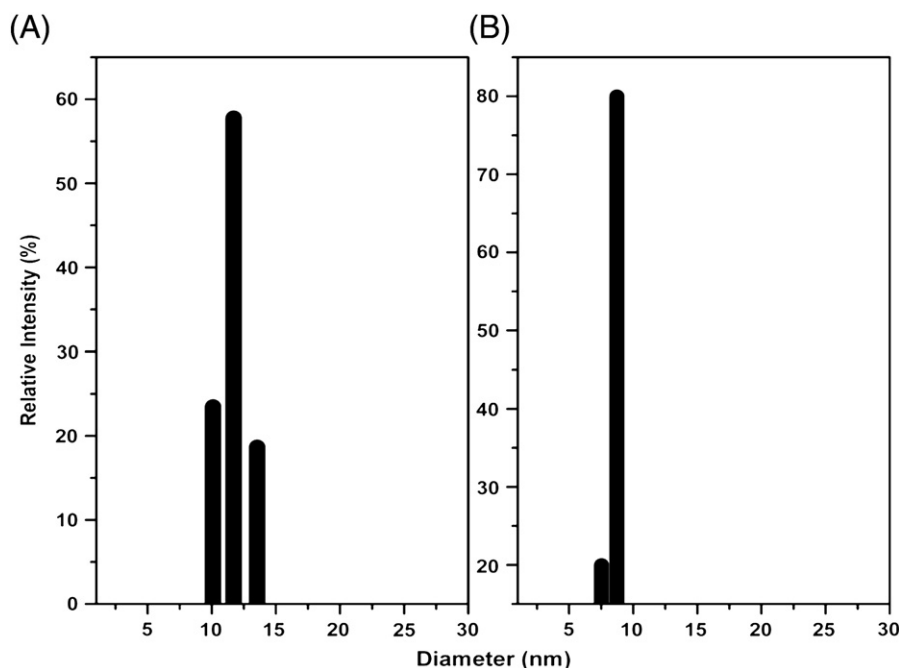


Fig. 1. DLS size distribution histogram (A) [Bmpy][Tfms]/TX-100/cyclohexane having weight percentages of 8, 58 and 34, and (B) [Bmim][BF<sub>4</sub>]/TX-100/cyclohexane having weight percentages of 5, 60 and 35.

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