



Non-aggregated divanadium pentoxide nanoparticles: A one-step facile synthesis. Morphological, structural, compositional, optical properties and photocatalytic activities



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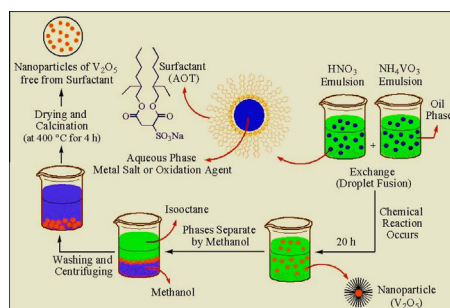
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HIGHLIGHTS

- Synthesis of perfectly spherical V_2O_5 nanoparticles stabilized by AOT.
- Uniform shape and non-agglomerated of V_2O_5 NPs made via reverse microemulsion route.
- XRD confirmed the orthorhombic phase pure structure of V_2O_5 nanoparticles.
- Highly crystalline & well-dispersed V_2O_5 nanoparticles were successfully obtained.
- The optical properties of these NPs were studied using UV–Vis absorption and PL spectra.

GRAPHICAL ABSTRACT

Flowchart for chemical processing of the V_2O_5 NPs synthesized in the reverse microemulsion system.



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ABSTRACT

In the present paper, pure phase and well-dispersed divanadium pentoxide nanoparticles (V_2O_5 NPs) with an orthorhombic structure were synthesized via the reverse microemulsion process and using low-cost NH_4VO_3 as the starting material in the absence of any co-surfactant. The synthetic method is facile, fast, low reaction temperature, environmental friendly, and easy controlled preparation condition. The structure, morphology and optical properties of these particles were characterized by field emission scanning electron microscope (FE-SEM), transmission electron microscopy (TEM), energy dispersive analysis of X-ray (EDAX), X-ray diffraction (XRD), thermo-gravimetric analysis (TGA), differential temperature analysis (DTA), fourier transform infrared spectroscopy (FT-IR), ultraviolet–visible absorption spectroscopy (UV–Vis), and photoluminescence (PL) spectroscopy. The synthesized NPs were found to be fully crystalline and spherical in shape with uniform distribution. The mean crystallite size of the V_2O_5 NPs was estimated by X-ray powder diffraction pattern, which was in close agreement with the particles size obtained by TEM analysis. Optical properties of the nano-structured V_2O_5 are also tested and discussed. The band gap value of the V_2O_5 NPs is about 2.93 eV calculated by UV–Vis spectroscopy. Considerable emissions due to oxygen vacancies and band transition of PL peaks were observed. In addition, the photocatalytic activities of these particles were evaluated by photodegradation of toluidine blue O (TBO) dye under visible light irradiation.

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1. Introduction

Transition metal oxides with diverse morphological structure have a wide range of potential applications. Vanadium forms a series of oxides, some of which have the formulas VO, V₂O₃, VO₂, and V₂O₅. The most important of the oxides is the V₂O₅, and is the most stable member of this series [1]. In addition, among the transition metal oxide semiconductors, divanadium pentoxide, with layered structure, has attracted considerable interest over the years owing to their unique physical and chemical properties. The multiple oxidation states of the vanadium oxides family entails facile reduction and oxidation chemistry which gives rise to potent catalysts [2]. Also, the observed photo- and electrochromic behavior of the V₂O₅ is exploited in information displays and color memory devices [3]. Furthermore, the V₂O₅ has been extensively studied for use as functional ceramics [4]. In general, these compounds with the excellent properties such as high energy density, capability of fast charge–discharge ability to exist in different oxidation states, wide optical band gap, good chemical and thermal stability, excellent reversibility, and superior thermoelectric property, plays a vital role in the catalysis, photocatalysis, thermochromic windows, ceramics, optoelectronic switching devices, solar cells, electrochromic devices, gas sensors, as cathode material in rechargeable lithium batteries, and a large variety of electrical and optical devices [2–12]. Consequently, this trait makes the V₂O₅ is well suited for the construction of functional materials or novel devices, which can be operated under ambient conditions.

In recent years, use of photocatalysis technology has attracted considerable attention around the world. This photocatalytic process has been represented as a potential means for complete mineralization of organic wastes (e.g., reduction of water and air pollution levels) while cost-effectiveness and simplicity of operation have been taken into account. Furthermore, it makes no use of toxic materials and can also be operated at ambient conditions. The fundamental aspects and potential advantages of photocatalytic remediation processes have been described in details [13–16]. Many semiconductor photocatalysts have been explored in recent years. Among the many semiconductor oxide photocatalysts available, V₂O₅ has been studied extensively owing to its chemical inertness, strong oxidizing power and long-term stability against photo and chemical decay [17,18]. However, practical utility of photogenerated electron–hole pairs has been limited by their rapid recombination. The photonic efficiency of a photocatalyst may increase as the rate of charge carrier recombination relative to that of interfacial charge transfer decreases. [19,20]. The photocatalytic activity of the photocatalysts depends on its intrinsic properties, such as unique morphology, crystal phase, band gap energy, and surface to volume ratio. Previous studies have reported that the specific surface area increases with decreasing average particle size of a photocatalyst in the nanometer regime. This in turn increases the number of active sites for photogenerated charge carriers to undergo interfacial charge transfer [21,22]. According to literature, the useful properties of vanadium oxides can be even more interesting in the nanometric scale [23]. In general, the properties of transition metal oxides depend not only on their chemical composition but also on their structure, phase, shape, size and size distribution. Nanostructured materials present unusual mechanical, electrical, optical and catalytic properties, deriving from both their large surface area and possible confinement of carriers [24]. Divanadium pentoxide nanostructures can be obtained by various physical and chemical techniques such as sol–gel method, soft template, hydrothermal, pulsed laser ablation, thermal evaporation method, electrochemical deposition method [2,25–31], etc. In spite of the fact that several methods have already been proposed for the synthesis of these materials, it is required to develop

a good alternative route to cost-effectively and commercially produce pure and homogeneous V₂O₅ nanostructures (with small particle size) at a relatively low temperature. Recently, intensive research efforts have been focused on the synthesis of well-defined uniformly sized nanocrystals in order to identify their size dependent properties. Shevchuk et al. [32] and Wang et al. [33] have reported that a couple of factors such as size and monodispersity of the V₂O₅ nanomaterials are the important features for sophisticated technological and industrial applications.

Among chemical methods, the reverse microemulsion route is the robust method to shrivel the particle size, shape, high crystallinity, control over the dimensionality of the product, non-requirement of post-thermal treatment, and high monodispersity. The size and shape of the NPs can be controlled by the components and structure of the microemulsion system. Generally, by adjusting the $\frac{(water)}{(surfactant)}$ molar ratio, which is equal to *W*, the size of the particles can be controlled. Consequently, preparation of nanostructures via the reverse microemulsion gives a proper control of size and composition [34–37]. According to literature, the present method is simple, time-saving, and does not require the complicated operation and expensive high temperature calcination process. Also, this microemulsion route can be employed to produce very fine particles of catalysts, which are efficient for photocatalytic processes [38,39]. Therefore, this technique is one of the most preferred candidates.

In this work, we describe a low-cost, simple and economical approach with uniform shape and excellent monodispersity for the production of single-crystal V₂O₅ NPs synthesized and stabilized by the reverse microemulsion and AOT, respectively. The present study has investigated the morphological, size, composition, and structure of these particles. Also, the optical properties of these NPs are characterized by UV–Vis absorption and photoluminescence spectra. Then, the synthesized NPs have been proven to be a potential photocatalyst for the photodegradation of toluidine blue O dye under visible light irradiation.

2. Experimental

2.1. Materials

Sodium bis(2-ethylhexyl) sulfosuccinate as a surfactant (C₂₀H₃₇NaO₇S, AOT (or aerosol-OT)) (purity 98%), ammonium metavanadate (NH₄VO₃) as precursor salt and toluidine blue O (85% dye content) were purchased from Sigma–Aldrich. Commercial titanium dioxide powder (P25, ca. 80% anatase, 20% rutile) from Degussa was used for the comparison of the photocatalytic activity. Isooctane (purity ~99.9%) as the oil phase was supplied from Merck. Deionized and double distilled water was used for microemulsion and solution preparation. All the chemicals and solvents were used as received without further purifications.

2.2. Characterization

The shape (or surface morphology), size, crystal structure and elemental composition of the V₂O₅ NPs were characterized by field emission scanning electron microscope (FE-SEM), transmission electron microscopy (TEM), energy dispersive analysis of X-ray (EDAX), X-ray diffraction (XRD), thermo-gravimetric analysis (TGA), differential temperature analysis (DTA), fourier transform infrared spectroscopy (FT-IR), ultraviolet–visible absorption spectroscopy (UV–Vis), and photoluminescence (PL) spectroscopy. FE-SEM image was obtained on a Hitachi S-1460 field emission scanning electron microscope using accelerating voltage of 15 kV. TEM measurement for the V₂O₅ NPs was performed on a Philips

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