



Preparation of vanadium pentoxide nanoparticles by ionic liquid-assisted sonochemical method: Effect of ionic liquid stericity on particle characteristics



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ABSTRACT

The V₂O₅ particles were synthesized in presence of 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide [emim][Tf₂N], and 1-ethyl-3-methylimidazolium trifluoromethane sulfonate [emim][TfO] ionic liquids (IL). Comparative experiments were conducted with heating and without use of IL. As-synthesized, as well as 400 °C/4 h heated V₂O₅ were evaluated by Fourier transform infrared spectroscopy, thermal gravimetric analysis, high resolution scanning electron microscopy and wide angle X-ray diffraction characterization. Particles morphology, as well as crystallinity, was influenced due to use of IL under ultrasonic irradiation. Formation of nano/micro rods increased with the use of bulky anion based IL—[emim][Tf₂N]. The V₂O₅ particles synthesized by IL assisted sonochemical method indicated enhanced performance for degradation of methylene blue as well as for phenol.

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1. Introduction

Recently sonochemical reactions are of interest to scientists due to low cost and easy set up for synthesis. Sonication process is characterized by formation of bubbles, its growth and implosive collapse. Formation of bubbles occurs due to diffusion of vapor into bubble and bubbles collapse when bubble sizes reaches to the maximum. This phenomenon is characterized by very high temperature (>5000 K), high pressure (>20 MPa) and high cooling rates (10¹⁰ K/s). Type of solvent plays an important role in the formation and collapse of the bubble in sonochemical reactions [1].

Sonation has been found to be an effective technique for synthesis various types of nanoparticles with unique morphology and properties. The sonochemical reactions have been used for Fe₃O₄/SiO₂ core-shell nanoparticles synthesis [2], titanium nanoparticles formation [3], dispersion of Fe₃O₄ nanoparticles on graphene oxide sheet [4], synthesis of Mn₃O₄ nanoparticles for super-capacitor and super-magnetic applications [5,6], copper supramolecular nanoparticles [7], synthesis of shear thickening of silicon dioxide (SiO₂)/polyethylene glycol (PEG) [8], manganese dioxide nanoparticles synthesis by permanganate reduction [9], etc.

In sonochemical reactions, low vapor pressure solvent can be used to maximize temperature and pressure during cavitation because such solvent does not participate in the reaction. Ionic liquid has relatively no vapor pressure and hence it can be explored for sonication approach. An IL has good chemical and thermal stability, high ionic conductivity, high heat capacity, wide electrochemical potential window, properties of acid or base or ligand, surfactant nature, and different solubility with various gases. Relatively less work is reported on IL assisted sonochemical method (ILASM) for organometallic and solid state chemistry [10]. Nickel sulfide submicron particles were formed by 1-butyl-3-methylimidazolium tetrafluoroborate [bmim][BF₄] assisted sonication process, which exhibited uniform shape and distributed particles compared to sintered particles without IL [11]. The ILASM was used for depositing nanoparticles of Pt, Pd, Au, Ag, SnO_x on to the surface of carbon nanotubes (CNT) to control concentration as well as structure of nanoparticles on the surface [12]. The [emim][BF₄] mediated sonication was used for decomposition of iron carbonyl precursor to form Fe₂O₃ nanoparticles without stabilizing/capping agent [13]. The Sb₂S₃ nanorods were synthesized by [bmim][BF₄] using Sb₂Cl₃ as starting material resulting in the formation of nanorods compared to round structure formation in the absence of IL [14].

Vanadium pentoxide (VP) is interesting material to explore further due to its applications for microelectronics, solid state ionics, sensing, energy storage devices, catalysis, biosensors,

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Nomenclature

Sample	Details
VPH	Vanadium pentoxide particles synthesized without ionic liquid by heating method
VPU	Vanadium pentoxide particles synthesized without ionic liquid by sonication
VPUT	Vanadium pentoxide particles synthesized with [emim][TfO] by sonication
VPUBT	Vanadium pentoxide particles synthesized with [emim][Tf ₂ N] by sonication
VPHC	Vanadium pentoxide particles synthesized without ionic liquid by heating method and calcined at 400 °C for 4 h
VPUC	Vanadium pentoxide particles synthesized without ionic liquid by sonication and calcined at 400 °C for 4 h
VPUTC	Vanadium pentoxide particles synthesized with [emim][TfO] by sonication and calcined at 400 °C for 4 h
VPUBTC	Vanadium pentoxide particles synthesized with [emim][Tf ₂ N] by sonication and calcined at 400 °C for 4 h

adsorption, etc. Vanadium pentoxide (V₂O₅) nanoparticles were synthesized by vanadium oxytriisopropoxide as starting material using 1-butyl-1-methyl pyrrolidinium bis(trifluoromethylsulfonyl) imide ([Py_{1,4}][Tf₂N]) and 1-ethyl-3-methyl imidazolium bis(trifluoromethylsulfonyl) imide ([emim][Tf₂N]) by conventional heating method [15]. Sonochemical process is used for synthesis of silver vanadium oxide micro/nanorods [1], vanadium doped TiO₂ nanoparticles [16] and lanthanide orthovanadate spindle like nanoparticles [17].

Vanadium pentoxide synthesis by sol-gel process using two different ILs has been reported in literature keeping same anion and changing cation—[Py_{1,4}] and [emim] [15]. Vanadium belongs to transition metal family with oxidation state varying from +2 to +5, hence it will be interesting to synthesize vanadium pentoxide with ILs differing in their anions which has tendency to have much more influence on sol-gel by manipulating metal-oxygen-metal network by its steric and electrostatic forces. Further to the best of our knowledge, synthesis of vanadium pentoxide micro/nanoparticles has not been reported using ILs under ultrasound irradiation. In this work, we have studied synthesis of VP under sonication in presence of two ILs, viz., 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide [emim][Tf₂N] and 1-ethyl-3-methylimidazolium trifluoromethane sulfonate [emim][TfO] (Fig. 1). These two ILs belong to ILs family that can apparently be distilled at reduced pressure with moderate temperature making them good candidates for recycling if used for large scale production of nanoparticles. Effect of IL was assessed by comparing structural and morphological properties characterized by wide-angle X-ray diffraction (WAXD), high resolution scanning electron

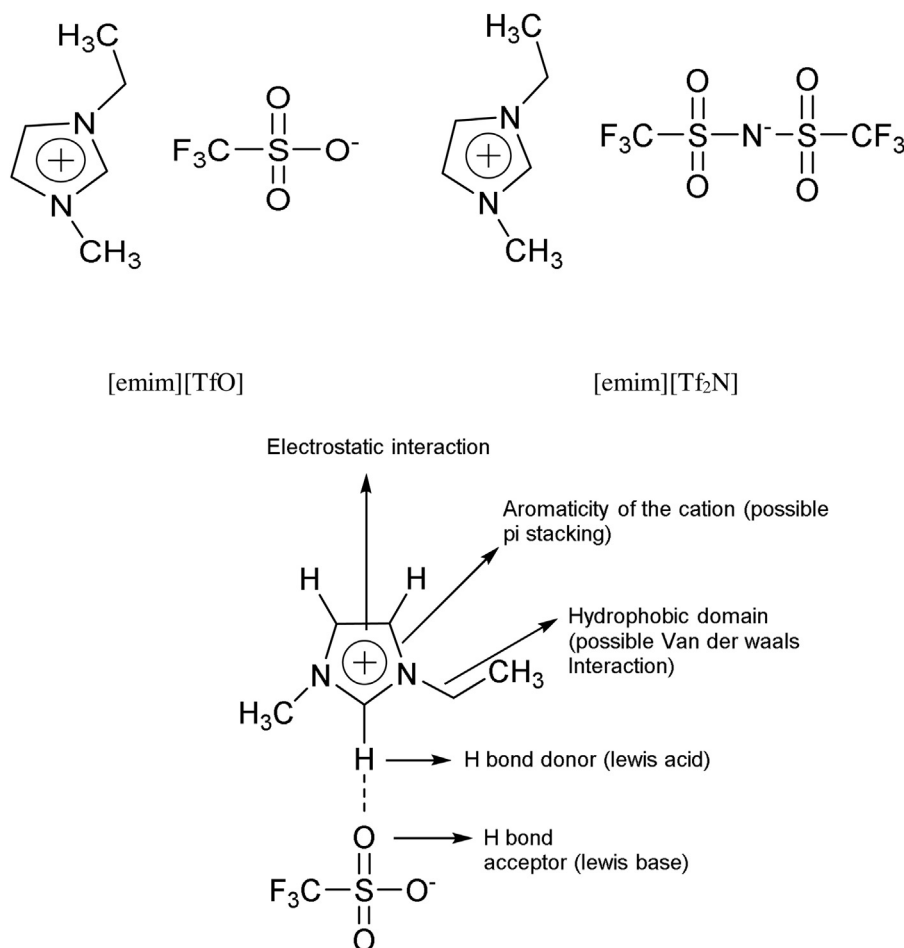


Fig. 1. Structure of ionic liquids used for nanoparticles synthesis and possible interaction between cation and anion.

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