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Original Research Article

Nickel Nanoparticles Synthesized in 1-Ethyl-3-Methylimidazolium Methanesulfonate and 1-Butyl-3-Methylimidazolium Methanesulfonate: A Comparative Study

Chioma C. Nwakanobi[1]*, Christopher Ejeomo[2], Blessing Oby Okoye[3], Isaac Akpan[4], Joy Akilapa Moses[5]

[1]Department of Chemistry, School of Integrative Biological and Chemical Sciences, University of Texas Rio Grande Valley
[2]Department of Industrial Chemistry, Faculty of Natural and Applied Science, Michael and Cecilia Ibru University, Agbarha-Otor, Ughelli, Delta State, Nigeria
[3]Department of Chemistry, University of California Davis, USA
[4]Chemistry Department, Saint Louis University, USA
[5]Department of Chemistry, University of Iowa, USA

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***Corresponding author:** Chioma C. Nwakanobi
Department of Chemistry, School of Integrative Biological and Chemical Sciences, University of Texas Rio Grande Valley

Abstract

The synthesis of metal nanoparticles (NPs) in ionic liquids (ILs) solvent has been adopted as a greener alternative to conventional organic solvent reaction media, due to their environmentally benign and tunable physicochemical properties benefits. This study aimed to synthesize and characterize nickel nanoparticles (NiNPs) using imidazolium-based ionic liquids (1-ethyl-3-methylimidazolium methanesulfonate ([EMIM]MS) and 1-butyl-3-methylimidazolium methanesulfonate ([BMIM]MS) as both solvent and stabilizing agents, via a chemical reduction pathway. The NiNPs were characterized by UV-Visible (UV-Vis) spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy. The UV-Vis analysis showed broad surface plasmon resonance bands between 300 and 350 nm, confirming NiNP formation, with spectral shifts revealing smaller particles in [EMIM]MS compared to [BMIM]MS. FTIR spectra revealed characteristic bands for O–H, C–H, C–C, and C–N vibrations, indicating strong coordination between ionic liquid molecules and the nickel surface. SEM micrographs showed that NiNPs synthesized in [EMIM]MS were smaller (45–60 nm), spherical, and uniformly dispersed, and those obtained in [BMIM]MS were larger (60–80 nm) with slight aggregation. Synthesis was also done in ethylene glycol and the resulting NPs were irregular and aggregated. These results confirm that imidazolium-based ILs enable controlled synthesis and stabilization of nickel nanoparticles, providing a green and efficient route for nanomaterial production.

Keywords: Nickel nanoparticles, Ionic liquids, Imidazolium-based solvents, FTIR, SEM, UV–Visible spectroscopy.

INTRODUCTION

Ionic liquids (ILs) are generally molten salts composed of organic cations and organic or inorganic anions, with characteristic melting points below 100°C [1,2]. Owing to their distinctive physicochemical properties such as negligible vapor pressure, nonflammability, thermal stability in the liquid phase, strong solvating ability for organic, inorganic, and polymeric materials, and high ionic conductivity, they are therefore considered environmentally friendly and have the potential to facilitate the creation of novel materials unattainable with conventional solvents or water [2]. These features have supported their expanding use as green solvents and industrial scale catalysts. Structurally, most ionic liquids consist of bulky organic cations paired with inorganic anions, forming crystalline lattices with low lattice energies, which enables them to remain liquid at or near room temperature. One notable application of ionic liquids is their use as substitutes for conventional organic molecular solvents. They have been employed in various chemical, catalytic, and biological reactions [3], inorganic synthesis [4], gas separation and purification [5], and in the removal of contaminants from aqueous streams [6]. Various studies have shown that ionic liquids (ILs) provide excellent opportunities as environmentally friendly solvents for the synthesis of metal nanoparticles [7,8]. Imidazolium-based ionic liquids (ILs) have been widely employed for

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the synthesis and stabilization of metal nanoparticles (M-NPs) [9].

Previous work demonstrated that nickel nanoparticles (NiNPs) can be successfully synthesized in imidazolium ILs via decomposition of organometallic precursors. For example, in a classic study $Ni(NBD)_2$ (NBD = 1,5-cyclooctadiene) dissolved in 1-alkyl-3-methylimidazolium ionic liquids yielded face-centered cubic NiNPs with mean diameters of ~5 ± 1 nm and narrow size distributions [10]. The IL medium not only facilitates controlled nucleation and growth, but also stabilizes the nanoparticles against agglomeration via electrostatic and steric effects arising from the IL's ionic double layers and alkyl-chain organization [11]. Despite this progress, there remains limited comparative data on how subtle variations in IL composition affect NiNP formation, particularly the influence of cation alkyl-chain length (e.g., ethyl vs butyl) in methanesulfonate-based ILs on particle nucleation, growth kinetics, size distribution, morphology, and stability.

Therefore, the present study aims to synthesize and characterize nickel nanoparticles (NiNPs) using imidazolium-based ionic liquids (1-ethyl-3-methylimidazolium methanesulfonate ([EMIM]MS) and 1-butyl-3-methylimidazolium methanesulfonate ([BMIM]MS) as both solvent and stabilizer, and to compare their properties to assess the effect of IL composition on NiNP formation and stability

EXPERIMENTAL METHOD

Synthesis of Nickel Nanoparticles in 1-Ethyl-3-Methylimidazolium Methanesulfonate (Ni–[EMIM]MS)

In a typical non-reflux synthesis, 0.0657 g (0.25 mmol) of nickel(II) sulfate hexahydrate ($NiSO_4·6H_2O$) was added to 1.0 g (5 mmol) of 1-ethyl-3-methylimidazolium methanesulfonate ([EMIM]MS) in a 100 mL beaker. The mixture was stirred at room temperature using a magnetic stirrer until a uniform green solution was obtained. Subsequently, 0.0095 g (0.25 mmol) of sodium borohydride ($NaBH_4$) was added gradually while stirring continuously for 30 minutes. The color of the solution changed from green to black, indicating the formation of metallic nickel nanoparticles. The reaction mixture was then centrifuged at 4,000 revolutions per minute (rpm) for 10 minutes to separate the product. The brown precipitate was oven-dried at 60 °C for 12 hours and labeled as nickel nanoparticles synthesized in 1-ethyl-3-methylimidazolium methanesulfonate (Ni–[EMIM]MS).

Synthesis of Nickel Nanoparticles in 1-Butyl-3-Methylimidazolium Methanesulfonate (Ni–[BMIM]MS)

In the reflux synthesis method, 0.0657 g (0.25 mmol) of nickel(II) sulfate hexahydrate ($NiSO_4·6H_2O$) and 1.0 g (5 mmol) of 1-butyl-3-methylimidazolium methanesulfonate ([BMIM]MS) were placed in a 50 mL round-bottom flask fitted with a reflux condenser. Sodium borohydride (0.0095 g, 0.25 mmol) was then added gradually to the solution with continuous stirring. The flask was immersed in a paraffin-oil bath maintained at 110 °C and stirred magnetically for 3 hours. A color transition from green to black and then to brown was observed during the reaction, confirming the reduction of Ni^{2+} ions to metallic nickel nanoparticles. After cooling to room temperature, the mixture was centrifuged at 4,000 rpm for 10 minutes, and the resulting solid residue was oven-dried at 60 °C. The dried sample was labeled as nickel nanoparticles synthesized in 1-butyl-3-methylimidazolium methanesulfonate (Ni–[BMIM]MS).

Synthesis of Nickel Nanoparticles in Ethylene Glycol Medium (Ni–EG) using sodium Borohydride as Reducing Agent.

For comparison, nickel nanoparticles were also synthesized in ethylene glycol (EG) without the use of ionic liquids to evaluate the stabilizing effect of the ionic liquid media. A 0.0657 g portion of nickel(II) sulfate hexahydrate ($NiSO_4·6H_2O$) was dissolved in 10 mL of distilled water to obtain a blue solution. A 1.5 mL aliquot of this solution was added to 2.0 mL of ethylene glycol with vigorous stirring. Subsequently, 1.0 mL of 0.2 M sodium borohydride ($NaBH_4$) prepared in ethanol was added dropwise to the reaction mixture, resulting in a cloudy suspension that gradually darkened. To remove excess reducing agent, 1.0 mL of 0.2 M hydrochloric acid (HCl) was added. The mixture was heated to 90 °C for 15 minutes to evaporate residual ethanol, centrifuged at 4,000 rpm for 10 minutes, and the resulting precipitate was oven-dried at 60 °C. The product was designated as nickel nanoparticles synthesized in ethylene glycol (Ni–EG).

Characterization of the Ni–ILs NPs

In this work, the physicochemical properties of the nickel nanoparticles synthesized in ionic liquid media (Ni–ILs NPs) were evaluated using various analytical techniques. The optical properties of the NiNPs were determined by ultraviolet–visible (UV–Vis) spectroscopy using a HELIOS ZETA spectrophotometer (serial no. 164613) within the wavelength range of 190–800 nm to confirm nanoparticle formation through surface plasmon resonance (SPR) absorption. The morphological features and particle size distribution were examined using scanning electron microscopy (SEM), which provided images of the surface texture and shape uniformity of the nanoparticles.

Fourier transform infrared (FTIR) spectroscopy was conducted on a Shimadzu IRPRESTIGE-21 spectrometer (serial no. A21004200709) over a spectral range of 400–4000 cm^{-1} to identify the surface functional groups and bonding characteristics of the synthesized nickel nanoparticles.

RESULTS AND DISCUSSION

FT-IR Analysis

The Ni-ILs NPs were analyzed by FTIR spectroscopy in the range of 400−4000 cm^{-1} to show the coating and stabilization of nickel NPs during the interaction with ILs. The FTIR spectra recorded are shown in Figure 1 and 2. The FTIR spectra of the synthesized nickel nanoparticles (NiNPs) in [EMIM]MS and [BMIM]MS ionic liquids revealed distinct absorption bands that confirm successful formation and stabilization of the nanoparticles. The characteristic vibrational bands for the nickel nanoparticles in [BMIM]MS were observed at 3400, 1620, 1690, 2900, 2100, and 2410 cm^{-1}, while the corresponding bands for the [EMIM]MS system appeared at 3400, 1610, 1650, 3100, 1050, and 1400 cm^{-1}. These peaks were assigned to O–H bonds in adsorbed water molecules (3400 cm^{-1}), bending vibrations of O–H in hydroxyl groups (1620–1610 cm^{-1}), aryl carboxylic group vibrations (1690–1650 cm^{-1}), C–H stretching of alkyl and aromatic groups (2900–3100 cm^{-1}), C–C stretching (2100–1050 cm^{-1}), and C–N stretching vibrations (2410–1400 cm^{-1}), respectively. The broad band around 3400 cm^{-1} is characteristic of O–H stretching vibrations and indicates the presence of hydroxyl groups or water molecules adsorbed onto the nanoparticle surface, a phenomenon commonly encountered in metal nanoparticle systems due to surface-hydroxylation or moisture adsorption [12]. The bending vibrations of O–H groups in hydroxyl entities, appearing near 1620–1610 cm^{-1}, further corroborate the presence of surface-bound hydroxyls or weakly coordinated water molecules. Similar frequencies have been attributed to δ(H–O–H) vibrations in aqueous or hydroxylated nanoparticle surfaces [13]. Distinct bands in the C–H stretching region (2900–3100 cm^{-1}) correspond to alkyl and aromatic C–H bonds of the imidazolium cation's side-chains. The presence of these bands validates that the imidazolium moiety remains intact and bound to the particle surface, acting as a capping ligand. In analogous systems, imidazolium ionic liquids have been shown to remain structurally preserved upon nanoparticle formation, as evidenced by characteristic C–H stretching vibrations [10].

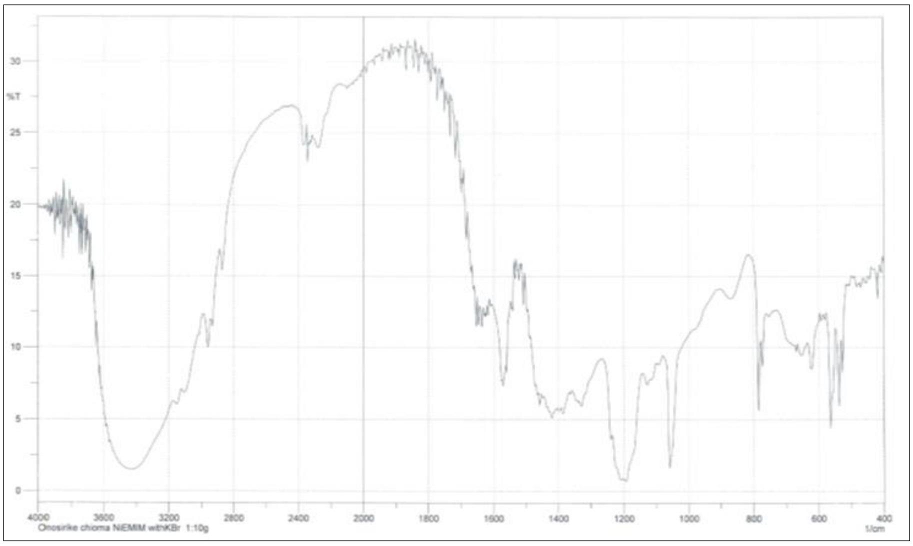

Figure 1: FT-IR Spectra of Ni (EMIM) MS

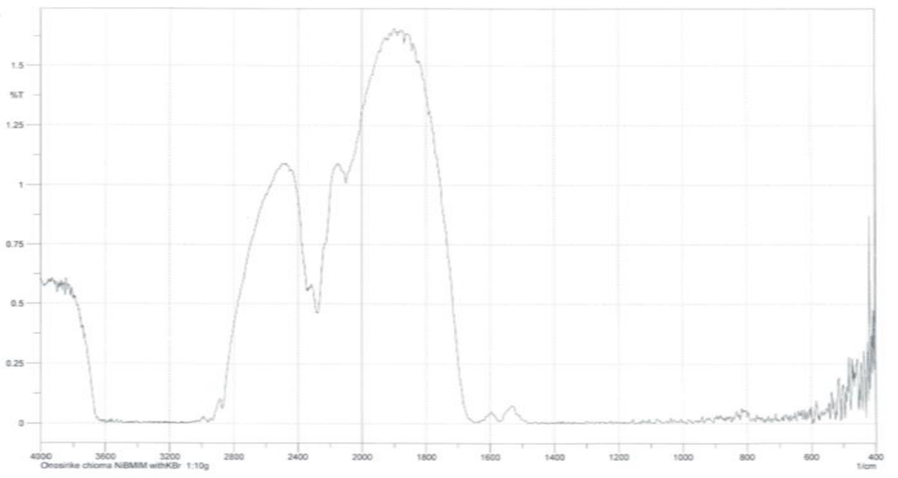

Figure 2: FTIR Spectra of Ni (BMIM)MS

UV–Visible Analysis and Discussion

The UV–Visible spectra of the synthesized nickel nanoparticles (NiNPs) in Figures 3 and 4 exhibit broad absorption bands within the 300–350 nm range, which are characteristic of the surface plasmon resonance (SPR) of metallic nickel nanoparticles. The appearance of this band, absent in the precursor nickel salt solutions, confirms the successful reduction of Ni^{2+} to Ni^0 during synthesis. Similar SPR features have been reported for chemically reduced nickel nanoparticles and are typically attributed to collective oscillations of conduction electrons in response to incident light [14]. A notable difference is observed between the two ionic liquid systems. The NiNPs synthesized in [BMIM]MS (Figure 3) show a slightly red-shifted and broader absorption band relative to those synthesized in [EMIM]MS (Figure 4). A red-shift in SPR typically indicates an increase in particle size or a higher degree of aggregation, as larger nanoparticles exhibit lower-energy plasmon transitions [15]. This observation supports the SEM findings, which revealed larger particle dimensions (60–80 nm) in the [BMIM]MS system.

In contrast, the NiNPs synthesized in [EMIM]MS display a blue-shifted and more defined absorption band, consistent with the formation of smaller and more uniformly dispersed nanoparticles. This trend is linked to differences in nucleation and growth kinetics mediated by the ionic liquids. The shorter ethyl chain in $[EMIM]^+$ provides less steric hindrance, allowing faster nucleation and leading to smaller particles. Conversely, the longer butyl chain in $[BMIM]^+$ increases viscosity and hydrophobicity, slowing nucleation and promoting particle growth. Similar ionic-liquid–dependent size effects have been reported in previous nanoparticle synthesis studies [16].

The broad nature of the SPR peaks in both spectra indicates a degree of polydispersity typical of chemically reduced transition-metal nanoparticles. This aligns with previous reports showing that reduction methods employing $NaBH_4$ often result in broad and diffuse SPR bands due to variations in particle size distribution [17].

The UV–Visible analysis demonstrates that the ionic liquid plays a crucial role not only in reducing Ni^{2+} ions but also in controlling particle size, morphology, and optical behavior. The spectral differences between Ni (EMIM)MS and Ni (BMIM)MS directly reflect the influence of ionic liquid cation structure on nucleation and growth processes.

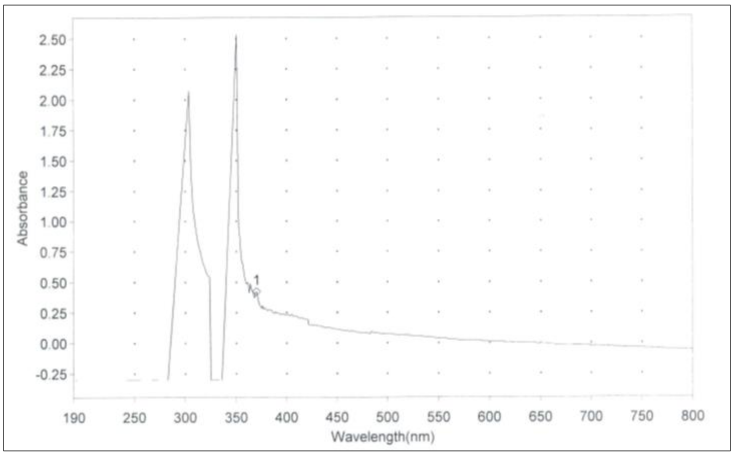

Figure 3: UV-VIS spectra of NI(BMIM)MS

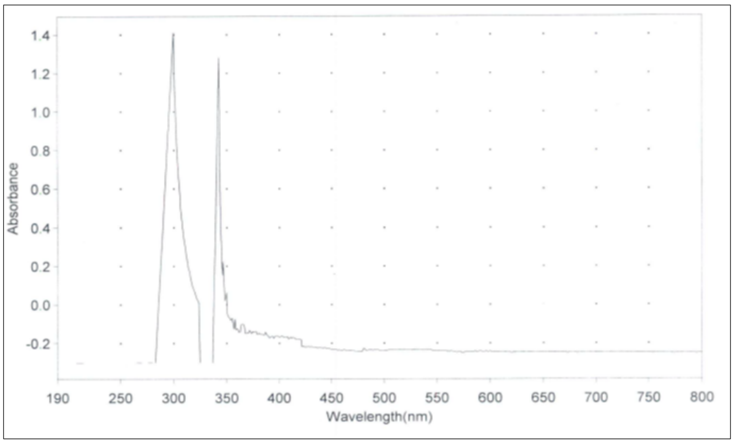

Figure 4: UV-VIS spectra of Ni (EMIM)MS

Scanning Electron Microscopy (SEM) Analysis

The SEM micrographs presented in Figures 5 and 6 reveal clear differences in particle morphology and dispersion arising from the choice of synthesis medium. Nickel nanoparticles synthesized in [BMIM]MS (Figure 5A) appear relatively larger, within the range of 60–80 nm, and exhibit moderate aggregation, a behavior attributed to the longer butyl chain of the $[BMIM]^+$ cation, which increases viscosity and reduces ionic mobility, thereby slowing nucleation and promoting particle coalescence during growth. Similar effects of extended alkyl chains in imidazolium ionic liquids on nanoparticle size and aggregation have been reported in earlier studies [16,18]. In contrast, NiNPs prepared in [EMIM]MS (Figure 5B) are smaller, approximately 45–60 nm, and more uniformly dispersed, reflecting the influence of the shorter ethyl substituent in $[EMIM]^+$, which enhances ionic mobility, accelerates nucleation, and facilitates effective electrostatic and steric stabilization of the nanoparticle surfaces. These observations are in agreement with previous work demonstrating that shorter-chain imidazolium ionic liquids provide superior capping efficiency and lead to well-defined, monodisperse metal nanoparticles [19]. In contrast, particles synthesized in ethylene glycol (Figure 6) are irregular, polydisperse, and significantly aggregated, highlighting the limited stabilizing ability of ethylene glycol compared to ionic liquids. Without the structured ionic environment and strong cation–surface interactions characteristic of imidazolium systems, particle collisions and fusion occur more readily, generating larger and less uniform aggregates, consistent with literature describing reduced size control in conventional polyol systems. Collectively, the SEM results confirm that the ionic liquid medium plays a crucial role in defining nanoparticle size and morphology, with Ni(EMIM)MS producing the smallest, most uniformly dispersed particles, followed by Ni(BMIM)MS, and finally the poorly stabilized particles obtained in ethylene glycol, reinforcing the widely reported influence of ionic liquid structure on nanoparticle nucleation and growth behavior.

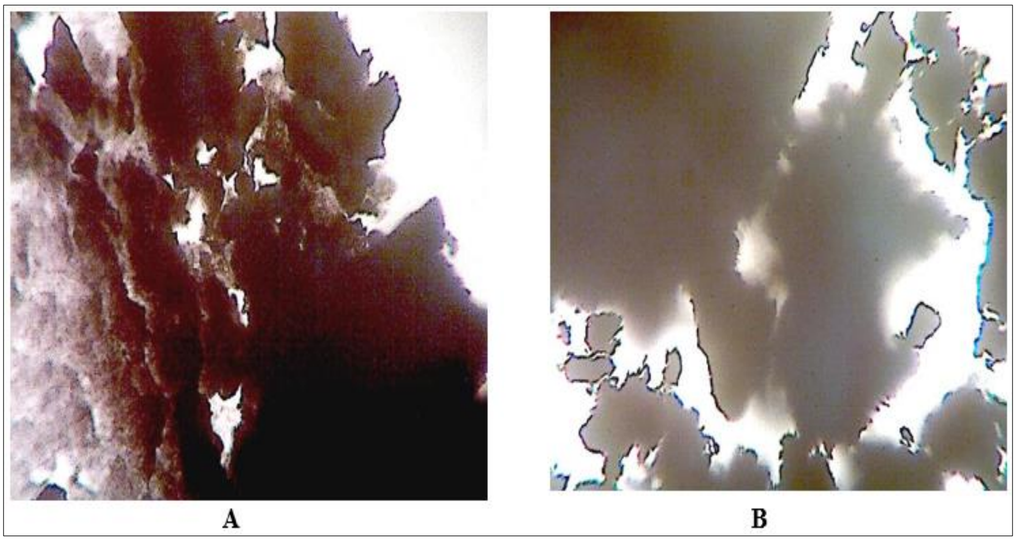

Figure 5: SEM micrographs of; A=Ni (BMIM)MS, B=Ni (EMIM)MS

Figure 6: SEM image of NiNPs in Ethylene glycol

CONCLUSION

This study successfully developed a size-controlled and stabilized method for synthesizing NiNPs. Spectroscopic analysis revealed that the NiNPs sizes were influenced by the structure if the ILs and were stabilized through a strong interaction between the ionic liquid functional groups and the nanoparticle surface, which led to the stabilization of the metallic nickel core. [EMIM]MS produced smaller, more uniformly dispersed, and well-defined nanoparticles compared to [BMIM]MS, which yielded slightly larger particles with moderate aggregation. Comparative study using ethylene glycol as the solvent media for the synthesis yielded irregular, highly aggregated nanoparticles, emphasizing the superior stabilization capacity of ILs. These findings demonstrate that the alkyl chain length of imidazolium ILs plays a crucial role in nucleation and particle growth, establishing these ILs as efficient, green and tunable reaction media for nickel nanoparticles synthesis.

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