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Water disinfection through photoactive modified titania

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ABSTRACT

 $TiO₂$, N-TiO₂ and S-TiO₂ samples have been prepared by various chemical methods. These samples were characterized by X-ray diffractometer (XRD), X-ray photoelectron spectroscopy (XPS), Laser Raman spectrometer, UV–Visible spectrophotometer, field emission scanning electron microscope (FE-SEM) and transmission electron microscope (TEM). X-ray powder diffraction study reveals that all three samples are single anatase phase of titania and the crystallinity of titania decreases with sulphur doping whereas nitrogen doping does not affect it. UV–Visible (diffuse) reflectance spectra shows that doping of titania with nitrogen and sulphur shift the absorption edge of titania from ultraviolet to visible region. XPS study confirms that both nitrogen and sulphur are well doped in the titania lattice. It is observed that nitrogen occupies at both substitutional and interstitial position in the lattice of titania. FE-SEM and TEM studies demonstrate that the particles are below 50 nm range. It is found that S and N doping of titania increased its water disinfection property in the order $TiO₂ < S-TiO₂ < N-TiO₂$ under 8 W UV/UV–Visible light irradiation.

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1. Introduction

The conventional water disinfection method involves chemical treatment such as hydrogen peroxide treatment, and chlorination; but, it has disadvantage due to its toxicity and other environmental problems. Hence, there is a need for development of non-toxic, environment friendly and cost effective method for efficient water disinfection. Photocatalytic approach using titania has been the subject of study for the last two decades for various environmental applications [\[1–7\]](#page--1-0). Titania is a semiconductor and well studied system due to its low cost, nontoxicity and stability. Photo-excitation of titania leads to generation of electrons in the conduction band and holes in the valence band. These holes help in the splitting of water to produce hydroxyl radicals (OH-). These OH- radicals are extremely reactive and have redox potential as high as 2.7 V. They are non-selective and readily attack pathogenic microorganisms. This photoexcitation phenomenon will happen only if titania is excited with ultraviolet radiation because titania (anatase) band gap energy is around 3.25 eV which is corresponding to 385 nm wavelength. Among various phases of titania such as anatase, rutile and brookite, anatase phase of titania is preferred due to its high surface area and performance in the photocatalytic activity $[8,9]$. Since the solar radiation contains only 5% UV radiation, complete use of it is not possible for photocatalytic activity. Therefore, band gap of titania needs to be reduced below 3.2 eV. Numerous efforts have been made to dope

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 $TiO₂$ with certain metals to shift its absorption to the visible range, but metal-doped $TiO₂$ shows poor photoactivities due to the detrimental effect of metal ions acting as favorable sites for charge recombination $[10]$. However, it has been reported that Ag/TiO₂ powder shows good antibacterial property which further improves with the increase in crystallinity of titania $[11]$. The same material in a different form such as $Ag/TiO₂$ nanofiber membrane exhibits good photocatalytic disinfection $[12]$. Pt/TiO₂ photocatalyst has also been found to show absorption in the visible light for elimination of soil-borne pathogens [\[13\].](#page--1-0) Recent reviews by Zaleska [\[14\]](#page--1-0) and Manoj et al. [\[15\]](#page--1-0) discuss the role of various dopants in titania and their effects in shifting of absorption from ultraviolet to visible region for water treatment applications. Asahi et al. [\[16\]](#page--1-0) reported that Ndoped TiO₂ absorbs visible light due to narrowing of band gap induced by nitrogen doping and they demonstrated the photocatalytic activity in the visible light. Since the anion doped titania brought major changes in both the electronic and photocatalytic activities, now major focus has been given on these materials in order to understand the photocatalytic behaviour. In this investigation, we present the N- and S-doped $TiO₂$ photoactive materials characterization and their effect on the bactericidal properties are explained on the basis of ultraviolet and visible light absorption properties.

2. Materials and methods

2.1. Preparation of TiO₂, N-TiO₂ and S-TiO₂

Titania (TiO₂) was prepared by the hydrolysis of titanium isopropoxide with water at room temperature, followed by separation

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Fig. 1. X-ray powder diffraction patterns of titania and doped titania samples.

Fig. 2. Laser Raman spectra of titania and doped titania samples.

Table 1 Raman spectral assignments for various samples.

Wavenumber (cm^{-1})			Spectral assignment
TiO ₂	STiO ₂	NTiO ₂	
144	146	144	$E_{\rm g}$
195		195	$E_{\rm g}$
396	397	397	B_{1g}
516	517	516	$A_{1g} + B_{1g}$
638	638	638	E _g

of precipitate by filtration and drying at room temperature. The dried sample was calcined at 450° C for 3 h. In order to obtain nitrogen doped titania (N-TiO₂), titanium isopropoxide (Sigma–Aldrich, reagent grade 97%) was hydrolyzed with an aqueous solution of ammonia (Analytical reagent grade) at room temperature for 4 h. The sample thus prepared was separated by filtration, dried at room temperature, and then calcined at 450 \degree C for 3 h. The sample was designated as N-TiO₂. Similarly, sulphur doped titania $(S-TiO₂)$ was prepared by hydrolysis in the presence of Na₂SO₄

Fig. 3. FTIR spectra of titania and doped titania samples.

Fig. 4. UV–Visible (diffuse) reflectance spectra of titania and doped titania samples.

(Fischer Chemicals, India, Analytical reagent grade). The weight ratio of TiOSO₄/Na₂SO₄/ethanol/water was maintained at 13.8:7.1:55.2:77.4 and the solution were stirred, followed by aging at 80 \degree C for 5 h. The final precipitate was filtered and dried at room temperature. The dried powders were calcined at 450 \degree C for 3 h to get a white colour powder. The sample was designated as $S-TiO₂$.

Phase identification of these powder samples were determined by X-ray diffraction (diffractometer: X'PERT PRO, PANalytical, Netherlands) using Cu K α radiation source (wavelength 0.154056 nm). Surface morphology and elemental composition were studied by the field emission scanning electron microscope (FESEM, model ZEISS SUPRA⁵⁵) and Energy dispersive specroscopy (EDS) attached to it. The transmission electron microscopic (TEM) images and the selected area electron diffraction (SAED) pattern were obtained with a TECHNAI G^2 , FEI operated at 200 kV. To get the TEM images, the powdered samples were dispersed in isopropanol by ultrasonication for 20 min. Then one drop of the dispersed solution was deposited on the carbon coated copper grid. The sample was dried completely under an IR lamp before taking the observation. The UV–Visible diffuse reflectance spectra (DRS) were recorded from 200 to 800 nm by a Varian Cary UV–Vis spectrophotometer. The Raman spectra were taken in a dispersive type Download English Version:

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