



Synthesis of TiO₂-SiO₂ composite photocatalyst for efficient dye decolorization using a custom-built photocatalytic reactor

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ARTICLE INFO

Received: 18 May 2025

Revised: 04 June 2025

Accepted: 06 August 2025

eISSN 2224-7157/© 2025 The Author(s).
Published by Bangladesh Council of
Scientific and Industrial Research
(BCSIR).

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DOI: <https://doi.org/10.3329/bjsir.v60i3.81575>

Abstract

This study addresses the urgent need for effective industrial wastewater treatment by developing a sustainable photocatalytic system. A TiO₂-SiO₂ composite photocatalyst was synthesized using the sol-gel method and applied for dye decolorization in a custom-designed photocatalytic reactor. In contrast to other designs, the reactor's U-shaped circulation system, pump, and wastewater reservoir improve UV light exposure area and photocatalytic efficiency. The performance of the reactor was evaluated and compared with a conventional laboratory reactor, demonstrating comparable efficacy. A 1:1.5 solution-to-catalyst ratio achieved complete decolorization of dye within 30 minutes. We also report the synthesis and application of a TiO₂-SiO₂ composite photocatalyst for dye decolorization. The photocatalyst, with anatase-phase TiO₂ uniformly dispersed over SiO₂. Photocatalytic activity was optimized at a 25% TiO₂ loading, which exhibited superior performance under UV irradiation. This work highlights the potential of TiO₂-SiO₂ composites and engineered reactors in promoting cost-effective and eco-friendly wastewater treatment technologies, supporting cleaner industrial practices.

Keywords: Photocatalyst; Reactor; Nanocomposite; UV Irradiation; Dye decolorization

Introduction

The population growth, economic development and improved living standard causing the increased demand in every sector like energy and fresh water. Just 2.5 percent of the water that covers the earth's surface is freshwater, despite the fact that 71% of it is. (Hasan *et al.* 2019). In current decades, the surface water that includes rivers, streams, canals and many other wetlands on the universe are frequently being contaminated due to rapid growth of readymade garments and textile industries. The effluents from these textile and dyestuffs industries mainly contains different types of strong organic dyes and huge amount of other chemical compounds. For this reason, water is getting warmer, losing dissolved oxygen and also fluctuating of its pH values day by day. In consequence, the whole ecosystem in the biosphere especially the aquatic life and its environment falls in serious hazardous situation (Dey and Islam 2015). Some developing countries have numerous rivers and abundant

water sources, but these are increasingly threatened by pollution. Industrial activities, agriculture, power generation, and other sectors contribute to water contamination, affecting both surface and groundwater quality. Without proper management, these pollutants can severely impact ecosystems and human health. (Uddin and Jeong 2021) The textile industry plays a crucial role in economic growth, yet it is considered one of the most environmentally harmful sectors if wastewater is not adequately treated. Many textile industries are located near riverbanks, leading to the direct discharge of large volumes of untreated effluents into water bodies. These effluents, containing various toxic substances, dyes, and chemicals, not only pollute water sources but also degrade soil quality. High levels of chemical oxygen demand (COD), total suspended solids (TSS), total dissolved solids (TDS), and electrical conductivity (EC) indicate increased toxicity in wastewater, posing significant environmental risks.

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To address this issue, the treatment of polluted water is essential. Governments should implement regulations and restrictions to control contamination from textile dyeing effluents, aiming to protect water quality and promote sustainable industrial practices (Sakamoto *et al.* 2019). ETPs (appropriate wastewater treatment plants) may be a useful remedy for this issue. The treatment of textile dyeing effluents is followed by three steps. First, excessive amounts of oil and suspended particles are eliminated, followed by colour control and other factors. Reverse osmosis, ion exchange, and electro dialysis are categorized as tertiary treatments (Ghaly *et al.* 2014; Suresh, 2014; Adane *et al.* 2021; Sathya *et al.* 2022). Numerous physicochemical techniques are non-destructive, including membrane separation, coagulation, and adsorption. (Patel, 2018; Tohamy *et al.* 2022). However, more potent and environmentally friendly oxidation techniques such as ozonation, supercritical water oxidation, photocatalytic oxidation, and electrochemical techniques are frequently employed in wastewater clean-up. (Honarmandrad *et al.* 2021; Radha and Sirisha, 2018; Khodadadia *et al.* 2020; Stefan, 2017; Taj *et al.* 2019). Of them, it has been demonstrated that photocatalytic oxidation is a very safe, efficient and reasonably priced method. Because of its facile manner of breaking down organic pollutants into mineral products and minimal energy usage, photo catalysis is a sophisticated oxidation process that has garnered a lot of attention (Ma *et al.* 2021). Because of the abundance of sun light, semiconductor-based heterogeneous photo catalysis is considered one of the most promising technologies for addressing water contamination (Wang *et al.* 2017). In essence, it's a chemical reaction where photonic energy is converted to chemical energy by using light energy and a catalyst.

This study seeks to design and construct a new type of photocatalytic reactor that extends the UV irradiation time on the photocatalyst by extending the surface area of pathway of water flow that will be decolorize. Different reaction settings, increasing photocatalyst efficiency, and assessing the system's capacity to break down organic contaminants in wastewater are the main areas of study. Additionally, a SiO₂-TiO₂ nanocomposite was synthesized, characterized and optimized to improve dye decolorization, indicating its potential for wastewater treatment which can contribute in sustainable and eco-friendly purification technologies.

Materials and methods

Figure 1 depicts the mechanism underlying photocatalytic activity. Generally, semiconductors like titanium dioxide (TiO₂) or ZnO acts as photocatalyst which generates hydrox-

yl and superoxide anion in irradiation of ultraviolet (UV) or visible light. Pairs of electrons and holes are created by the photocatalyst when it takes in ultraviolet (UV) light from the sun or a lit source, like fluorescent lights. When exposed to light, the electron in TiO₂'s valence band becomes excited. Equation (3) explains how the extra energy of this excited electron drives the electron to the titanium dioxide's conduction band, where it forms the negative-electron (e⁻) and positive-hole (h⁺) pair. The semiconductor is said to be in its "photo-excitation" state at this point. Photo-excitation in this material requires light with a wavelength of $3.2 \text{ eV (band gap energy) / } 1240 \text{ (Planck's constant, h) = } 388 \text{ nm}$ (Etacheri *et al.* 2015; Anucha *et al.* 2022).

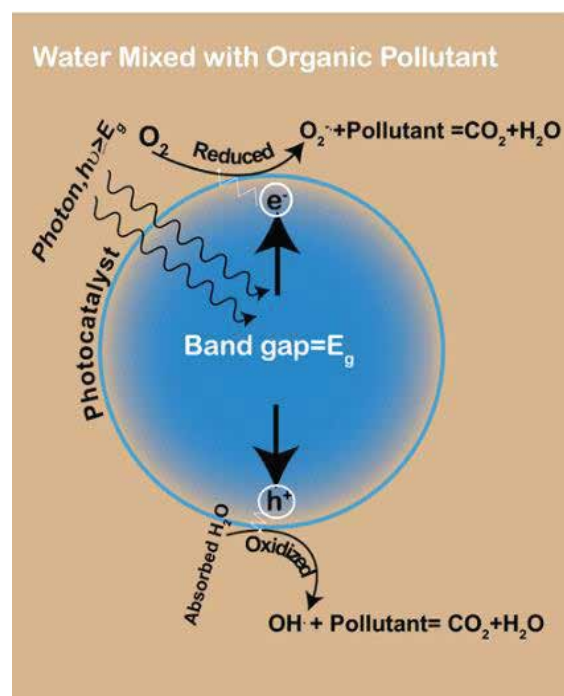
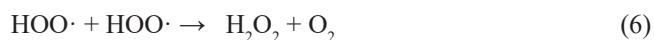
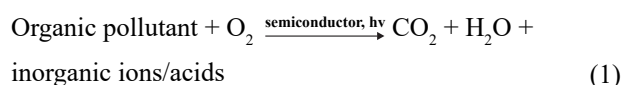


Fig. 1. Mechanism of photocatalytic reaction

When photon energy is irradiated, electrons are released from the photocatalyst TiO₂ surface. The super oxide anion (O^{-1/2}) is generated when the released electron combines with oxygen. Electrons are released from the surface of photocatalyst TiO₂ upon photon energy radiation. The released electron binds with oxygen to become super oxide anion (O^{-1/2}). Additionally, the TiO₂ surface acquires a positive charge and absorbs electrons from the surrounding moisture. Electron-depleted moisture turns into hydroxyl radicals (OH•). By using their superoxidation destructive capability, the hydroxyl radical (OH•) and super oxide anion (O^{-1/2}) break down organic materials like dyes, oils, undesirable germs, etc., into H₂O and CO₂ that are then released into the atmosphere. (Lin *et al.* 2020; Choi *et al.* 2010).



Dye decolorization by using photocatalyst material under UV irradiation can be affected by several key factors such as the design of reactor, dye concentration, dosage of catalyst, pH, light source etc. It is also observed that the presence of inorganic salts also affects the decolorization or degradation. (Sujatha *et al.* 2020). After degradation, material separation is another challenge in this process. Towards this end, Kim *et al.* designed a reactor where catalyst separation was done by using submerged microfiltration membrane. In many reactor designs, natural sedimentation of the catalysts has been achieved. The use of Nano crystalline titanium dioxide (NTO) pellets in photocatalytic reactors allowed for the avoidance of post-separation needs. (Lazar *et al.* 2012). TiO₂ pellets were demonstrated by Mc Cullagh *et al.* to degrade methylene blue dye solution (McCullagh *et al.* 2010). Three paddle-equipped cylinders inside the reactor held TiO₂ pellets. Each cylinder had UV lights installed, and a motor was attached to cause the cylinders to rotate.

Factors to be considered for measuring photocatalytic activity

(a) Photocatalytic Reactor

A few essential elements work together to form a structure in a photocatalytic reactor where the pollutants are broken down. Different types of reactors, including flat plate, honeycomb monolith, fluidized bed, fiber/membrane, and optical fiber reactors, have been developed in the past (Li *et al.* 2022; Ola and Maroto-Valer 2014; Rincon and Motta 2019). The typical components of a photo reactor include a reactor chamber, photocatalyst, light irradiation, auxiliary equipment, additives, etc.

(b) Photocatalyst

Photocatalyst activates by the photon absorption and accelerate the reaction. The main factors to take into account for photocatalytic activities are the composition and shape of the photocatalyst material. Different semiconducting materials work as photocatalyst (Hernández and Medina-Ramírez

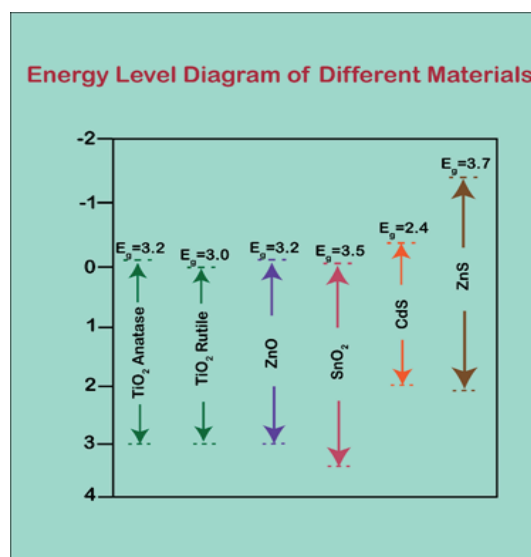


Fig. 2. Band gap of different materials

2015). The features of these determine their photocatalytic activity. For a catalyst to be photo excited, the minimum energy (light) needed must be greater than the band gap energy. Figure 2 shows the energy level diagram of different materials. Among the various semiconductors, TiO₂ is widely studied as photocatalyst material in energy related issues especially environment pollution control research sector. TiO₂ is abundant in nature, non-toxic, insoluble in water, cheap and resistant to most chemicals, such as acids, bases or solvents and that's why it takes more attention to the researchers. In certain instances, even doped mono-component photo-catalysts display low chemical stability, a narrow absorption range, and charge recombination. However, employing TiO₂ and ZnO semiconductors for photocatalytic degradation under natural solar irradiation is limited by their wide bang gaps (~ 3.2 eV). Photo degradation activities may only benefit from 4–6% of the total solar intensity (Lin *et al.* 2014; Anand and Srivastava 2016). The catalytic activity of TiO₂-SiO₂ mixed oxides was investigated, and it was shown that TiO₂ - SiO₂ had superior photocatalytic activity in comparison to TiO₂ and SiO₂, as demonstrated by UV findings (Yaseen *et al.* 2017). The semiconductors displayed above are investigated using various dyes. Several dyes, such as thionine, eosin Y, rhodamine B, methylene blue, and methylene orange, have been studied for their ability to degrade contaminants when exposed to UV and visible light using TiO₂ catalyst (Ma *et al.* 2021). Photocatalytic degradation of nitrobenzene (NB) and an azo dye was carried out using zinc oxide (ZnO) nanoparticles which were prepared by electrochemical method in another experiment (Vikky and Vimal, 2016). Here, maximum NB degradation efficiency of 98%

was observed at catalyst dosage=1 g/l, which was found as 78% at catalyst dosage=1.25 g/l. Optimizing the active surface area is essential for achieving high photocatalytic efficiency (Parul *et al.* 2020). It can accomplish this by enhancing surface morphologies and creating a surface that resembles a flower, which can support a high level of photocatalytic activity and manage a lot of surface-active sites (Zhang *et al.* 2018). One of the main obstacles in the treatment process is catalyst separation.

(c) Light Source

Photocatalyst material activates only under light irradiation. The photocatalytic activity performance is influenced by the radiation source position, spectral range, and light intensity within the reactor. During designing a reactor, constant light intensity inside the chamber must be considered. As glass cannot transmit UV light, reactor size and material availability are one of the limitations of this system. UV/visible light emitting diodes (LEDs) are employed as light sources in laboratories. Halogen or tungsten lamp, LED lamp is normally used for visible range and UVA, UVB and UVC is also used as artificial lamp.

During design of photocatalytic reactor, other parts also must be in consideration such as glass material, pump, protector etc. As silica or Pyrex glass cannot transmit UV light, quartz glass must be used for UV specially UVC light. For visible or UVA light, the reactor can use silica glass or directly.

Results and discussion

Photocatalytic reactor design and its performance analysis

In this work, to degrade the organic pollutants a photocatalytic reactor has been developed (Fig-3). Photo-catalytic reactor system that including waste water and catalyst mixing tank, a pipe comprising inlet and outlet, a pump and a rectangular type UV light source-based reaction chamber. A rectangular type UV light-based reaction chamber that comprises three UVA light of 6W fixed to upper surface and an array of U shaped several glass tubes at the bottom surface of the chamber. This idea comes from the necessity of effective illumination of light source to increase surface area so that waste water gets much irradiation time under UV light source (Sujatha *et al.* 2020). To guarantee consistent wastewater treatment, the UV photo-reactor system employs a pump-driven recirculation process and modifies the number of UV lamps to control light intensity. The system constantly circulates wastewater from the reservoir input to the UV reactor exit using 21 nm TiO₂ nanoparticles as a photocatalyst. To improve the dye-photocatalyst interaction and measure the decolorization rate, the model solution contain-

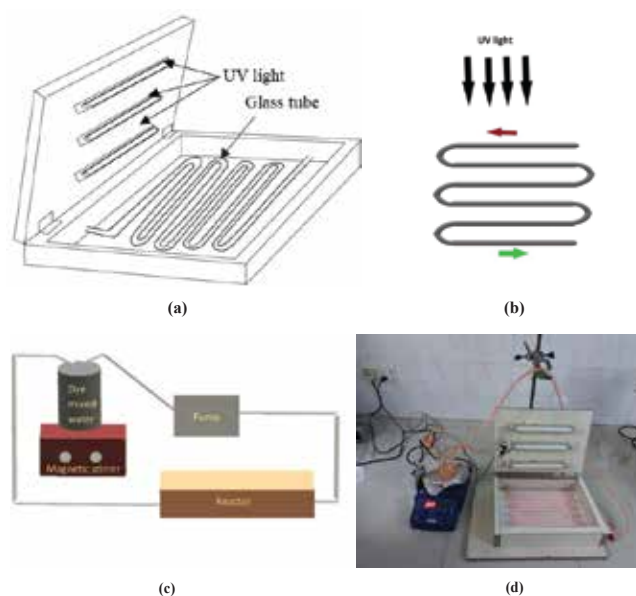


Fig. 3. (a) Reactor configuration, (b) Active area of photocatalytic method (c) Mechanism of reactor (d) Total experimental setup of photocatalytic reactor chamber

ing eosin dye was stirred at 550 rpm.

A centrifuge was used to remove the remaining TiO₂ in the sample before the dye concentration was measured. With the aid of a standard curve and absorbance values obtained from the UV-visible spectrophotometer across a wavelength range of 300–800 nm, the dye concentration was ascertained. The following equation also can be used to compute the % color removal of the dye, which is another way to assess the procedure efficiency (Hasan *et al.* 2019).

$$\% \text{ Color removal} = (C_0 - C_t) / C_0 * 100\%$$

Where, C_0 and C_t are the dye concentration in initial and at time t .

The decolorization rate of Eosin dye in varying concentrations of TiO₂ photocatalyst is displayed in Fig. 4. To ensure the reactor activities, different experimental results were compared of this reactor with a simple laboratory setup by using measuring cylinder and UV light. This figure clearly shows that as the amount of photocatalyst material grows, so does the decolorization efficiency. This results in an increase in dye adsorption on the photocatalyst surface, which boosts the production of hydroxyl radicals ($\bullet\text{OH}$). Kumar *et al.* also developed a photocatalytic reactor and evaluated the performance by degrading phenol within one hour. Here, the newly designed reactor took 180 min to degrade the dye when the

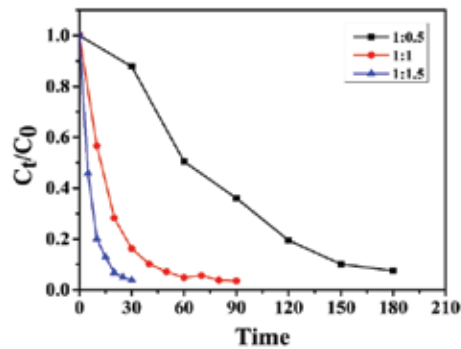


Fig. 4. Dye decolorization in different photo catalyst material ratio with visual representation

solution and photocatalyst material ratio was 1:0.5. The degradation time reduces from 90 to 30 min when the ratio was changed from 1:1 to 1:1.5 which is the same as the reactor used by measuring cylinder. This result clearly indicates that the designed reactor acts properly for photo degradation. It can be enhanced its capacity by changing the area of the reactor by keeping the same ratio. Same result was obtained in laboratory setup using measuring cylinder. Here, photon energy of UV light affects degradation which has been explained in section 2.

Preparation of TiO_2 - SiO_2 composite photocatalyst

(a) Silica Nanoparticle

Sol-gel route has been obtained to synthesis silica nanoparticles. The production process made use of ultra-high pure components such as n-hexane, sodium hydroxide (NaOH), tetraethyl orthosilicate (TEOS), and cetyltrimethylammonium bromide (CTAB) (Fig. 5). These materials were used in a molar ratio TEOS: CTAB: H_2O : NaOH: n-Hexane = 1: 0.12: 1268: 0.32: 2.7. CTAB and NaOH were mixed in the aqueous solution. The aforementioned solution was combined with n-hexane after it had cleared. To enable the separation of the polar and non-polar phases, the stirring was ceased. TEOS was gradually added to the mixture while being vigorously stirred. The solution was filtered and washed with methanol. The filtered gel was dried overnight at 60°C. The obtained

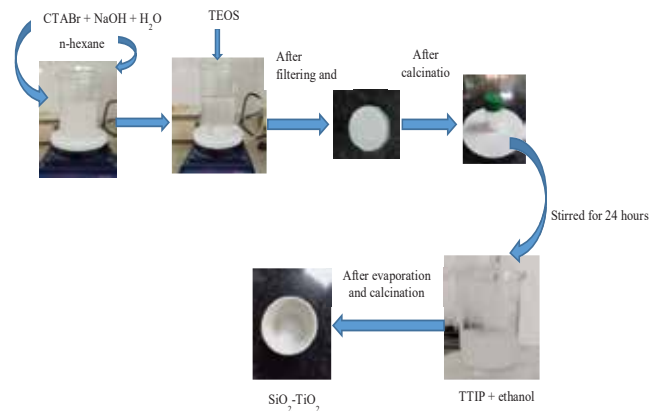


Fig. 5. Schematic representation of synthesis method of SiO_2 - TiO_2 nanocomposite

dried powder was calcined at 550°C for 5 hours.

(b) TiO_2 nanoparticle into Silica Nanoparticle

Titania nanoparticles were synthesized by dissolving titanium isopropoxide in ethanol. The mixture was added to the calcined silica. The amount of titania incorporation varied in 10 wt%, 20 wt%, 25 wt%, 30 wt% and the samples were labelled as S-10%, S-20%, S-25%, S-30%. The mixture solution was stirred at room temperature for 24 hours. The solution was kept in the open air for evaporation and the obtained solid was then calcined for 5 hours at 550°C.

An X-ray Diffractometer (GBC-XRD, EMMA) with a step size of 0.02 deg. and a $\text{Cu_K}\alpha 1$ ($\lambda = 1.54062\text{\AA}$) radiation source operating at 35.5 kV and 28 mA was used to perform the structural and phase identification measurements. The thin films were subjected to morphological and elemental analysis using a dispersive electron X-ray (EDX) scanning electron microscope (Carl Zeiss, EVO 18). FT-IR (Frontier Perkin Elmer, UK) samples of the solid coagulants were prepared as potassium bromide (KBr) pellets. An integrating sphere and a UV-VIS-NIR (Hitachi, UH4150) spectrophotometer was used to determine the optical transmittance.

3.3 Characterization of TiO_2 - SiO_2 composite photocatalyst

(a) Structural analysis

X-ray diffraction (XRD) pattern of synthesized SiO_2 and TiO_2 - SiO_2 composites is depicted in Fig. 6. Each composite sample exhibits good diffraction patterns, with all diffraction peaks showing good agreement with pure TiO_2 (anatase phase). The composite photocatalyst exhibits no rutile phase characteristic diffraction peaks. All the samples were calcined at temperature 550°C. The rutile phase is widely

thought to arise when the TiO₂ is calcined at a temperature of roughly 650°C (Taj *et al.* 2019). The structure of the synthesized SiO₂ nanoparticles is amorphous. According to JCPDS file 73-1764, the primary diffraction peaks in the XRD spectrum were associated TiO₂ peaks in the anatase phase. At $2\theta = 25.23^\circ, 37.71^\circ, 47.72^\circ, 54.16^\circ,$ and 62.54° , the diffraction peaks were observed. These values correspond to the

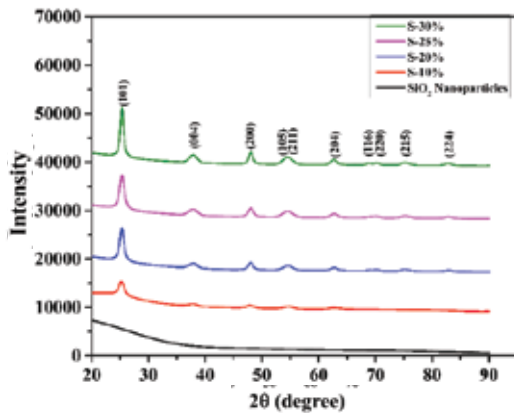


Fig. 6. XRD pattern of SiO₂ and SiO₂-TiO₂ nanocomposite material

corresponding tetragonal crystal planes (101), (004), (200), (105), and (204), in that order.

(b) FTIR Spectroscopy Analysis

SiO₂ and SiO₂-TiO₂ composites' FT-IR spectra (500–4000 cm⁻¹) show structural alterations and functional groups as the TiO₂ content rises. The peaks at 3436 cm⁻¹ and 1628 cm⁻¹, respectively, represent -OH stretching and bending vibrations, whereas the diminishing SiO₂ absorption bands indicate TiO₂ coating (Shabalina *et al.* 2022). Stretching of Si-O-Si symmetrically and asymmetrically is related to 805

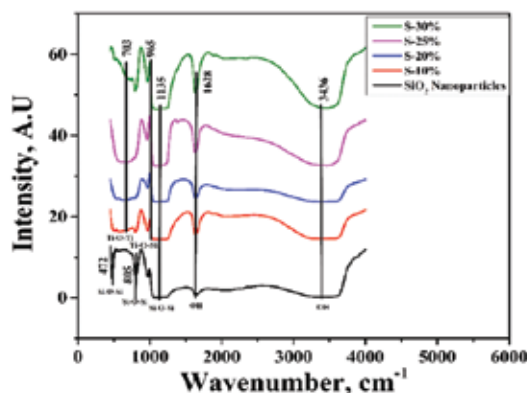


Fig.7. FTIR of SiO₂ and SiO₂-TiO₂ nanocomposite

cm⁻¹ and 1135 cm⁻¹ (Lin *et al.* 2014). The vibration's peak 472 cm⁻¹ belonged to the bending mode as a weak band. Meanwhile, the vibration of the Ti-O-Ti bond stretching was revealed by the peaks found at 703 cm⁻¹ (Ma *et al.* 2021; Wang *et al.* 2017). In mixed materials, the band located at 945 cm⁻¹ is superimposed onto the band of the Si-O-Ti stretching mode, demonstrating the connection in TiO₂-SiO₂ composite material. The stretching mode of Si-OH or Si-O groups is thought to be responsible for this band (Etacheri *et al.* 2015; Anucha *et al.* 2022). The FTIR result at Fig. 7

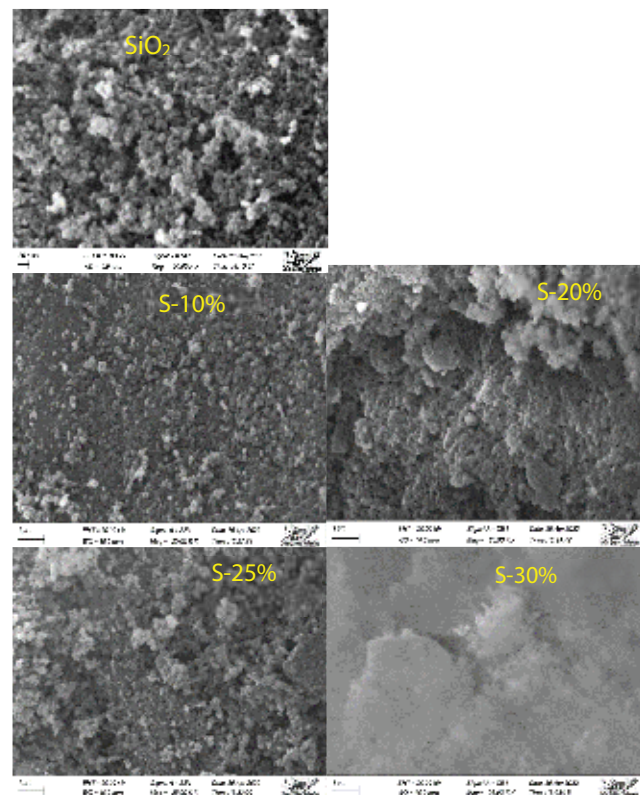


Fig. 8. SEM images of TiO₂ and SiO₂ nanocomposite

confirms the full breakdown of the organic compounds to generate materials with a high degree of purity, and the absence of any residues of contaminants.

(c) SEM and EDX analysis

SiO₂ nanoparticles and TiO₂-SiO₂ composites were subjected to scanning electron microscopy (SEM) analysis (Fig. 8) in order to ascertain the morphology and form of the particles. It is indicated that the incorporation of TiO₂ on SiO₂ has no significant effect on the surface morphology. Particles are found slightly agglomerated. Particles are densely packed together forming the surface of the agglomerates (Zhang *et al.* 2019). Agglomerated particle formation is aided by the

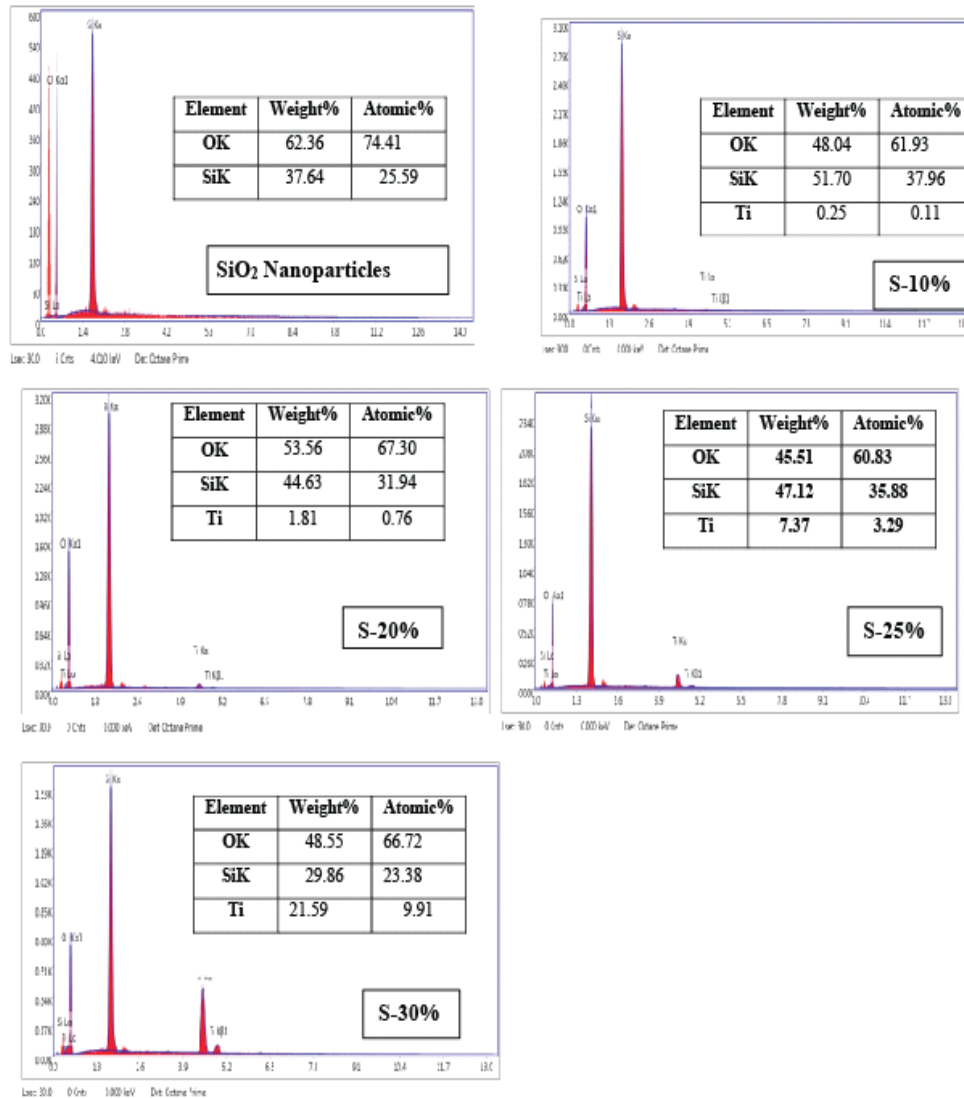


Fig. 9. EDX of SiO₂ and TiO₂-SiO₂ nano composites

greater specific surface energy and the larger specific surface area. SiO₂ nanoparticles, S-10%, S-20%, and S-25% samples are aggregation of extremely small particles. This enhances the photocatalytic activity by increasing the specific surface area and roughening the surface of bigger particles (Ma *et al.* 2021). Due to the excess TiO₂, which also supports the decolorisation curve, the addition of TiO₂ had a major impact on the morphology of the samples, which did not display any grain-like appearance. From EDX mapping (Fig. 9), it is observed that photocatalysts material uniformly dispersed. The principal components found in the materials are Ti, Si, and O. The area under each

element peak is used to determine the elemental composition (of both weight percent and atomic percent) and compute the Ti/Si ratio of each sample. We can infer from the result that the compositions that were pre-fixed in the sol were nearly preserved in every film because of the semi-quantitative character of the EDS analysis.

3.4 Application of TiO₂-SiO₂ composite for efficient dye decolorization

SiO₂ and TiO₂-SiO₂ nanocomposite materials have been synthesized for the application of photo degradation. Here, methyl orange (12 ppm) was used as model solution. The

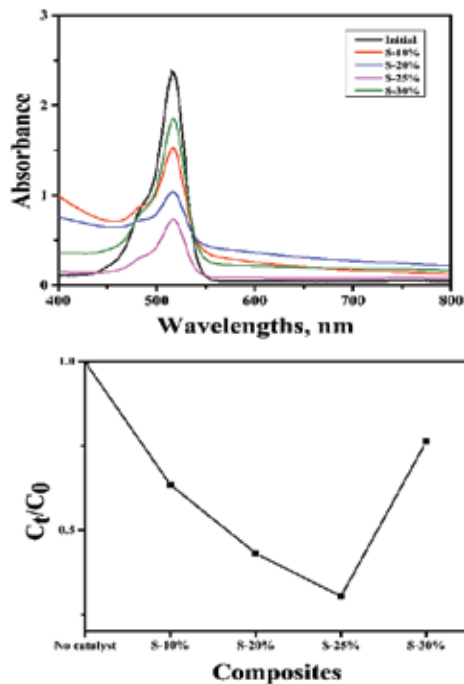


Fig. 10. Dye decolorization of methyl orange solution using SiO₂-TiO₂ nanocomposites

experiment was done at room temperature (25°C). The solution was stirred with nanocomposite under UVA light for 5 hours. Fig. 10 shows the decolorization rate with the change of amount of composites used in the experiment. Photocatalytic activities of these materials were dedicated by get rid of methyl orange as anionic dyes model with molar concentration 12 ppm under UV irradiation where methyl orange show absorption spectra peaks at 517 nm wavelengths, with about 300 min irradiation time. The decolorization of organic dye used in this study could be explained by the oxidative dye photo degradation, the photocatalytic performance was significantly improved with TiO₂-SiO₂ NCs due to their higher surface area, improved dispersion, and better adsorption of reactants. SiO₂ helps suppress electron-hole recombination and enhances the stability of the composite which make the nanocomposite more efficient in photocatalytic applications (Zayed *et al.* 2022). TiO₂-SiO₂ NCs with 25% TiO₂ were showing the best performance. After that, decolorization seemed lower which may be due to the excess number of inactive TiO₂ on surface.

Conclusion

A photocatalytic reactor operates depending on the operation mode, irradiation sources, photocatalysts and pollutants parameters (concentration, composition, pH etc.). Some other components also use for the support to ensure a proper

reaction. In this work, a photocatalytic reactor was designed and developed by using glass tube and UVA light in a closed system. The dye mixed water flowed through the system by pump and recirculate that in a loop. The performance of this reactor was studied by using commercial TiO₂ as photocatalyst and compared with the laboratory scale simple reactor. Eosin dye was completely removed in a same pattern for both reactors. SiO₂-TiO₂ nanocomposite were also obtained by sol gel synthesis as photocatalyst material. They were characterized by XRD, FTIR and SEM. The incorporation of TiO₂ was varied and found all samples to be effective in the decolorization of dye molecules. Highest removal efficiency of methyl orange dye decolorization was obtained in composite with 25% of TiO₂ into SiO₂. With the help of the effective TiO₂-SiO₂ nano composite and the specially constructed reactor, this research could result in a sustainable water treatment technique.

Acknowledgement

The authors acknowledge Bangladesh Council of Scientific and Industrial Research (BCSIR) authority for providing the grant for doing R&D work (Development of Photocatalyst material for waste water treatment; 39.02.0000.011.14.128.2020/636) and Islamic Development Bank for granting Post-Doctoral Research Fellowship (Contract no.: 2020-347493). The authors also gratefully acknowledge Institute of Glass and Ceramics Research and Testing (IGCRT) for the support in XRD measurements.

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