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Synthesis of TiO_2 nanoparticles by solution combustion technique using different plant extracts

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Abstract
Titanium dioxide (TiO_2) nanoparticles was synthesized by solution combustion technique using *Nigella sativa, Euphorbia milii, Synadenium grantii* as different fuels. The obtained products were characterized by Powder X-ray diffraction (PXRD) and Diffuse reflectance spectroscopy (DRS). The X-ray diffraction pattern was in good agreement with the standard JCPDS card No.84-1285. The crystallite size was estimated by Scherrer's method and was found to ~10 nm. The optical band gap for TiO_2 nanoparticles synthesized using *Nigella sativa,Euphorbia milii, Synadenium grantii* as a fuel were found to be 3.37,3.41 and 3.46 eV respectively.

Keywords:Nanoparticles, plant extracts, band gap

1. Introduction

Titanium dioxide nanoparticles have gained a lot of attention by the researchers due to its high physical, chemical stability, wide-energy band gap having Rutile, Anatase and Brookite phases. Therefore, it has been used in various applications such as photo catalytic activity, cancer cell treatments, gas sensors, photo-electrochemical solar cells, low cost of production, stability, high refractive index and non-toxicity [1, 2]. In recent years, Green synthesis approach has received considerable attention because the byproducts released during this reaction are nontoxic, environmentally benign reactants and solvents do not cause pollution considerably [3]. In green synthesis, plant based materials such as leaves, steam, roots, seeds, flowers, etc. are used which are the main components of metal oxides.

Different methods are used for synthesizing TiO_2 nanoparticles namely sono-chemical method, electrochemical method, microwave irradiation, solid state reaction method, Sol-gel technique, thermal decomposition technique, Hydrothermal and hydrolysis techniques [1-4]. Among these solution combustion synthesis approach is one of the fast and easiest technique for the synthesis of nanoparticles. Therefore, we have made an approach for synthesizing TiO_2 nanoparticles by SCS using *Synadenium grantii*, *Euphorbia milii and Nigella sativa as fuel.*

2. Experimental

2.1. Synthesis of TiO_2 nanoparticles

Titanium(IV) isopropoxide was procured from Sigma Aldrich and leaves of *Synadenium grantii and Euphorbia milii were* collected from Devarayana durga forest in the region of Tumkur, *and seeds of Nigegella sativa* were collected from local market in Tumkur, Karnataka, India. The clean dried leaves and seeds were powdered and sieved separately. Further, the successive solvent extraction of the powder plant materials were double distilled individually using Soxhlet apparatus at 40–60°C for 72 hrs [5]. The obtained extract was filtered and concentrated in vaccum using rotary flash evaporator and the solvent left over was completely removed on water bath and finally dried in the desiccator. Secondly, the powdered plant of *Euphorbia milii* and *Nigella sativa* was followed same above procedure in individual experiment separately. Finally, the crude extract of plant material obtained from aqueous solvents was labeled and then product was stored in air-tight glass vials in the refrigerator.

The stoichiometric ratio of Titanium (IV) isopropoxide (10ml) and plant extract (5ml) was individually taken in a beaker and stirred well for 20-30 min. The well resolved mixture was introduced into preheated muffle furnace maintained at 450±10 °C. The solution undergoes exothermic reaction resulting in voluminous TiO_2 nanoparticles. Further, the obtained particles were subjected to PXRD and DRS studies.

2.2. Instruments used

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20 May

PXRD was recorded using Shimadzu made Model PXRD-7000 by using Cu-Kα radiations at a wavelength of 1.54 Å. DRS was recorded by Lambda-35, Perkin Elmer UV-vis spectrophotometer.

3. Results and discussion

3.1. Powder X-ray diffraction (PXRD)

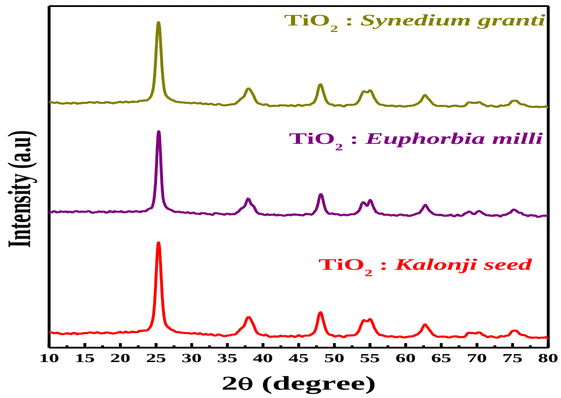


Fig 1. The PXRD patterns of TiO_2 nanoparticles

Fig.1. shows the PXRD patterns of TiO_2 nanoparticles synthesized by *Synadeniumgrantii, Euphorbia milii and Nigegellla sativa plant extracts respectively*. The obtained diffraction patters shows anatse phase and were in good agreement with standard JCPDS card no. 84-1285. Further, the particle size were estimated by Debye Scherrrer's formulae [6]

$$D = \frac{0.9\lambda}{\beta\cos\theta} \tag{1}$$

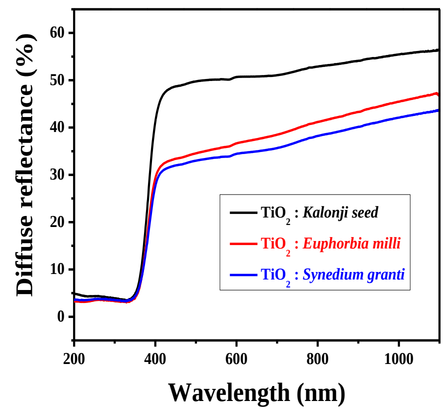
Where, λ: wavelength of incident X-rays, β: full width at half maximum of diffraction peak, θ: angle. The average size of synthesized nanoparticles was found to be 10±2 nm.
Further, phase composition was estimated using the below equation

$$X_A=100/(1+1.265I_R/I_A) \tag{2}$$

Where X_A; weight fraction of anatase in the samples, I_A and I_R; intensity of anatase (101) and rutile diffraction.

3.2. Diffuse reflectance spectroscopy (DRS)

The DRS spectra gives the information regarding the proportion of absorbed incident photons and optical energy band gap of the material. Fig.2. shows the DRS spectrum of TiO_2



nanoparticles synthesized by *Synadeniumgrantii, Euphorbia milii and Nigegellla sativa plant extracts respectively.*

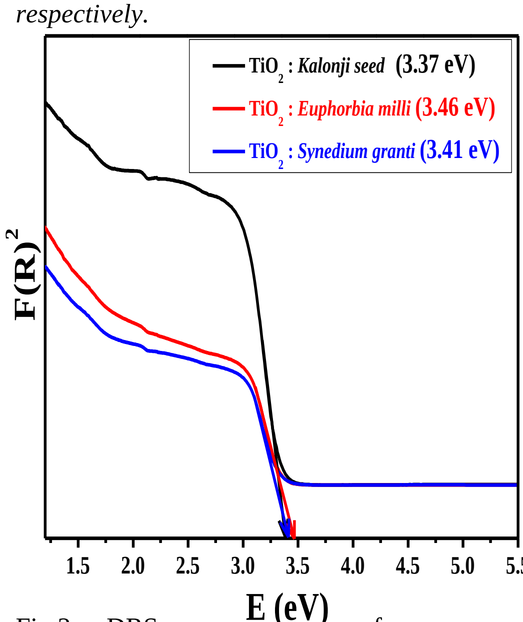


Fig.2. DRS of TiO_2 nanoparticles

Fig.3. E_g diagram of TiO_2 nanoparticles

The reflectance data obtained can be used to acquire information on the proportion of incident photons that are absorbed, and hence the optical band gap energy of the material in the sample. The Kubelka–Munk equation [7] relates the reflectance to absorption coefficient and is given by

$$F(R) = \frac{(1 - R^2)}{2R} \quad (3)$$

where R; Reflectance of the spectra

Further, inorder to estimate energy band gap (E_g) value Tauc relation [8] was reconstructed using Kubelka Munk equation and is given by

$$(F(R)h\nu)^2 = C(h\nu - E_g) \quad (4)$$

Where hν; photon energy and C; proportionality constant. The E_g was estimated by plotting $(F(R)\ h\nu)^2$ vs hν (Fig.3) and was found to be 3.37, 3.41 and 3.46 eV *for Nigella sativa*,*Euphorbia milii*, *Synadenium grantii* mediated solution combustion synthesis.

Conclusions

TiO_2 nano particles were synthesized by SCS using plant extracts as a fuel. The fuels used for the synthesis of TiO_2 nanoparticles plays an important role for varying the particle size and energy band gap values. Further, to study the role of fuels in SCS are in progress.

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