



## Synthesis, characterization and photo catalytic studies of the composites by tantalum oxide and zinc oxide nanorods



K. Chennakesavulu<sup>a,b,\*</sup>, M. Madhusudhana Reddy<sup>c</sup>, G. Ramanjaneya Reddy<sup>d</sup>, A.M. Rabel<sup>b</sup>, J. Brijitta<sup>b</sup>, V. Vinita<sup>b</sup>, T. Sasipraba<sup>b</sup>, J. Sreeramulu<sup>c</sup>

<sup>a</sup> Department of Chemistry & Centre of Excellence in Energy Research, International Research Centre, Sathyabama University, Jeppiaar Nagar, Chennai 600 119, India

<sup>b</sup> Centre for Nanoscience and Technology, International Research Centre, Sathyabama University, Jeppiaar Nagar, Chennai 600 119, India

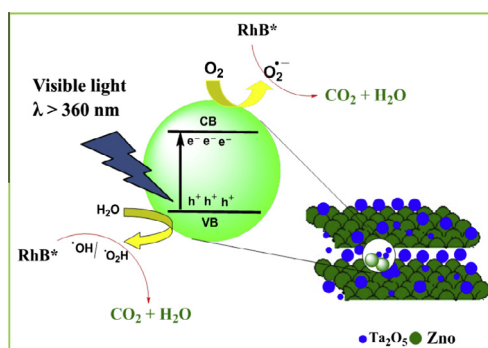
<sup>c</sup> Department of Chemistry, Sri Krishna Devaraya University, Anantapur 515 003, India

<sup>d</sup> Department of Inorganic Chemistry, School of Chemical Sciences, University of Madras, Guindy Campus, Chennai 600 025, India

### HIGHLIGHTS

- In-situ chemical synthesis of ZnO–Ta<sub>2</sub>O<sub>5</sub> composite was achieved in basic medium.
- An attempt made to impregnate the Ta<sub>2</sub>O<sub>5</sub> with ZnO nanorods and its composites.
- The composites were characterized by the FTIR, RAMAN, DRS, XRD, BET, TGA and SEM/EDX.
- The catalytic activity of nanocomposites was studied in the degradation of RhB.
- The photocatalysts were reused and compared the activity with fresh catalyst.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In-situ synthesis of ZnO:Ta<sub>2</sub>O<sub>5</sub> composites in basic medium by using tantalum chloride and zinc chloride as precursors. The prepared composites were characterized by Fourier Transform Infrared spectroscopy (FTIR), confocal Raman spectroscopy, diffuse reflectance UV–Vis spectrophotometer (DRS), X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET) method, N<sub>2</sub>-sorption isotherms, Thermo Gravimetric Analysis (TGA), High Resolution Transmission Electron Microscope (HRTEM), X-ray Photoelectron Spectroscopy (XPS), and Field Emission Scanning Electron Microscopy (FESEM/EDS). The composite materials were used as photocatalyst in the degradation Rhodamine-B (RhB) dye under visible light irradiation. The catalytic activity and removal percentage of the dye was determined by the spectrophotometric method. This indicates the percentage of degradation was more for the ZnO:Ta<sub>2</sub>O<sub>5</sub> composites. The kinetic parameter obeys *pseudo*-first order reaction. It may be due to fixed amount of the catalysts and concentration of dye solution. The catalytic activity of the recycled ZnO:Ta<sub>2</sub>O<sub>5</sub> catalyst was compared with fresh catalyst.

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### Introduction

Tantalum oxide and tantalum containing composites are most significant materials in the field of heterogeneous catalysis. The tantalum and its oxides do not occur naturally as free metals, but these are available in the form of minerals such as microlite,

\* Corresponding author at: Department of Chemistry & Centre of Excellence in Energy Research, Sathyabama University, Jeppiaar Nagar, Chennai 600 119, India. Tel./fax: +91 4424503814.

E-mail addresses: [chennanml@yahoo.com](mailto:chennanml@yahoo.com), [gonturamunom@gmail.com](mailto:gonturamunom@gmail.com) (K. Chennakesavulu).

loparite, tantalite and iimenorutile [1–4]. Tantalum oxide has high durability, electrical resistivity, corrosive resistance, super conductivity, surface area and melting point, etc. Thus its usage was essential in the catalysis, sensors, optics, coatings, batteries, drug delivery, gene delivery, photonics and microelectronics [5–7]. The composites of the tantalum oxide with the highly common oxides such as zinc, titanium, ferric oxides are the good catalytic materials for various industrial applications. Zinc oxide is among all good catalytic material owing the high catalytic activity, very less toxicity and cost effective, when compared with tantalum and niobium oxide [8,9]. Although, the combination of these composite materials were used as heterogeneous catalyst in many organic transformations such as liquid phase oxidation, gas phase oxidation, hydration, condensation, alkylation, dehydrogenation and photo degradation of the various organic pollutants [10]. The water was contaminated with the effluents released by food, agricultural, pharmaceutical and textile industries. The degradation of halo-phenols, xanthenes under UV–Visible light is challenging task [11]. The degradation of cationic dyes such as RhB, rhodamine-6G with either zinc or tantalum oxides are very slow under visible light irradiation [12]. The catalytic activity of zinc oxide was enhanced by doping it with niobium oxide and tantalum oxide. The tantalum oxide doped materials would lower the band gap energy and increase the photocatalytic performance under UV–Visible light irradiation [13].

The tantalum and niobium oxides are better substituents to the commercially available toxic chemicals such as  $H_2SO_4$ , HF,  $HNO_3$ ,  $COCl_2$  and chromic acids. The  $Ta_2O_5$  can provide the strong surface acidity and stability in aqueous medium even at high temperature for gas phase reactions. The  $Ta_2O_5$  and ZnO composites are eco-friendly materials for the many chemical transformations [14–16]. The catalytic efficiency also depends on the surface properties such as morphology and size. The percentages of tantalum oxide with host zinc oxide also alter the photocatalytic activity. The surface properties and adsorption ability of photocatalyst depends on metal oxide, which minimize the electron–hole recombination process. The homogeneity can be achieved by the in-situ nanosynthesis of the composite [17–19]. The relatively delocalized Ta state near to conduction band give more mobility for the photo induced electrons to improve photocatalytic activity [20]. The tantalum (V) posses  $d^0$  configuration allows the individual Ta atoms to contribute multiple electrons to vary the electrical conductivity. So, the  $ZnO:Ta_2O_5$  hierarchical structure can perform well, when used as catalysts in the degradation of cationic dyes [21]. The literature related to  $ZnO:Ta_2O_5$  composite with various percentages of  $Ta_2O_5$  were yet to be reported in the degradation of RhB.

The present study aims the in-situ chemical synthesis of  $ZnO:Ta_2O_5$  composites. The composites were used as photocatalyst in the degradation of RhB under visible light irradiation. Spectrophotometric method was used for the determination of percentage degradation of dye. The recycled catalyst catalytic activity was compared with the fresh catalyst.

## Experimental

### Materials

Zinc chloride, Ammonia, (Merck Pvt. Ltd, India), Tantalum chloride and Rhodamine-B (Sigma–Aldrich, India) were used, without further purification. Millipore water was used throughout the work.

### Physicochemical measurements and characterization

The FTIR spectra were recorded on a FTIR Perkin–Elmer 8300 spectrometer with KBr disk. The UV–Visible absorption spectra of

liquid samples were analyzed on a Perkin Elmer Lambda-35 spectrophotometer. The UV–Vis/DRS analyses were carried out on a JASCO-V-670 spectrophotometer. Raman spectra were on a NANO PHOTON11i confocal Raman microscope using a He–Ne laser emitting at 532 nm. The crystalline nature of the  $ZnO:Ta_2O_5$  was ascertained by the powder X-ray diffraction using Rigaku XRD-Smart Lab with  $Cu K\alpha_1$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). TGA experiments were performed with Versa Therm Cahn Thermo balance TG-151 with a sensitivity of 10  $\mu\text{g}$ . It was conducted between the temperature range of 30–900  $^\circ\text{C}$  with  $20 \pm 0.01 \text{ mg}$  of the samples and the analyses were carried out at a heating rate of 10  $^\circ\text{C}/\text{min}$  under static air atmosphere. The  $N_2$  adsorption–desorption isotherms and BET surface area measurements were carried out on a Micrometrics ASAP (Model 2020) surface area analyzer with nitrogen and helium gases with a purity of 99.999% at  $-196 \text{ }^\circ\text{C}$ . The FESEM was obtained on a SUPRA 55- CARL ZEISS scanning electron microscope. The XPS analysis was carried out on XM1000 Omicron nanotechnology XPS system with Al- $K\alpha$  monochromatic wavelength. The samples were made in to pellets and were used as such for X-ray Photoelectron Spectroscopic (XPS) studies. HRTEM analysis was carried out by using a FEI TECNAI G2 (T-30) transmission electron microscope with an accelerating voltage of 250 KV.

### Synthesis of the Zn–Ta composites

Anhydrous zinc chloride of 0.05 M was well dispersed in 200 mL of ethanol solution, in double neck round bottom flask, 0.3 M ammonia solution was added dropwise in above solution at room temperature. The resulting precipitate was centrifuged and repeatedly washed with the milli-Q water. The white precipitate was heated in furnace at 200  $^\circ\text{C}$  for 4 h. The well dispersed ethanolic solution of  $TaCl_5$  containing 55 mg/160 mg/260 mg and 360 mg were added during the synthesis of composites. The resulted sol–gel mixture was centrifuged and washed with the milli-Q water. The undoped ZnO and composites prepared with 1%, 3%, 5% and 7% of tantalum oxide in ZnO were represented as Zn– $Ta_0$ , Zn– $Ta_1$ , Zn– $Ta_3$ , Zn– $Ta_5$  and Zn– $Ta_7$  respectively.

### Photocatalytic degradation of the RhB under visible light irradiation

The photocatalytic activity of the Zn– $Ta_0$ , Zn– $Ta_1$ , Zn– $Ta_3$ , Zn– $Ta_5$  and Zn– $Ta_7$  was carried out in a cylindrical glass reactor containing RhB under visible light. Each catalyst of 0.05 g of Zn– $Ta_0$ , Zn– $Ta_1$ , Zn– $Ta_3$ , Zn– $Ta_5$  and Zn– $Ta_7$  and 0.01 mmol of 100 mL aqueous dye solution was added. The reaction conditions were optimized in dark at room temperature and start irradiation under visible light ( $>360 \text{ nm}$ ). The removal percentage and consequent spectral changes at predetermined time intervals were monitored by the UV–Visible absorption spectra at  $554 \pm 1 \text{ nm}$  for 3 h. The percentage conversion is calculated from Eq. (1).

$$A = \varepsilon \cdot c \cdot l \quad (1)$$

Here  $\varepsilon$  = molar extinction coefficient [ $M^{-1} \text{ cm}^{-1}$ ],  $c$  = RhB concentration,  $l$  = path length of cuvette (1 cm).

## Results and discussion

### FTIR analysis

The FTIR spectra of Zn– $Ta_0$ , Zn– $Ta_1$ , Zn– $Ta_3$ , Zn– $Ta_5$  and Zn– $Ta_7$  were given in Fig. 1(a–e). The composites shows peak in region of  $1665 \text{ cm}^{-1}$  was due to the in-plane bending vibration of the O–H group. The bands in the range of  $3300\text{--}3560 \text{ cm}^{-1}$  were appeared due to the surface hydroxyl groups. A strong band around  $446 \text{ cm}^{-1}$  was assigned to the stretching frequency of Zn–O group

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