



# Photocatalytic degradation of textile-dyeing wastewater by using a microwave combustion-synthesized zirconium oxide supported activated carbon

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## ABSTRACT

Zirconium oxide nanoparticles supported activated carbon (ZrSAC) was successfully synthesized using a simple microwave irradiation method. The derived UV light sensitive ZrSAC composites were characterized using powder X-ray diffraction (XRD), Fourier transform infra-red spectroscopy (FTIR), high resolution scanning electron microscope (HRSEM) with energy dispersive X-ray analysis (EDX), X-ray photoelectron spectroscopy (XPS), and Brunauer Emmett Teller (BET) surface area analyzer. Optical properties of ZrSAC composites were studied using UV–vis diffuse reflectance spectroscopy and photoluminescence spectroscopy (PL), which exposed prolonged light absorption in UV light region and hold better charge separation capability, respectively as compared to pure ZrO<sub>2</sub>. The photocatalytic activity was tested by the degradation of textile dye waste water (TDW) under UV light irradiation. Chemical oxygen demand (COD) of TDW was measured before and after the photocatalysis experiment under UV light to evaluate the mineralization of wastewater. The results demonstrated that ZrSAC composites showed a splendid photocatalytic enrichment over pure ZrO<sub>2</sub>. The coordinated blending of the oxygen vacant sites, structural defects of ZrO<sub>2</sub> along with electron transmission capacity and presence of surface oxygen on AC has led to the lasting light absorption, delayed charge recombination, and sustenance, which favor the enrichment of the photocatalytic activity of ZrSAC.

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## 1. Introduction

The textile industries are generating large amount of effluents, which contains considerable amount of organic dyes [1]. The letting out of these effluents into water bodies creates great ecological damage, and poses enormous threat to aquatic creatures, and human life [2].

The textile effluent is characterized by high chemical oxygen demand (COD). The textile industries are making huge effort to reduce the COD level, by many methods, such as, hydrolysis in a basic medium, chemical processes, membrane filtration, biological treatment and foam flotation [3–5]. However, these methods were inadequate for the highly toxic textile wastewaters [6]. The advanced oxidation processes (AOPs), such as, photocatalysis, Fenton and photo-Fenton has been used in the treatment of textile wastewater [7–9].

Among many treatment methods employed to treat the textile dyeing effluents, heterogeneous photocatalysis stands out as the best, because it is a profitable treatment

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process [10–13]. In the past decade, many metal oxide semiconductors such as titanium dioxide ( $\text{TiO}_2$ ), zinc oxide ( $\text{ZnO}$ ), and  $\text{ZrO}_2$  have been used as heterogeneous photocatalysts.  $\text{ZrO}_2$  is a n-type semiconductor, with a wide band gap and large negative value of the conduction band [14–16]. The photocatalytic activity of  $\text{ZrO}_2$  is usually investigated at around 250 nm, but studies were carried out for some samples in the range between 290–390 nm [17]. It was also reported that  $\text{ZrO}_2$  showed photocatalytic activity at 320 nm, due to absorption band emerging from an impurity level [18].  $\text{ZrO}_2$  was used in the oxidation of 2-propanol to acetone [19], in the photocatalytic degradation of 4-chlorophenol, 4-nitrophenol and methylene blue [20–22]. But, due to its limitation factor of high percentage of electron–hole recombination and practical difficulties, exploration is on for  $\text{ZrO}_2$  composite material [23].

Recent studies have shown that the photocatalytic efficiency of  $\text{TiO}_2$  and  $\text{ZnO}$  increased to a great extent, when it was supported on activated carbon [24]. AC support brings in benefits and advantages for a cheaper and effective waste water treatment and remediation privilege [25]. The primary advantage of AC would be its high adsorption capability, which can help to enrich the organic substrate around the catalyst, promoting the pollutant transfer process, and hence increasing the photocatalytic efficiency [26–29]. It can also increase the life time of electron–hole pairs and facilitate the target reactions [30]. The role of synergistic effect of adsorption by AC and photocatalytic decomposition by  $\text{TiO}_2$  and  $\text{ZnO}$  has been observed in the degradation of several types of organic pollutants [31–34].

In the present work, we focused on the preparation and characterization pure  $\text{ZrO}_2$  and  $\text{ZrO}_2$  nanomaterials supported activated carbon (ZrSAC) composite catalysts by microwave combustion synthesis. There are many methods available to impregnate  $\text{ZrO}_2$  onto carbon surface, but solution combustion synthesis is a productive method for the fabrication of  $\text{ZrO}_2$  onto the carbon support [35]. Microwave assisted combustion synthesis is environment friendly, rapid, and cost effective, and the resultant  $\text{ZrO}_2$  nanoparticles are pure [36].

The influence of carbon support on  $\text{ZrO}_2$  photocatalytic efficiency was investigated from the degradation of textile dyeing waste water under UV light irradiation. The as-prepared novel ZrSAC exhibited extended UV light absorption and greater adsorptivity of textile dye and enhanced photocatalytic activity than pure  $\text{ZrO}_2$ .

Prior to this study of ours, there has been no attempt made to prepare zirconium oxide supported activated carbon by microwave combustion synthesis, and also no literature is found on the use of ZrSAC for the degradation of textile dyeing waste water.

## 2. Material and methods

### 2.1. Preparation of ZrSAC

Zirconium oxide supported activated carbon (ZrSAC) was prepared by using the following procedure. The carbon support for the anchoring of  $\text{ZrO}_2$  nanomaterials was synthesized from agricultural waste. The rice husk

procured from public rice factory was activated at 800 °C using chemical activating agents, such as HF and KOH. The carbon support (AC) thus prepared had high surface area (446.44  $\text{m}^2/\text{g}$ ), pore volume (0.21  $\text{cm}^3/\text{g}$ ) and average pore diameter (0.89 nm).

Zirconyl nitrate hydrate and urea of analar grade (Merck, India) were used as the precursors for the synthesis of ZrSAC. Stoichiometric amounts of zirconyl nitrate hydrate and urea were dissolved separately in 10 ml of distilled water. Liquefied urea was added to aqueous zirconyl nitrate hydrate solution. To this mixture, 1 g of AC was added, and stirred for 5 h. The mixture obtained was heated in a microwave (2.45 GHz, 750 W) for 10 min. The product was washed with distilled water, and dried in an hot air oven at 100 °C for 1 h and is labeled as ZrSAC. Five samples of ZrSAC were prepared with varying the  $\text{ZrO}_2$  loading from 10% to 50%. The samples were named as ZrSAC1, ZrSAC2, ZrSAC3, ZrSAC4 and ZrSAC5. The pure  $\text{ZrO}_2$  was also prepared using the same procedure, without adding carbon support for comparison purpose.

### 2.2. Characterization of ZrSAC

The crystallinity of AC and ZrSAC were analyzed by using a Philips X'pert X-ray diffractometer for  $2\theta$  values ranging from 5° to 80° using  $\text{CuK}\alpha$  radiation at  $\lambda = 1.540 \text{ \AA}$ . A Perkin-Elmer infrared spectrometer was utilized for analyzing the functional groups on the surface of the samples in the spectral range of 4000–400  $\text{cm}^{-1}$ . Morphological analysis and energy dispersive X-ray analysis were done by using a JeolJSM6360 high resolution scanning electron microscope. The band gap energy of the samples was estimated from the data obtained from a Cary100 UV–vis spectrophotometer. The photoluminescence properties of the nanostructures were recorded by using a Varian Cary Eclipse Fluorescence spectrophotometer. The specific surface area and porosity was evaluated from the surface area and porosity analyzer (ASAP 2020 V3.00H, Micromeritics Instrument Corp., Norcross, GA).

### 2.3. Photocatalytic reactor set-up and degradation procedure

The photocatalytic degradation of textile dyeing waste water (TDW) obtained from dyeing industry, Tiruppur, Tamil Nadu, India was carried out in a Multi lamp photocatalytic reactor. The reactor contains low pressure mercury lamps (8/8 W) emitting 254 nm UV radiations, which is sufficient for the photocatalytic degradation of TDW. The TDW were taken in quartz reactor tubes, which can hold 100 ml of the dye solution. The procedure to study the photocatalytic degradation study is of the following sequence. The initial COD of the TDW was estimated and to it a known weight of ZrSAC or pure  $\text{ZrO}_2$  were added and kept in dark for 12 h to reach the adsorption equilibrium, and the resultant COD was calculated. This solution was taken in the photocatalytic reactor and UV irradiation was supplied for 2 h. At a regular interval of 20 min, equal aliquot was removed from the reactor tube, centrifuged and the COD was estimated to measure the extend of TDW

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