Recycling of TiO$_2$ containing waste and utilization by photocatalytic degradation of a reactive dye solution

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ABSTRACT

Recently, the utilization of wastes, recovery of high value-added products from waste, and their use as raw materials in other industries with the logic of industrial symbiosis has an important issue day by day. In this study, removal efficiency of Reactive Orange 16 (RO16) dye from aqueous solution was studied using TiO$_2$ catalyst recycled from an industrial waste effluent. The recycling of TiO$_2$ waste was acquired from a paints manufacturing industry by sintering the TiO$_2$ containing waste. The catalyst usability of TiO$_2$ containing powder was then investigated in the removal of RO16 dye by photocatalytic oxidation process. In photocatalytic oxidation studies, TiO$_2$/UV and TiO$_2$/UV/H$_2$O$_2$ processes were investigated, where the effect of annealing temperature, the effect of H$_2$O$_2$ concentration, the adsorption and photocatalytic effects on RO16 dye removal were studied. The results showed that 100% RO16 removal was obtained for the 10 mg/L initial RO16 concentration using TiO$_2$ (1 g/L)/UV. At high initial RO16 concentration (50 mg/L), 100% RO16 removal was achieved by adding 10 mM H$_2$O$_2$.

Key words | photocatalysis, reactive dye, recycled TiO$_2$, sintering effect

HIGHLIGHTS

- TiO$_2$-containing wastes consisting of paints manufacturing was used as catalyst.
- Reactive Orange 16 (RO16) was degraded by photocatalytic oxidation process.
- 100% RO16 removal can be achieved by adding 10 mM H$_2$O$_2$.

GRAPHICAL ABSTRACT

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INTRODUCTION

Textile industry has recently gained and expanded its facilities globally. Textile industry poses serious environmental issues due to high consumption of water and excessive use of chemical/paint and eventually generating problematic wastewater (Sarayu & Sandhya 2012; Pang & Abdullah 2013). In general, chemical and classical biological activated sludge systems are used in wastewater treatment. However, toxic components and color cannot be removed with such treatment systems, despite its effective elimination of organic matter. It is an immense problem due to decrease light transmittance in the receiving media, either for plant or living life, if color containing wastewater is discharged to the receiving environment without any treatment (Vakili et al. 2017). In addition, untreated wastewater has a harmful effect and impact for human being and living organisms because of containing toxic and carcinogenic dyes (Verma et al. 2012).

Advanced oxidation processes (AOPs) is used quite widely in the previous years because of their advantage of converting toxic and permanent organic matters into harmless final products (Ghanbari & Moradi 2015). Photocatalytic advanced oxidation process form hydroxyl radicals with catalyst and UV light. Moreover, dye molecules are converted to end products like water (H2O) and carbon dioxide (CO2). The mechanism described earlier is represented by Equations (1)–(5) as illustrated below (Thiruvenkatachari et al. 2008). TiO2/UV is a photocatalytic oxidation process that is widely used for dyes removal in textile industry (Thiruvenkatachari et al. 2008; Han et al. 2009; Šíma & Hasal 2013). Nevertheless, TiO2 catalyst causes an additional cost in oxidation processes. Thus, it can be a good option to use waste TiO2 catalyst. TiO2 containing wastes used as catalyst in photocatalytic oxidation can significantly reduce operating costs of the process.

\[ \text{TiO}_2 + h\nu \rightarrow e^- + h^+_\nu \]  
\[ \text{O}_2 + e^- \rightarrow \text{O}_2^\cdot \]  
\[ h^+_\nu + \text{Organic} \rightarrow \text{CO}_2 \]  
\[ h^+_\nu + \text{H}_2\text{O} \rightarrow \text{HO}^\cdot + \text{H}^+ \]  
\[ \text{HO}^\cdot + \text{Organic} \rightarrow \text{CO}_2 \]

The aim of this study was to investigate the use of waste TiO2 catalyst as recycled material in the photocatalytic oxidation process and RO16 dye removal that has got two sulfonate groups with negative charges in aqueous solution. This dye has pretty dying properties, especially for silk and cotton materials. Nevertheless, it is extremely resistant to conventional wastewater treatment methods (Gomes et al. 2011). The effect of sintering temperature, catalyst amount, and dye concentration were investigated for the decolorization of a reactive dye solution. The reuse number of the catalyst was also tested.

MATERIAL AND METHODS

Chemicals

Hydrogen peroxide (H2O2) aqueous solution (37%) was supplied from Sigma. Reactive Orange 16 (RO16) dye was used as reactive dye obtained from Dystar. The chemical structure and the most important properties of the dye were shown in Table 1. The required lower concentrations (10, 25, and 50 mg/L) were prepared by dilution using the stock solution (100 mg/L).

Waste TiO2 catalyst

In the paints manufacturing industry, industrial wastes generated during production are called paint waste sludge. It is well known that paint mud is a hazardous waste material. TiO2, calcite, dolomite, chemical binders, and auxiliary chemicals are used in paint production. Due to the high

| Dye name        | Chemical structure | Formula and Molecular weight (g/mol) | UV absorption λmax (nm) |
|-----------------|--------------------|-------------------------------------|-------------------------|
| Reactive Orange 16 (RO 16) | ![Chemical structure](image.png) | C20H17N3Na2O11S3 617.54 | 493 |
consumption of TiO₂ in the paint industry, there is also a high proportion of TiO₂ in paint waste sludge. In this study, waste TiO₂ powders were prepared and recycled from paint mud. The waste TiO₂ powders was calcined at 300, 600, and 900 °C for 1 h in an ambient atmosphere.

Photocatalytic activity experiments

Experimental studies were performed in a batch Pyrex photo-reactor (column-shaped) with 500-mL capacity. All properties related to the reactor were given elsewhere (Bilici et al. 2019). All experiments were performed using 200 mL dye solutions. To accelerate the oxidation process, ambient air is blown from the bottom of the reactor at a flow rate of 150 mL/min. Six UVA lamps (Philips TL8 W Actinic BL, emitting wavelengths at 365 nm) were placed around the reactor in hexagonal position. The reactor assembly was also surrounded by an aluminum-coated film for uniform reflection. In the pyrex reactor, temperature of the reaction was kept around 25 ± 1 °C with cooled air. During the reaction (180 min), samples were taken from the reactor at determined time and centrifuged at 6,000 rpm for 5 min. Maximum absorbance was measured by using a UV–vis spectrophotometer (T90 + UV/VIS Spectrometer, PG Instruments Ltd) to calculate dye removal efficiency. All experiments were performed in duplicate. Dye removal efficiency was calculated using Equation (6):

\[
\text{Dye Removal Efficiency (\%)} = \left(\frac{C_i - C_f}{C_i}\right) \times 100
\]  

where \(C_i\) was the initial dye concentration and \(C_f\) was the dye concentration after defined reaction time \(t\) (min).

Reuse stability of waste TiO₂ powders catalyst was also tested with five consecutive reuse. After each experiment, the treated solution was discharged from the reactor after the catalyst was precipitated. Then, a new solution was filled into the reactor and the catalyst was used again without any additional treatment of the catalyst.

Characterization methods

The crystal structure of waste TiO₂ powders was investigated by X-Ray Diffraction (XRD, Rigaku, Dmax-Rapid II) with an X-ray source of Cu Kα radiation (\(\lambda = 0.1692 \text{ nm}\)). The scattering angle (2θ) was scanned from 2° to 80° at a scanning speed of 6°/sec. The X-ray tube voltage and current were fixed at 40 kV and 40 mA, respectively. Zeta potential measurements were achieved using a Particle Size Analyzer 90 plus (Brookhaven Instruments).

RESULTS AND DISCUSSION

Characterization of waste TiO₂ powders

XRD analysis

XRD analysis (Rigaku Smartlab, Texas, USA) were performed to identify crystal structure and lattice parameters of synthesized powder materials (Figure 1). The sample consisted mainly of calcite (CaCO₃) and Talc (Mg₃Si₄O₁₀(OH)₂) minerals. Waste TiO₂ powders were rutile form and showed intensive peaks at 27.438°, 36.040°, and 54.321°. XRD results have given a better explanation of high RO16 removal efficiency when waste TiO₂ powder was calcined at 900 °C of sintering temperature. Si mineral is known to diffuse at high levels into the film at relatively modest annealing temperatures. In addition, Si is known to be a grain growth inhibitor and to increase the temperature required for recrystallisation of anatase. It means that Si mineral represents the main contaminant among TiO₂ powder components. Therefore, it was clear that the level of contamination was dependent on annealing temperature (Lin et al. 2013).

SEM-EDX analysis

Scanning electron microscope fitted with energy dispersive X-ray spectrometer (SEM-EDX) analyses were elaborated to study the particles morphology and the homogeneity of the TiO₂ distribution. Figure 2(a)–2(d) show the SEM images...
analysis of the studied samples: raw waste TiO$_2$ powder and samples calcined at 300, 600, and 900 °C, respectively. The different samples displayed irregular morphology with an average size of 2 μm. It is obvious that the annealing temperature has impacted particles morphology, shape, and surface roughness. The EDX spectra and SEM-EDX elemental mapping spectra of the waste powder at different conditions showed the elementary components (O, Mg, Al, Si, K, Ca, and Ti) with uniform distribution. The SEM-EDX elemental mapping spectra of all the studied samples further confirmed the presence of Ti with relatively similar amounts of about 5.67 ± 0.21 wt.%. The calculated concentrations by EDX were found relatively similar for the rest of found elements. The EDX peaks revealed that each of O, Mg, Al, Si, K, and Ca contents were uniform concentration in the different investigated samples. However, only Figure 2(a), which corresponds for the raw TiO$_2$ waste, that affirmed the presence of the known spherical shape particles of TiO$_2$, which does not clearly display in Figure 2(b) and 2(c) indicating the formation of TiO$_2$ fine particles after applying 300, 600 and 900 °C of annealing temperature on raw material. These results suggest that, annealing temperature has just effected surface morphology and roughness, but not the waste powder composition.

Zeta potential analysis

Waste TiO$_2$ powders and RO16 dye were analyzed for surface charges as zeta potentials in different pH ranges and the values were represented in Figure 3. The catalyst powders calcined at 300 °C and 600 °C had negative values at pH solution between 3 and 9, whereas the catalyst powders calcined at 900 °C had positive value below pH 7.5. The isoelectric point (where zeta potential is zero) can only be determined for the annealing temperature of 900 °C which is around the value of pH 7.5. At 900 °C of temperature, the zeta potential of the TiO$_2$ colloid reaches the highest positive values at lower pH values, which means that at lower pH, this colloid accepts more negative ions. It is known that the grain size increases significantly upon

![Figure 2](https://example.com/image1)

**Figure 2** | SEM images of waste TiO$_2$ powders (a) Raw, (b) 300, (c) 600, and (d) 900 °C.

![Figure 3](https://example.com/image2)

**Figure 3** | Zeta potential of waste TiO$_2$ powders calcined at different temperatures vs. pH.
recrystallisation, (Lin et al. 2013), where interaction between particles is more important giving positive zeta potential in lower pH values (Azouri et al. 2006). At the same temperature, at 900 °C, and at higher pH, the strong reduction of the zeta potential from positive to negative value, can be explained by charge exchange and redistribution due to this interaction (Azouri et al. 2006). In the other temperatures, negative zeta potential explains that there are other ions present with TiO₂ effecting its surface charge (Azouri et al. 2006). The obtained results explain the strong catalyst surface adsorption phenomenon because of different surface charges (Bilici et al. 2019).

The effect of sintering temperature

The waste TiO₂ powder was calcined at different annealing temperature (300, 600 and 900 °C) to investigate the relationship between annealing temperature and photocatalytic oxidation of dye. Figure 4 showed the effectiveness of waste TiO₂ powders calcined at different temperatures for RO16 dye removal efficiency. The results showed that calcination of the waste TiO₂ powders significantly affected RO16 dye removal efficiency which reached 100% after 900 °C calcination for 180 min. It could be explained as calcination of the waste TiO₂ powders changed zeta potential (ZP) values of the powders as illustrated in Figure 3. The ZP of lower pH is high and drops with the increase of pH. However, it showed a particularity for 900 °C of sintering temperature, where zeta potential values were shifted from positive (+6.00 mV) to negative side (−6.71 mV) between pH values of 3 and 9 as discussed subsequently. The total removal of RO16 dye for sintering temperature of 900 °C could be explained by the presence of positive charge on the surface of TiO₂ powder surfaces. Hence, the anionic dye RO16 with a negative charge was totally adsorbed on the surface of the positively charged TiO₂ through a strong electrostatic attraction and the electrostatic interaction was beneficial for improving the adsorptive property and eventually the degradation or removal efficiency. Oppositely, TiO₂ prepared or calcined at the other temperatures, 300 °C, 600 °C or raw material, showed very low RO16 removal efficiencies. These results could be attributed to the repulsion between the dominant different same charges in the suspension and the anionic dye (Azeez et al. 2018).

Adsorption experiments for RO16 dye

Adsorption experiments were also performed to investigate the adsorption effect of waste TiO₂ powders on the removal of RO16 dye solution using 1 g/L catalyst loading. At this stage, initial dyes concentration (10 mg/L) was selected for better and clear assessing of adsorption affinity. Results in Figure 5 indicated that in the first 15 min, for both mechanisms adsorption and photocatalysis, the initial increase of dye removal efficiency was caused by the physical phenomena (adsorption) between the TiO₂ catalyst and RO16 dye before the suspension took the effect of the UV light (Mezugi et al. 2014). After sintering of waste TiO₂ powders at 900 °C, zeta potential of powders shifted towards the positive region. Therefore, RO16 dye molecules were adsorbed on waste TiO₂ powders due to strong negative zeta potential of RO16 dye and positive zeta potential of waste TiO₂ powders which acted both strong adsorbent and oxidative catalyst material (Figure 5).

Figure 4 | The effect of annealing temperature on photocatalytic oxidation of RO16 dye (Dye concentration: 10 mg/L; catalyst loading: 1 g/L).
Figure 5 | The adsorption and photocatalytic effects on RO16 dye removal efficiency (Catalyst loading: 1 g/L; dye concentration: 10 mg/L).
The effect of waste catalyst loading on photocatalytic oxidation of RO16 dye

The results for photocatalytic oxidation of RO16 dye performed in the range of 0.5–1.0 g/L waste TiO$_2$ powder catalyst are given in Figure 6. In overall, the results showed the same behavior when increasing waste catalyst loading from 0.5 to 1.0 g/L, it is noticed a clear improvement of anionic dye removal efficiency where after 3 h of oxidation time, the RO16 removal efficiencies varied from 64.1% to 100%. For all experienced TiO$_2$ concentrations, there was a substantial increase of RO16 removal efficiency in the first 15 min. However, for 1.0 g/L of TiO$_2$ loading, there was a total dye degradation after 180 min (100% of RO16 removal efficiency). This means that the required photons, when the loading of TiO$_2$ raised up to 1.0 g/L, were thoroughly absorbed. This illustrates that an increase in the amount of catalyst to the level consistent with the optimized level of light absorption increases the efficiency of dye removal (initially 10 mg/L of RO16 dye). Therefore, any further increase of the catalyst loading have no effect on the photodegradation efficiency (Mahvi et al. 2009; Mezughi et al. 2014).

The effect of dye concentration on photocatalytic oxidation of RO16 dye

The effect of initial RO16 concentration on the dye removal is given in Figure 7 for different concentrations of RO16 dye solution while the catalyst concentration was kept constant (the optimum value defined in the previous section 1.0 g/L). Increasing of dye concentration from 10 to 50 mg/L engendered competing more dye molecules on same active sites on catalyst surface; subsequently, the generation of OH$^-$ radicals on the surface of catalyst was declined (Mahvi et al. 2009). Eventually, color removal efficiency decreased (Ozbey Unal et al. 2019). Other possible reason for such results is the impact of UV screening of the own dye. In addition, RO16 removal efficiency may decrease due to increased initial dye concentration and decreased UV light transmittance in the sample containing more intense color. This is explained that a major amount of UV is apt to be absorbed by dye molecules proportional to OH$^-$ particles and this reduces the efficiency of titanium dioxide catalytic reaction due to the decrease of OH$^-$ and OH$_2^+$ concentrations (Mahvi et al. 2009). One more possibility is the...
intervention of the by-products formed during the degradation or removal of RO16 dye molecules.

The effect of H$_2$O$_2$ concentration on photocatalytic oxidation of RO16 dye

Since there was 35.2% RO16 removal with waste TiO$_2$/UV powders at 50 mg/L RO16, H$_2$O$_2$ was added to the reactor and waste TiO$_2$/UV/H$_2$O$_2$ process was applied. The effect of H$_2$O$_2$ concentration on RO16 removal is given in Figure 8. As it is well known, H$_2$O$_2$ forms OH$^-$ radicals by photolysis reaction with UV irradiation and these OH$^-$ radicals increase photocatalytic oxidation (Konstantinou & Albanis 2004). While RO16 removal did not change with the addition of 1 mM H$_2$O$_2$, RO16 removal efficiency increased when H$_2$O$_2$ concentration increased. While 100% RO16 removal was achieved at 10 mM H$_2$O$_2$ concentration, it did not observe more dye removal when more H$_2$O$_2$ was added into the reaction medium. This is due to excessive H$_2$O$_2$ addition preventing OH$^-$ formation and sweeping effect (Daneshvar et al. 2007). In overall, the direct photolysis and the oxidative potential of H$_2$O$_2$ were proven to have slight contribution on the RO16 dye removal efficiency. Particularly, titanium dioxide (TiO$_2$) waste powder and UV light together indicated a marked effect (Tseng et al. 2015).

Reuse stability of waste TiO$_2$ powders

Reuse stability of waste TiO$_2$ powders catalyst was tested for RO16 dye decolorization (Figure 9). The results showed that no decrease in the RO16 dye removal efficiency was observed up to 5 cycles for waste TiO$_2$ powders catalyst. The process herein described is ecofriendly, cost-effective, and reusable for the treatment of contaminated water (Teixeira et al. 2016).

CONCLUSION

As a result of the studies, RO16 was removed by either photocatalytic TiO$_2$/UV or TiO$_2$/UV/H$_2$O$_2$ processes. TiO$_2$ can efficiently catalyze the removal of dye RO16 in the presence of light and oxygen. The RO16 dye is resistant to direct photolysis and there was no degradation for RO16 in the dark. The optimum conditions where RO16 was totally removed (100% removal efficiency) can be achieved with 1.0 g/L TiO$_2$ at 10 mg/L RO16 dye concentration in photocatalytic oxidation using waste TiO$_2$ powders. 100% RO16 removal dye could be achieved at high RO16 concentration (50 mg/L) at 1.0 g/L TiO$_2$ and 10 mM H$_2$O$_2$ with photocatalytic oxidation. Therefore, the use of waste TiO$_2$ powders in photocatalytic advanced oxidation process is thought to be effective both in terms of waste reduction and in reducing the cost of advanced oxidation process.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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