



Novel Schiff base ligand-assisted in-situ synthesis of $\text{Cu}_3\text{V}_2\text{O}_8$ nanoparticles via a simple precipitation approach

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ABSTRACT

The effect of different Schiff-base ligands on size, morphology and uniformity of $\text{Cu}_3\text{V}_2\text{O}_8$ nanoparticles prepared via a simple in-situ precipitation route was investigated. Different calcination temperatures were applied and the as-prepared products were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared (FT-IR) spectrum, Electron Dispersive X-ray spectroscopy (EDX) and ultraviolet–visible (UV–Vis) spectroscopy. Vibrating sample magnetometer (VSM) was used to study the magnetism properties of $\text{Cu}_3\text{V}_2\text{O}_8$ sample. According to the obtained results, using the appropriate amount of Schiff-base ligand is a crucial parameter for the control in particle size. The photocatalytic activity of $\text{Cu}_3\text{V}_2\text{O}_8$ nanostructures was evaluated by the degradation of methylene blue in aqueous solution under UV and visible light irradiation.

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1. Introduction

The nanoparticles have received considerable attention due to their wide range of applications in various fields like catalysis, solar cells, batteries, photocatalysis and sensors. Transition metal vanadates, as a considerable class of materials, have been intensively pursued in recent years because of their applications in optical devices [1], catalysis [2], paramagnetic materials [3,4], lithium batteries [5,6], etc. Among transition metal vanadates, $\text{Cu}_3\text{V}_2\text{O}_8$ with crystal structure of porous consisting Cu—O octahedra and V—O tetrahedra has been studied as a material with good photocatalytic performances and electrochemical properties [7]. It is good to know that particle size and morphology of nanostructures depend on their synthesis method. A variation of possible routes including hydrothermal method [8,9], simple template-free solution method [10] and co-precipitation method [11] have been applied to obtain different types of copper vanadates. Herein, we develop the precipitation method to provide $\text{Cu}_3\text{V}_2\text{O}_8$ nanocrystals. The precipitation method is a suitable synthesis process for the preparation of many inorganic powders. This method is simple, convenient and cost effective synthetic procedure and provides an effective way to the synthesis of uniform nanocrystals. In this method, crystallization is performed at low temperature and design of reaction condition is very flexible. To optimize the $\text{Cu}_3\text{V}_2\text{O}_8$ size, morphology and ultimate properties, ligand shell was chosen. The ligand shell is a molecular

monolayer encapsulating the core. In the case of organic species, ligands have a functional head group and one or more hydrocarbon tails that both elements play a role in the control of nucleation and growth [12]. Beside the well-known surfactants, recently our team has focused on the effect of ligands containing hard atoms (N, O) as well as bulky aromatic groups on the properties of obtained products [13–17]. In this work, the effect of different Schiff base ligands as capping agents on morphology, size and uniformity of $\text{Cu}_3\text{V}_2\text{O}_8$ nanoparticles was studied. Schiff base ligands containing four hard binding sites (N and O), are capable of stabilizing transition metal centers very well. These kinds of molecules are of concern in preventing crystal growth of particular surface planes, or limiting the size of crystals in the synthesis of nanoparticles [18]. Herein, the $\text{Cu}_3\text{V}_2\text{O}_8$ nanostructures were synthesized with different Schiff base ligands via in-situ precipitation method using $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and NH_4VO_3 as copper and vanadium sources, respectively. The effects of various parameters such as different Schiff base ligands and calcination temperatures on the size, morphology and uniformity of products were also investigated. Moreover, degradation of methylene blue (MB) was carried out to study the photocatalytic activity of $\text{Cu}_3\text{V}_2\text{O}_8$ nanostructures under UV and visible light irradiation.

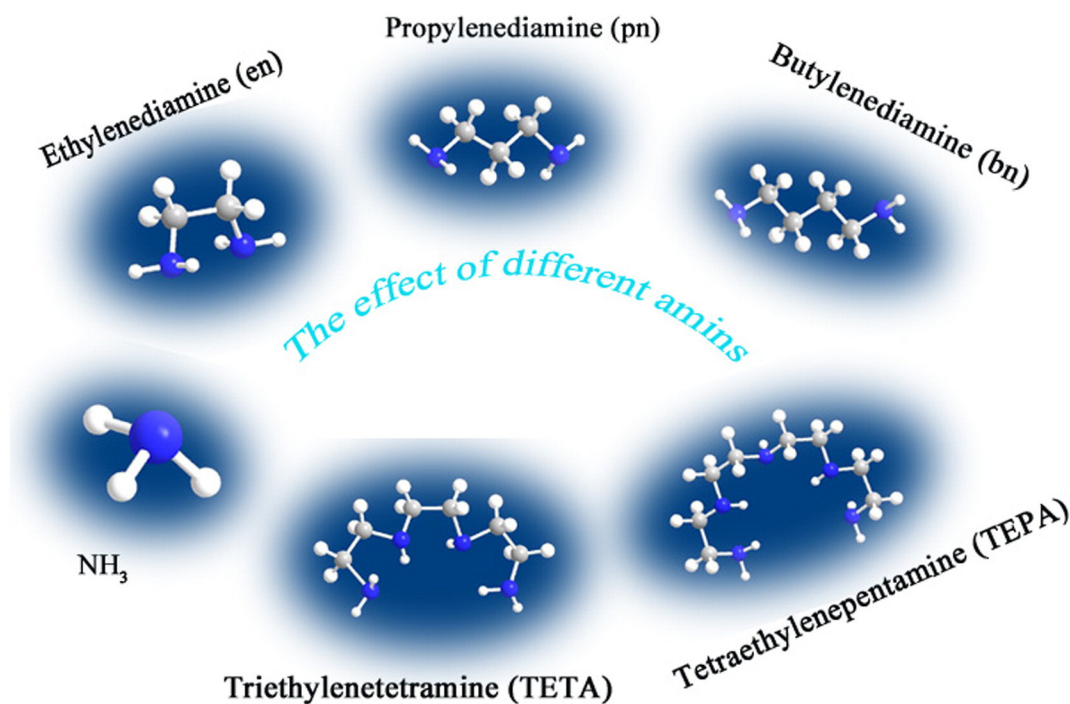
2. Experimental

2.1. Materials and physical measurements

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, NH_4VO_3 , acetyl acetone (acac), ammonia, ethylenediamine (en), propylenediamine (pn), butylenediamine (bn),

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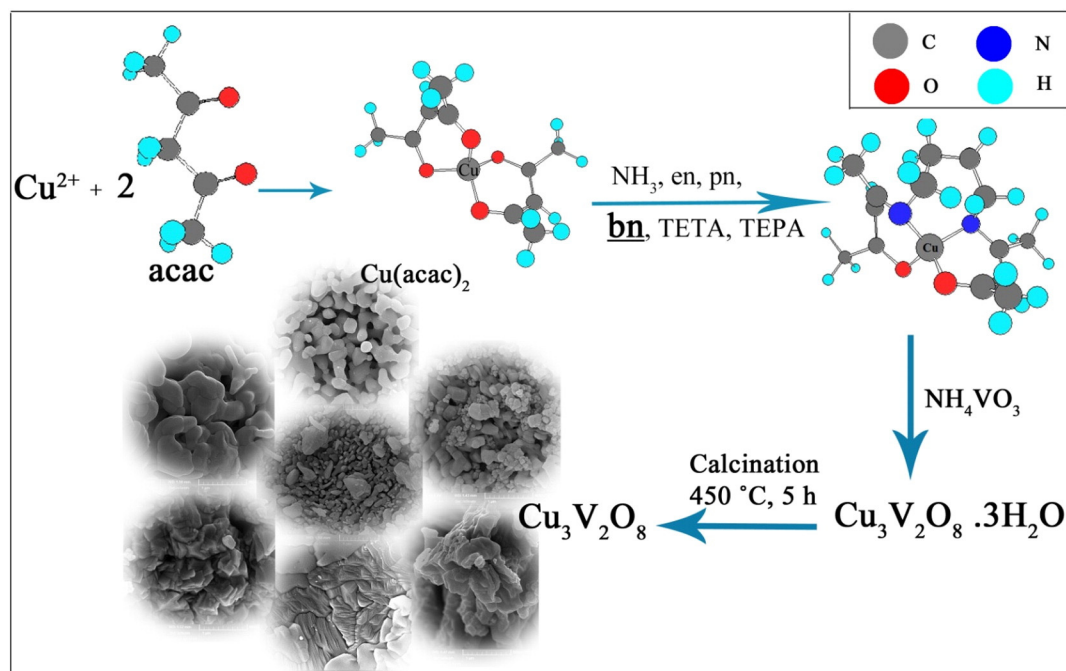
Scheme 1. Different amines used for synthesizing of Schiff base ligand.

Table 1
Molar ratios of starting materials.

Amine	Cu:V:acac:Amine
NH ₃	3:2:6:6
en	3:2:6:3
pn	3:2:6:3
bn	3:2:6:3
TETA	3:2:6:3
TEPA	3:2:6:3

purchased from Merck Company. All of the chemicals were used as received without further purifications. For characterization of the products, X-ray diffraction (XRD) patterns were recorded by a Rigaku D-max C III, X-ray diffractometer using Ni-filtered Cu K α radiation. Scanning electron microscopy (SEM) images were obtained on a Philips XL-30ESEM. Transmission electron microscopy (TEM) image was obtained on a Philips EM208 transmission electron microscope with an accelerating voltage of 200 kV. Fourier transform infrared (FT-IR) spectra were recorded on a Shimadzu Varian 4300 spectrophotometer in KBr pellets. Optical analyses were performed using a V-670 UV–Vis–NIR Spectrophotometer (Jasco). The magnetic properties of the samples

triethylenetetramine (TETA) and tetraethylenepentamine (TEPA) were



Scheme 2. Schematic depiction for the preparation of Cu₃V₂O₈ nanoparticles.

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