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Synthesis and characterization of titanium dioxide nanoparticles using *Euphorbia heteradena Jaub* root extract and evaluation of their stability

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Abstract

The present study reports an environmental friendly and cost effective method for green synthesis of rutile TiO_2 nanoparticles (NPs) from titanyl hydroxide using *Euphorbia heteradena Jaub* root extract as a reducing and capping agent. The hydroxyl functional group of phenolics in *E. heteradena Jaub* root extract act as reducing and capping/stabilizing agents. Also, the kinetics of the reaction was studied using UV–visible spectrophotometry which revealed surface plasmon resonance (SPR) around 360 nm. The crystalline nature of TiO_2 NPs was identified using XRD analysis which confirmed the formation of rutile phase. The possible mechanism leading to the formation of TiO_2 NPs is suggested. The synthesized TiO_2 NPs by this method is quite stable under inert atmosphere for two months.

Keywords: Nanoparticles; FT-IR; X-ray techniques; UV–visible spectroscopy; *Euphorbia Heteradena Jaub*

1. Introduction

The field of nanotechnology is one of the most interesting areas used to describe the creation and utilization of materials with structural features between those atoms and bulk materials with at least one dimension in nanoscale [1]. Nanoparticles exhibit completely new or improved properties based on specific characteristics such as size, distribution and morphology. Nanomaterials may provide solutions to technological and environmental challenges in the areas of solar energy conversion, catalysis, medicine, and water treatment [1].

Among several metal oxide nanoparticles, TiO_2 NPs have become the focus of intensive research owing to their wide range of application in the development of new techniques in the areas of medicine, materials sciences due to their long-term thermodynamic stability, strong oxidizing power, and relative non-toxicity, photo catalytic activity for the decomposition of organic contaminants in water and aqueous wastes, photo induced super-hydrophilicity and anti-fogging effect [2–4].

Traditionally most of metal and metal oxide NPs were routinely synthesized and stabilized through chemical and physical methods such as solvothermal, reduction in solutions, chemical and photochemical reactions in reverse micelles, radiation assisted, sonochemical, microwave assisted process, sol–gel technique and electrochemical technique. However, most of these methods are extremely expensive, toxic, high pressure and energy requirement, difficult separation and potentially hazardous [5–7]. Also, chemical synthesis methods lead to presence of some toxic chemical absorbed on the surface that may have adverse effect in the medical applications.

Biological methods of nanoparticles synthesis using environmentally benign materials like gum or plant extract [8–16] have been suggested as possible ecofriendly alternatives to chemical and physical methods. These methods provides advancement over chemical and physical method as it is cost effective, environment friendly, easily scaled up for large scale synthesis of nanoparticles. Furthermore, in this method there is

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no need to use high pressure, energy, temperature and toxic chemicals.

The family *Euphorbiaceae* also known as spurge family is one of the most diversified families of flowering plants. The used parts of the *Euphorbia* species include roots, seeds, latex, stem wood, and stem barks, leaves and whole plants [17]. *Euphorbia heteradena Jaub* from the family of *Euphorbiaceae* has a wide spread in different regions of the world. Plants in the family *Euphorbiaceae* are well known for the chemical diversity of their phytoconstituents especially antioxidant phenolics, terpenoids, saponins, aromatic esters and steroids [18,19]. Furthermore, a survey of literatures reported on the *E. heteradena Jaub* revealed the presence of potent antioxidant flavonoid aglycones and C or O-glycosides such as kaempferol glycosides, rutin glucoside and quercetin glycoside in different parts of the plants [20–22].

In continuation of our recent works on the synthesis of NPs [10–16], we wish to report a new and simple protocol for the preparation of rutile TiO_2 NPs by using *E. heteradena Jaub* root extract as a reducing and stabilizing agent.

2. Experimental

2.1. Instruments and reagents

High-purity chemical reagents were purchased from the Merck and Aldrich chemical companies. All materials were of commercial reagent grade. FT-IR spectra were recorded on a Nicolet 370 FT/IR spectrometer (Thermo Nicolet, USA) using pressed KBr pellets. X-ray diffraction (XRD) measurements were carried out using a Philips powder diffractometer type PW 1373 goniometer (Cu $K\alpha$=1.5406 Å). The scanning rate was 2°/min in the 2θ range from 10 to 50°. Ultraviolet–visible (UV–vis) absorption spectra were recorded by a Shimadzu UV 2100 PC UV–visible spectrophotometer. The shape and size of TiO_2 NPs was identified by transmission electron microscope (TEM) using a Philips EM208 microscope operating at an accelerating voltage of 90 kV.

2.2. Preparation of Euphorbia heteradena Jaub root extract

50 g of dried root of *E. heteradena Jaub* was powdered and refluxed at 70 °C with 250 mL of sterile distilled water for 2 h and the mixture was allowed to cool to room temperature. Then, the aqueous extract of the root was centrifuged at 6500 rpm and supernatant separated by filtration.

2.3. Preparation of Euphorbia heteradena Jaub root extract

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2.4. Green synthesis of rutile TiO_2 NPs using Euphorbia heteradena Jaub root extract

0.5 G of $TiO(OH)_2$ was added to the 15 mL aqueous extract of the root of *E. heteradena Jaub* with constant stirring at 60 °C for 2 h. After 3 min the color of the solution changed to light gray due to the excitation of surface plasmon resonance which indicates the formation of nanoparticles. Bioreduction process was monitored by UV–vis and FT-IR spectra of the solution. Then the mixture of NPs was centrifuged at 7000 rpm for 30 min to completely precipitation of TiO_2 NPs. The obtained precipitate was then washed three times with chloroform and ethanol, respectively, then air dried for 24 h at room temperature.

3. Results and discussion

The UV spectrum of root extract of the plant (Fig. 1) shows bands at λ_{max} 390 nm (band I) due to the transition localized within the ring of cinnamoyl system; whereas the one around 240 nm (band II) is for absorbance of ring related to the benzoyl system. Although, they are generally related to the $\pi \rightarrow \pi^*$ transitions of double bonds but this absorbance bands of cinnamoyl and benzoyl systems as shown by spectrum are specification of flavones nuclei inside the extract as strong antioxidants [23,24].

Fig. 2 shows the absorption spectrum of TiO_2 NPs due to SPR of NPs (Scheme 1). Changing the color of the reaction

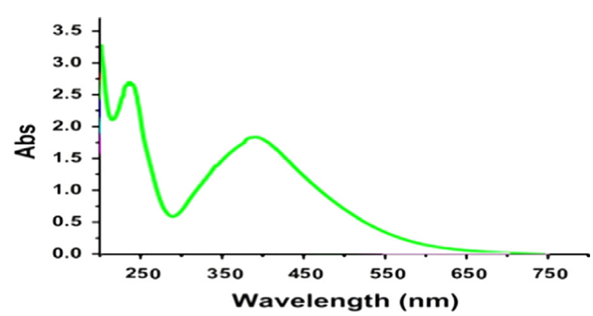

Fig. 1. UV–vis spectrum of the aqueous extract of the root of the *Euphorbia heteradena Jaub*.

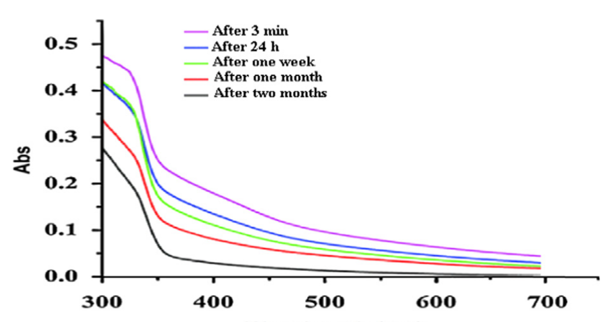

Fig. 2. UV–vis spectrum of the green synthesized TiO_2 NPs at times ranging 3 min to two months. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)