

Effect of Sonication Pre-treatment of TiO₂ Catalyst for Photo-Degradation of Acid Orange 7 Azo Dye

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Abstract

In this study, Acid Orange 7 azo dye was degraded on TiO₂ catalyst layer illuminated with ultra-violet light. The TiO₂ suspension had prior been sonicated at 20 kHz before electrophoretic deposition on smooth stainless steel surfaces, with the aim of increasing the efficiency of azo dyes degradation. The effect of sonication on the TiO₂ suspension of 10 g.L⁻¹ and electrophoretic deposition loading to layers on surfaces was studied. Morphological properties of the electrophoretic layers from two different suspensions, Alpha and Sigma TiO₂ were characterized by Scanning Electron Microscopy to establish the specific surface properties, particle loading and crystalline sizes. A four-position reactor was used for dye degradation experiments under Ultra Violet light at 355 nm wavelength. The degradation of the azo dye was monitored at 30minutes interval for a total of 2 hours using Ultra Violet-Visible Spectrophotometer at $\lambda = 485$ nm. It was found out that 60% of dye degradation was achieved after 120 minutes without sonication pretreatment. Sonication pre-treatment resulted in 71.42% increase on the rate of photo-degradation, at a loading of 0.32 mg/cm². Particulate layers coated with Alpha TiO₂ showed more photoactivity compared to Sigma TiO₂. Scanning Electron Microscopy indicated that Alpha TiO₂ had 50 m²/g surface area and 28 nm crystal size compared to Sigma TiO₂ with 10 m²/g and 169 nm crystal size. This study shows that sonication pretreatment of Alpha TiO₂ /UV light system is most effective in photo-degrading Acid Orange 7 dye.

Keywords: sonication, photo-degradation, TiO₂ suspension, acid orange 7 dye

1. Introduction

The Acid Orange 7, *p*-(2-hydroxy-1 naphthyl azo) benzene sulfonic acid (AO7) is a monoazo dye commonly used for high temperature dyeing of wool in strong acid bath in textiles and paper manufacturing among other industrial applications. The dye is stable at normal temperature as it has a melting point of 164 °C and a solubility of 116 g/L in water. The presence of AO7 in effluents from these industries causes obvious environmental problems. Sonication utilizes sound of frequency above 20 kHz beyond human being hearing, leading to agitation of particles in a colloidal suspension to make a homogenous solution (Suslick, 1988, 1998). In this work, 250 cm³ TiO₂ suspension of 10 g.L⁻¹ was placed into a water bath and soundwaves of 20 kHz were projected for 60 seconds. Sonication improves the dissolution of solids in suspensions there by promoting “degasification” or expulsion of gases present (Mason and Lorimer, 1980). Sonication allows gas bubbles to coalesce together and easily leave the colloid thereby increase the strength of agitation which, breaks up the colloidal particles with a net effect of increasing their surface area (Mason and Lorimer, 1980). Increased particle surface area directly influences the efficiency of TiO₂ catalytic degradation rate (Cernigoj *et al.*, 2006). Electrophoretic Deposition (EPD) applies an electric field between two electrodes in a colloidal suspension of material resulting in a cathodic deposition on the electrodes (Boccaccini and Zhitomirsky, 2002, Besra and Liu, 2007). The electric-field transports the suspended charged particles to the oppositely charged electrode thereby depositing a coated layer (Zhitomirsky, 2000; Stochet *et al.*, 2001). Advantages of this technique include: -good control of layer thickness, equipment’s simplicity and short formation time (Kanamura and Hamagami, 2004). The Electrophoretic deposition of TiO₂ has been carried out in suspensions of mainly two solvents, acetyl acetone and acetone (Doret *et al.*, 2009). The process involves a two-step procedure, leading to the deposition of uniform layers on suitable substrate materials (). The charged particles move in a suspension medium followed by deposition on stainless steel plates under an electric field caused by direct current voltage (Boccaccini and Zhitomirsky, 2002; Kanamura and Hamagami, 2004, Cabanas-Polo and Boccaccini, 2015). In addition to stainless steel, other materials such as films from nanoparticles and carbon nanotubes have found practical application (Corni *et al.*, 2008; Wang *et al.*, 2000). Several investigators, (Wang *et al.*,

2000; Shrestha *et al.*, 2001; Windes *et al.*, 2002; Knote *et al.*, 2007) have reported that thin films prepared from electrophoresis display several advantages compared to other techniques. These include, corrosion resistance, versatility, simplicity, low-cost and ability to immobilize material on 3D structures and porous substrates (Knote *et al.* 2007). It has been suggested that the hardness of the thin films is dependent on the initial colloidal suspension concentration and electrophoretic deposition time (Yousefipoura *et al.* 2013).

Titanium dioxide is first illuminated with light and subsequently initiates the photo-degradation process. However, only light of wavelengths below 400nm is absorbed by TiO₂ particles, e.g., 3.2eV approx. 388nm just as the organic pollutants such as dyestuffs absorbs Ultra-Violet light thereby allowing energy differential exchange between them. (Augugliaro *et al.*, 2002) Titanium dioxide as a semi-conductor photo-catalyst has found numerous applications such as in water purification, photo-voltammetry (Fernandez *et al.*, 1995, Fujishima *et al.*, 2008; Feng *et al.*, 2008). It has also found application in corrosion protection coatings, coatings in biomedicine, self-cleaning glazes and sensors (Boccaccini *et al.*, 2001; Mills *et al.*, 2003; Varghese *et al.*, 2003; Ma *et al.*, 2011; Cordero-Arias *et al.*, 2013; Wu *et al.*, 2014; Kulkarni *et al.*, 2015). Several methods to deposition TiO₂ layer on metallic surfaces have been investigated; these include plasma spraying, micro-arc oxidation (MAO), sol-gel, dip-coating and electrophoretic deposition (EPD) (Mohan *et al.*, 2012; Farnoush *et al.*, 2012). This paper reports the effect of sonication pre-treatment of TiO₂ for degradation of Acid Orange 7 dye and the analysis of the morphological properties of the deposited layers on stainless steel using Scanning Electron Microscope (SEM).

2. Materials and Methods

2.1 Chemicals and Reagents

2.1.1 Acid Orange 7 Dye (AO7)

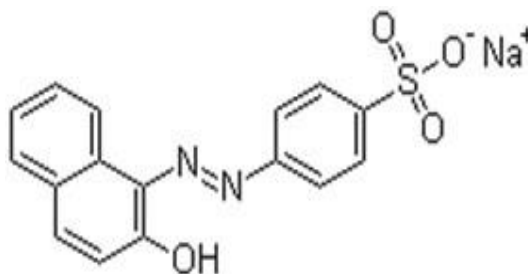


Figure 1. Structural formula of Acid Orange 7 Dye

Acid Orange 7 dye 95% purity was sourced in powder form. This was used to make a solution in water.

2.1.2 Alpha and Sigma Titanium Dioxide

Commercial Alpha TiO₂ and Sigma TiO₂ Anatase was used for the various suspensions in the methanol-water solutions for electrophoretic deposition.

2.2 Equipment and Apparatus

2.2.1 Ultra Sound Sonicator

An ultrasound Sonicator Model 150 V equipped with a probe was used on the various suspensions for pretreatment of TiO₂ suspensions. The Sonicator probe was immersed about 1 cm deep into the 250cm³ solution in a beaker that was placed in a water bath, and a sound of frequency 20kHz projected for 60 seconds.

2.2.2 Electrophoretic Deposition Cell

Stainless steel plates of 7.5 cm² and a thickness of 3 mm were used for deposition of layers from two different powders, Alpha and Sigma TiO₂. A solution of methanol-water (80:20) of 500 cm³ was used to make colloidal suspensions of TiO₂ at a concentration of 10g.L⁻¹. The suspensions were stirred and then sonicated at 20 kHz for 60 seconds. The stainless steel plates were initially polished to get a mirror finish before thoroughly being washed with distilled water, rinsed, ultrasonically degreased with acetone and dried for 30 minutes. The distance between the electrodes in the cell was maintained at 10 mm and deposition was carried at time intervals, 5, 10, 30 and 60 seconds at 4 V. The coated stainless steel plates were then removed, placed on ceramic tiles and dried for a period of 48 hrs in ambient air at room temperature to constant weights. The coated layers were thereafter characterized by Scanning Electron Microscope (SEM).

2.2.3 Photo-catalytic Reactor for the Azo Dye

A batch mode photo-catalytic reactor of capacity $2.5 \times 10^{-5} \text{ mol L}^{-1}$ was fabricated to evaluate the degradation of Acid Orange 7 dye. The photo-catalytic reactor was made of four-hole compartments, through which ultra violet light was irradiated using lamps of 18 W. The stainless steel plates coated with TiO_2 layers were suspended into the containers 25 cm^3 dye solutions maintained at constant stirring as shown in Figure 1 below.

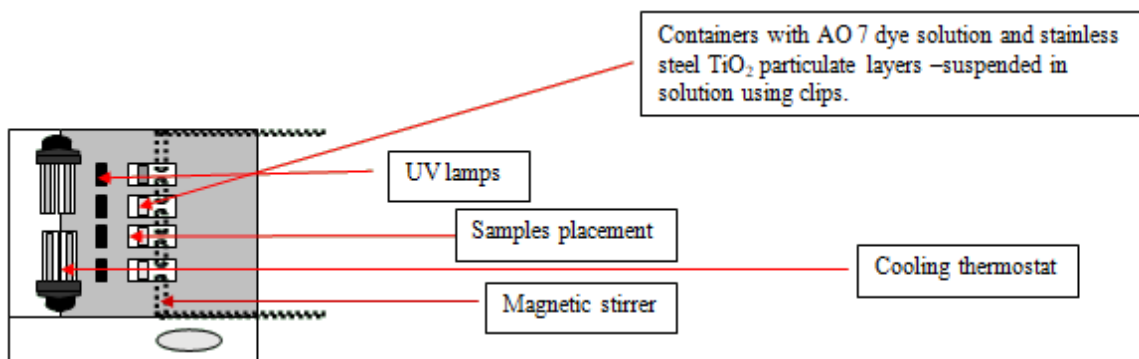


Figure 2. Four-Hole Photo-reactor for the Degradation of Acid Orange 7 Dye

2.3 Experimental– Ultra Violet TiO_2 Treatment System

A stock solution of Acid Orange 7 dye containing $1.0 \times 10^{-4} \text{ mol. L}^{-1}$ was made by dissolving 0.037g of the dye in 1000 cm^3 water. A volume of 25 cm^3 of the solution was put in each of the four containers placed on the four-hole apparatus and stirred at 1000 rpm so as to maintain homogeneity throughout the experiment. The solutions were then irradiated with Ultra Violet light of 355nm for a duration of 120 minutes. Evaluation of photo-degradation of the dye was carried out by drawing 5 cm^3 solution from each of the container after every 30 minutes and measuring the absorbance using UV/Vis spectrophotometer at $\lambda_{\text{max}} = 485 \text{ nm}$. The rate of photo-degradation of the solution was then calculated as follows;

$$J = \frac{V \, dc}{A \, dt} \tag{1}$$

J (AO7mol/ (min.cm²))

V (volume of dye solution in the containers)

A (area of stainless steel particulate layer

Containing deposited powder catalyst)

t (1hr.)

$$r_A = \frac{(c_0 - c_{60})V}{t_{60} - t_0} \cdot \frac{1}{A} \tag{2}$$

Where r_A is the rate of dye photo-degradation [(mol/ (min.cm²))]

c_0 ...initial dye concentration [mol.dm⁻³]

c_{60} ...dye concentration after 1 hour [mol.dm⁻³]

t time at which irradiation started [0 hrs]

t_{60} time of irradiation [1 hr]

V....volume of dye in containers (0.025 dm³)

A....irradiated geometric surface area of particulate film (0.00075 m²)

2.4 Molar Extinction Coefficient of Acid Orange 7 Dye

The maximum absorption wavelength (λ_{max}) for the dye solution was established by scanning from 200-800nm wavelength using the UV/Vis Spectrophotometer. The Acid Orange 7 dye’s molar extinction coefficient was determined

by preparing solutions at concentrations of; 1×10^{-5} , 2.5×10^{-5} , 5×10^{-5} , 7.5×10^{-5} and $1 \times 10^{-4} \text{ mol L}^{-1}$ and their absorbance measured at the established wavelength $\lambda=485 \text{ nm}$. A calibration curve was then plotted and the value determined.

3. Results and Discussions

3.1 TiO_2 Morphological Characterization by Scanning Electron Microscope

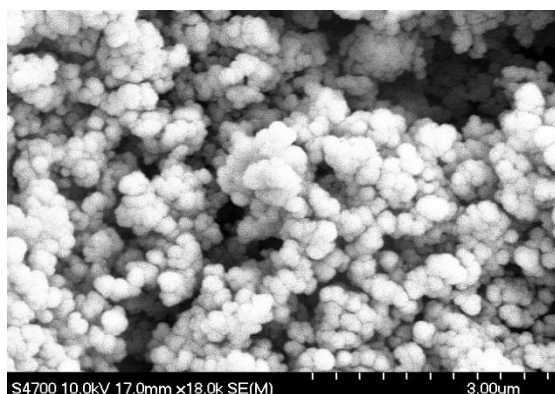
A morphological characterization of the TiO_2 powder catalysts is shown in Table 1 and Figure 3 below:

Table. 1 SEM Characterization of TiO_2 Powders

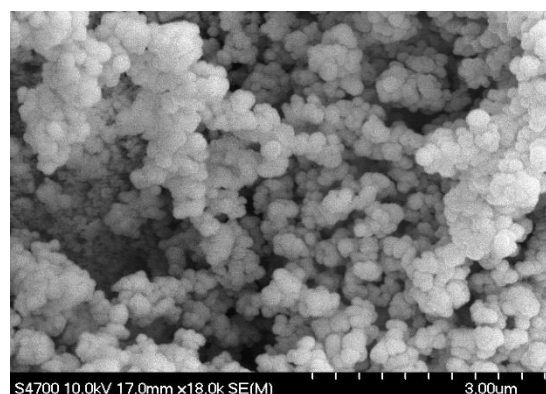
Powder	Surface area (m^2/g)	Particle size (nm)
Alpha TiO_2	50	28
Sigma TiO_2	10	169

In Table 1, the Alpha TiO_2 had a greater surface area of $50 \text{ m}^2/\text{g}$ compared to Sigma TiO_2 at $10 \text{ m}^2/\text{g}$. Sigma TiO_2 has bigger particles sizes of about 169 nm than Alpha TiO_2 's of 28 nm.

Sigma TiO_2 particles presented a surface area five (5) times lower than Alpha TiO_2 which is evident from its particle size that is six (6) times larger than Alpha TiO_2 .



Alpha TiO_2



Sigma TiO_2

Figure 3. SEM images of TiO_2 Particulate Layers Produced by Electrophoresis

Smaller Alpha TiO_2 particle sizes corresponds to greater surface area and in this case exhibits increased crystallinity that leads to an increase in photo-degradation rate.

3.2 Effect of Electrophoresis on the Catalyst Coating

The relationship between the electrophoretic time and amount of catalyst coating mass is shown in Figure 4 below.

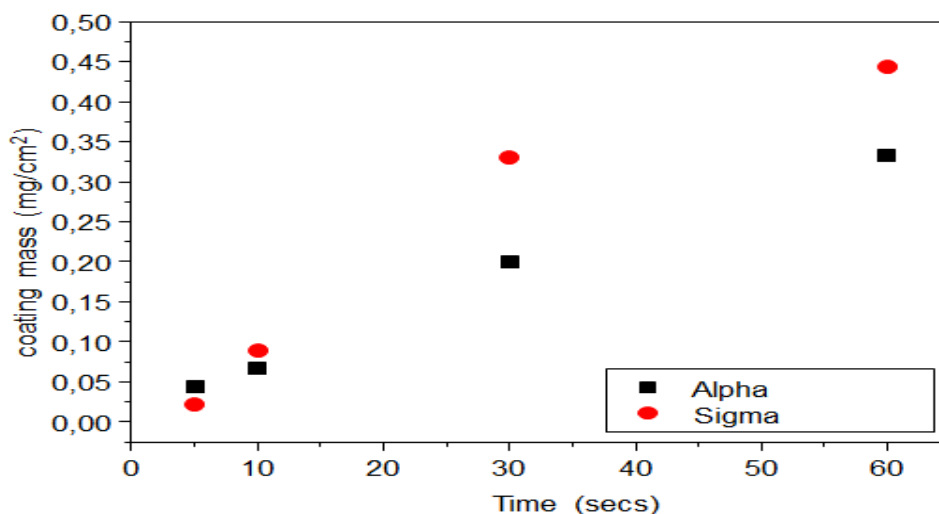


Figure 4. Electrophoresis Deposition of AlphaTiO₂ and Sigma TiO₂ per Second

After 5 seconds of Electrophoretic deposition experiments, 0.05 mg/cm² Alpha TiO₂ catalyst had been deposited compared to 0.25 mg/cm² Sigma TiO₂ while at 10 seconds of deposition time, more of SigmaTiO₂ catalyst 0.10mg/cm² had been deposited compared to Alpha TiO₂ 0.07mg/cm². After 30 seconds deposition time, the amount of Sigma TiO₂ catalyst deposited on the stainless steel coated layers was more by 0.15 mg/cm² compared to 60 seconds of deposition where the difference was 0.13mg/cm². The results obtained show that the powders have different independent rates of electrophoretic deposition under similar conditions. This is influenced by their different, concentrations, particle sizes, crystallinity, particle shape and conductivity on transport. Overall the amount of catalyst deposited in both experiments increases with electrophoretic deposition time.

3.3 Ultra Violet -Visible Spectral Analysis of Acid Orange 7 Maximum Absorption Wavelength (λ_{max})

A spectral scan between wavelength 200– 800 nm of the Acid Orange 7 dye solution carried across the UV-Visible spectrum is presented here below in Figure 5.

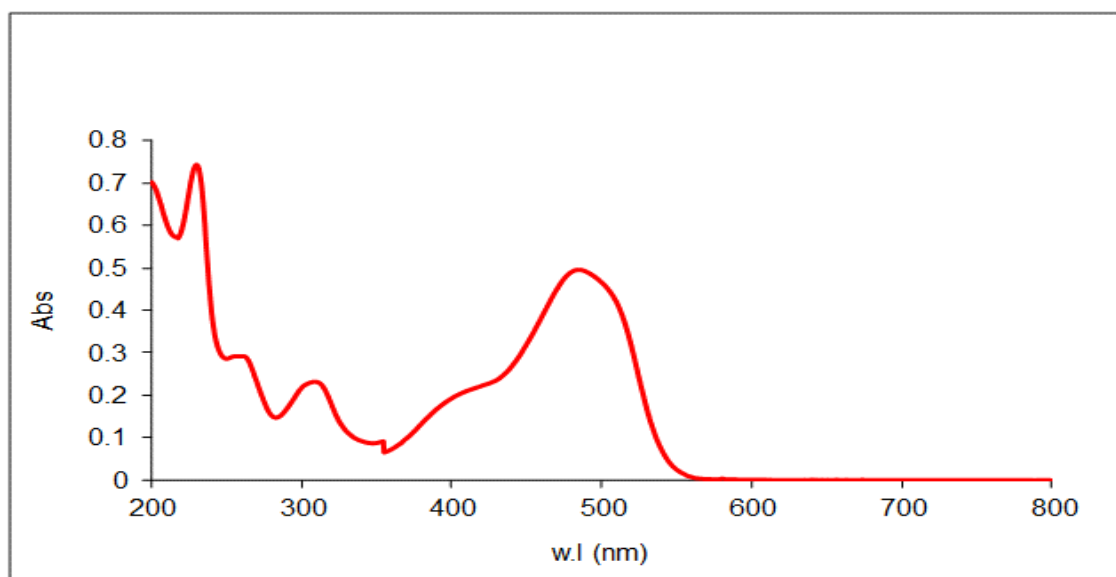


Figure 5. UV/Vis Spectrum before Photo-degradation Acid Orange 7

The scan showed a maximum absorption wavelength peak at 485 nm in the visible region.

This was similar to the value obtained by Tripathi and Srivastava, (2011). In this study, all photo-degradation experiments of the Acid Orange 7 dye solutions were measured at this wavelength $\lambda = 485$ nm.

3.3.1 Spectral Analysis Acid Orange 7 Molar Extinction Coefficient (ϵ)

The Molar Extinction Coefficient was determined through calibration curve plotted as shown in Figure 6 below.

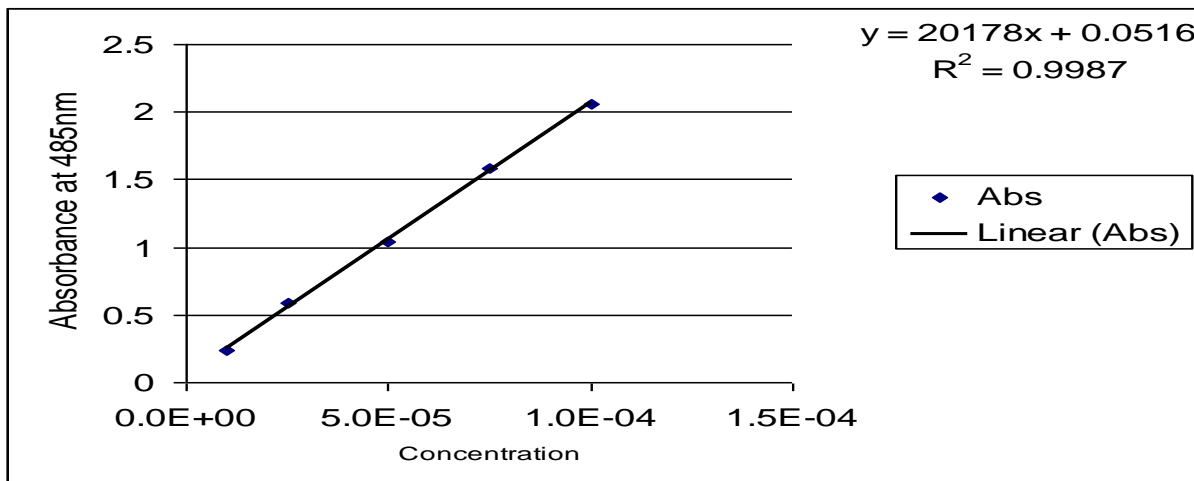


Figure 6. Calibration curve Acid Orange 7 dye

The calibration curve gave a regression of $r^2 = 0.9987$ that yielded a Molar Absorption Coefficient (ϵ) value of $2.02 \times 10^4 \text{ mol}^{-1} \text{ dm}^3 \text{ cm}^{-1}$. This is close to $2.1 \times 10^4 \text{ mol}^{-1} \text{ dm}^3 \text{ cm}^{-1}$ reported by Ferguson and Mau (1972).

3.4 Evaluation of TiO_2 Catalyst Photo-degradation of Acid Orange 7 Dye

Figure 7 below shows photo-degradation of $2.5 \times 10^{-5} \text{ mol. L}^{-1}$ Acid Orange 7 dye solution over a duration of 120 minutes without sonication pretreatment.

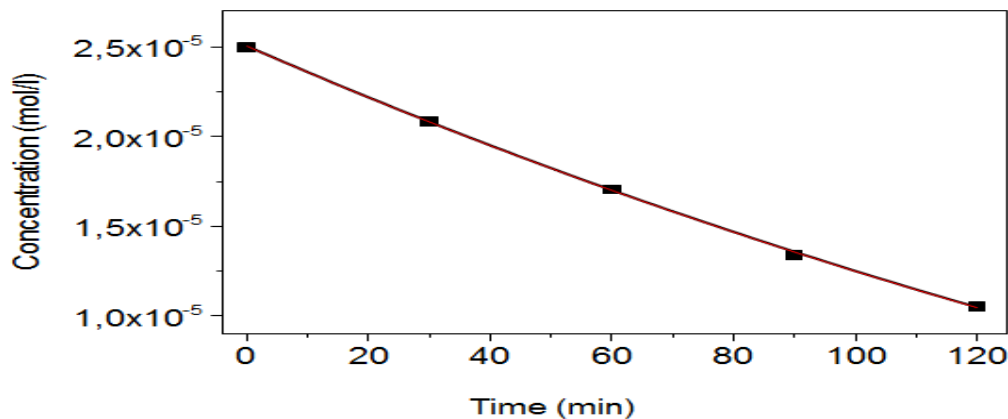


Figure 7. Photocatalytic Degradation Rate of Acid Orange 7 Dye Solution

The results show that both Alpha and Sigma TiO_2 achieved a 32% of photo-degradation in 30 minutes and 60% at 120 minutes.

3.5 Evaluation of Sonication Pretreatment of TiO_2 Catalyst on Photo-degradation of Acid Orange 7 Dye

Figure 8 below shows the evaluation of sonication pretreatment of TiO_2 suspension on photo-degradation of Acid Orange 7 dye.

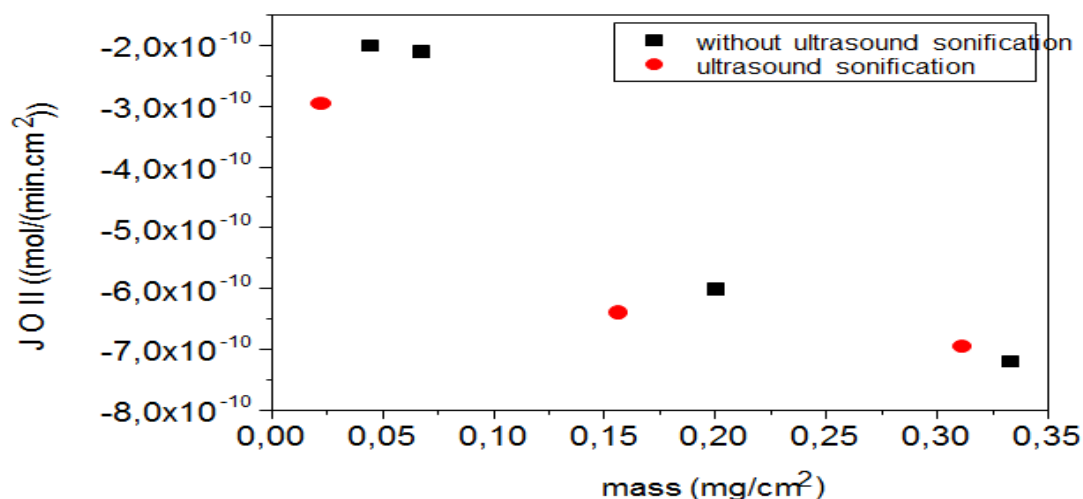


Figure 8. Effect of Sonication on the Rate of Acid Orange 7 Photocatalytic Degradation

Sonication resulted in a rate increase of Acid Orange 7 dye solution photo-degradation with 0.02 mg/cm² catalytic loading giving -3.0×10^{-10} mol/min/cm² compared to a -2.0×10^{-10} with a catalytic loading of 0.05 mg/cm² of non-sonicated suspension. An increase in catalyst loading from 0.02 to 0.32 mg/cm² led to a corresponding increase of 71.42% in dye photo-degradation.

3.6 Effect of TiO₂ Catalyst Loading on Acid Orange 7 Dye Photo-degradation

Figure 9 below shows the relationship between Alpha and Sigma TiO₂ catalyst loading and their rates of Acid Orange 7 dye photo-degradation.

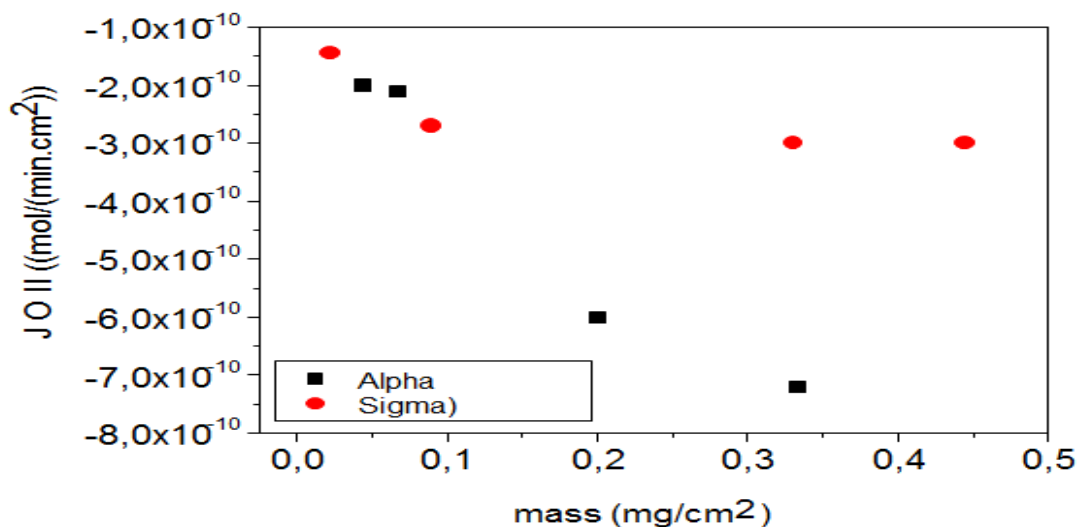


Figure 9. Effect of TiO₂ Catalyst type and Loading on Acid Orange 7 Photo-degradation Rates

At catalyst loading of 0.02-0.06 mg/cm² range gave a rate of dye photo-degradation for Alpha TiO₂ as -2.0×10^{-10} mol⁻¹.min⁻¹.cm² while that of Sigma TiO₂ as -3.0×10^{-10} mol⁻¹.min⁻¹.cm². This shows that at low catalyst loadings, the degradation rates of both Alpha TiO₂ and Sigma TiO₂ are similar. At catalyst loading of 0.2 mg/cm², there was a significant difference in photo-degradation between the Alpha TiO₂ at -6×10^{-10} mol⁻¹.min⁻¹.cm² compared to Sigma TiO₂ -3×10^{-10} mol⁻¹.min⁻¹.cm². After catalyst loading of 0.2 mg/cm², the rate of photo-degradation with Sigma TiO₂ remained constant at -3×10^{-10} mol⁻¹.min⁻¹.cm² despite further increase in catalyst loading while that due to Alpha TiO₂ reduced exponentially to -7.5×10^{-10} mol⁻¹.min⁻¹.cm² at a catalyst loading of 0.32 mg/cm².

4. Conclusion

The Acid Orange 7 dye degradation rate, using electrophoretically deposited TiO₂ photo-catalyst layer was significantly increased. The rate increase is due to the pre-treatment of TiO₂ suspension with ultrasonic sound at 20 kHz that allowed for homogenized smaller TiO₂ particles suspension and their electrophoretic deposition of uniform ultra thin layer coating on stainless steel plates. This optimizes the effective photo-degradation load to be 0.32 mg/cm² Alpha TiO₂ at a degradation rate of 71.42% compared to 60% of unsonicated photo-catalyst layer in 2 hrs. The sonicated layer has a smaller particle size, hence higher surface area that is effective to degrade the azo dye at a higher rate. Sonication pre-treatment of TiO₂ photo-catalytic suspension and its electrophoretic deposition increases the rate of photo-degradation of Acid Orange 7 dye.

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