SiO$_2$ based TiO$_2$ and Al$_2$O$_3$ nano composites for assessment of water quality of textile dyeing industrial effluent of Tirupur (Tamilnadu) India

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Abstract: *In this investigation, we have compared the water quality parameters of dyeing effluent discharged from the dyeing industries in Tirupur district of Tamilnadu, India with the some of the physiochemical data analyzed from these results. The present work represents a novel method for treatment and decolorization of dye industrial wastewater using both SiO$_2$/TiO$_2$ and SiO$_2$/Al$_2$O$_3$nanocompositesunder sunlight radiation.These effluents, with their high biological oxygen demand (BOD), chemical oxygen demand (COD) and suspended solids are very toxic in nature. The study has been analyzed for the water characterizing parameters under various catalysts load(0.1 to 1.0g) for the raw effluent water under optimum conditions for both the composites and reports SiO$_2$/TiO$_2$ nanocomposite as a better catalyst lowering the effluent characteristics thanSiO$_2$/Al$_2$O$_3$.*
Keywords:*Nanocomposite,Textile effluent, Photodegradation, decolorization, BOD, COD*

I. Introduction

Water pollution by toxic organic compounds is a topic of Worldwide concern[1].Wastewater is intensely dyed, polluted with highconcentration of organic materials such as; suspended and dissolved salts in addition to other recalcitrant compounds. In textile and dyestuff industries all the dyes are released instantly as crude dye in the wastewater and their ejection into water streams is very dangerous to environmental health[2]. Numerous studies have been carried out across the globe focusing on the decolorization of textile wastewater.However, most of these methods are difficult in practical uses[3].The importance of these types of research is being increasing in the recent years and it has become a subject of major public health concern and scientific interest. It is considered that the removal of colour from wastewater is more important than the removal of other organic colorless chemicals[4].Decolorization of effluent from textile dyeing industry is regarded important because of aesthetic and environmental concerns[5].A major encouraging technique used now-a-days among many oxidation processes isheterogeneous photocatalyst. It may be used effectively for organic pollutants degradation in water, such as textiles[6],cosmetics and pharmaceutical wastewater [7],paper mill[8], grey water[9], and municipal wastewater[10].Recent studies have focused on the most important photocatalytic applications oftitanium dioxide and silica and different systems have been used, such as, ozonating[11], H$_2$O$_2$/UV [12], photo catalysis [13], photo-Fenton[14], electrocoagulation[15],sonolysis[16],gamma–radiolysis[17],biological [18], and combined anaerobic–photo catalytic treatment[19].TiO$_2$ as aphotocatalyst has recently attracted great interest using solar radiation as a light source for water and wastewater treatment[20],photodegradationof variouspollutants[21], killing bacteria[22] and cancer treatments[23].The aim of the present study is to investigate the photocatalytic decolorization of textile wastewater using SiO$_2$ basedTiO$_2$/Al$_2$O$_3$nanocompositeunderphotocatalytic irradiationwith sunlight and reports of their effect on the water characterizingprofile of the treated effluent.

II. Materials and Methods

2.1 Sample collection

Tirupur, the leading cotton knitwear industrial cluster in Tamilnadu, South India was chosen for effluent sample collection.The effluent samples were collected from different points of the area.Standardprocedures (spot and Grap) were followed during sampling.The effluent was collected from the textile dyeing units of Tirupur, Tamilnadu, India. The five samples were collected from different industries, mixed andwere used for analysis.The pH and temperature were determinedinstantly at the sampling site using digital pH meter and laboratory thermometer.The sample was transported to laboratory at 4°C as in accordance with the standard methods [24].

2.2 Analysis of effluent

The physicochemical parameters (colour, electrical conductivity (EC),BOD,COD,total suspended solids [(TSS),total dissolved solids (TDS)]were determined as soon as the samples was brought to the laboratory.Sample colour was analyzed by Spectrophotometer(Genesys10uv, ThermoElectronCorporation) while electrical conductance was determined by Digital conductivity meter (RI503),BOD was determined by employing evaporation method byDO meter andCOD was measured by COD instrument directly. chloride and sulphate contents were assessed by titrimetric and turbiditymethod [25].

2.2.1 Determination of Biological Oxygen Demand (BOD)

The BOD of collected samples was analyzed using standard procedure. Two bottles were filled with the sample. At first, the dissolved oxygen (DO) for one BOD bottle was calculated and the remaining bottles were kept in dark condition for five days. After five days, the DO for the dark bottles was calculated. Then the BOD level of the sample was determined from the difference between the initial and final DO.
The BOD was calculated using the following formula:
$$DO \ (mg/l) = 8 \times C \times V_B - V_A / V_S \times 1000 \quad (1)$$
Where, C is normality of sodium thiosulphate; V_A is volume of initial titration amount of sodium thiosulphate; V_B is volume of final titration amount of sodium thiosulphate; V_S is volume of sample
$$BOD \ (mg/l) = Initial \ D.O – Final \ D.O.$$

2.2.2 Determination of Chemical Oxygen Demand (COD)

COD refers to the oxygen consumed by the oxidisable organic substances. The chemical oxidant such as $K_2Cr_2O_7$ or $KMnO_4$ is used to measure the oxidisability of the organic matter of water where the oxidants oxidize constituents. Then KI is added. The excess amount of oxygen reacts with KI and liberates iodine. The excess amount of oxygen is equal to the amount of oxygen present. To determine COD of the effluent sample, 50ml of water was poured in a 100ml conical flask. Similarly, 50ml distilled water was taken in a flask as control. 5ml $K_2Cr_2O_7$ solution was poured separately in both the flasks. The flasks were incubated at $100^{\circ}C$ for 1h keeping in a water bath. There after the flasks were removed to cool for 10 minutes. 5ml KI solution and 10ml of H_2SO_4 solution were mixed in each flask. 0.1M sodium thiosulphate solution was transferred in burette fitted in titration assembly and titrated with both thesample in flasks till pale yellow color disappeared. In each case the amount of sodium thiosulphate wasnoted. 1ml of starch solution was added to both the flasks. The colour turned blue. Again, these were titratedwith sodium thiosulphate till the complete disappearance of blue color. The volume of sodium thiosulphate usedwas noted for the both samples.
The COD was calculated using the following formula,
$$COD \ (mg/l) = 8 \times C \times V_B - V_A / V_S \times 100 \quad\quad (2)$$
Where, C is the concentration of titrant; V_B is volume of titrant (ml) used for water samples; V_A is volume of titrant (ml) used for control; V_S is volume of water sample taken.

2.2.3 Determination of TS, TDS and TSS

250ml of effluent sample was filtered through Whatmannno.44 filter paper. Then the filtrate was dried in a beaker at110°C. After cooling, the residue was weighted and TDS in mg/l was calculated. For TSS, 250 ml effluent sample was taken in a beaker and evaporated to dryness on a hot plate. After cooling, the residue was weighted and TSS in mg/l was calculated.

$$TS \ (in \ mg/l) = Wt. \ of \ residue \times 1000000 \ / \ Amount \ of \ sample \ (ml) \quad\quad (3)$$

$$TDS \ (in \ mg/l) == Wt. \ of \ residue \times 1000000 \ / \ Amount \ of \ sample \ (ml) \quad\quad (4)$$

$$TSS \ (in \ mg/l) = (3)-(4)$$

2.3 Preparation of nano composites
2.3.1Chemicals

Silicic acid,TTIP (Titanium tetra isopropoxide) as a precursor,hydrochloric acid as peptizing agent and ethanol was used as a solvent medium.Tetrahydrofuran (THF) and aluminum chloride have been used in this study. All otherreagents used were of analyticalgrade puritypurchased from Merck, India.

2.3.2 Preparation ofSiO₂-TiO₂and SiO₂-Al₂O₃Nanocomposites

SiO₂-TiO₂ nanocomposite was synthesized using the following procedure.Sol-geltechnique was successfully adopted for synthesis of TiO₂ nanoparticles. SiO₂ sol was prepared by mixing 6 gm of silicic acid with 40ml of THF in the ratio of 1:2. SiO₂ sol was added drop-wise to the TiO₂ gel, which resulted in yellowish

brown solution. This mixture was stirred for 3 h at room temperature then raised to 80℃ and stirring was continued for an hour. Finally, yellowish brown color changed to yellow and the solution was dried at room temperature. Yellow powder was obtained and it was heated in microwave oven at about 100℃ for one hour. Finally, the composite was calcined at 400 °C [26].TTIP (Titanium tetra isopropoxide)was used as a precursor;HCl was mixed with ethanol and was stirred for ten minutes. To this mixture TTIPwas added in the ratio of 1:4:2 and the stirring were continued for 1 h at room temperature. Then 50 ml of deionized water was added, the temperaturewasraisedto50℃ and stirred for 3 hrs until the solution changed into colorlessgel.
 SiO₂ sol was mixed drop-wise to aluminiumchloride withHCl and deionized water wasadded and the procedure was repeated as above

2.4 Photocatalyticactivity evaluation of SiO₂/TiO₂ and SiO₂/Al₂O₃
2.4.1 Effect of decolorization with time
Thesynthesizednanocompositeswere studiedonthedecolorization of the textile effluent and water quality parameters under sunlight irradiation.100ml of the solution were placed in 250ml beaker witha photocatalystload of 0.8g.The solution was magnetically stirred to ensure homogeneous mixing before irradiation.The samples were collected every 30min upto240min,centrifugedat12,000rpm for 15min andanalyzedusing UV–Visspectrophotometer at 654nm. Decolorization efficiency (%) was calculated by the formula (5) and all experiments were done in triplicate.

% decolorization = ((Initial Absorbance – Final Absorbance)/Initial Absorbance) X 100   (5)

2.4.2 Effect of catalyst load
To explain the efficiency of SiO₂ based TiO₂/Al₂O₃ photocatalyst decolorization experiments were set up with varying the dose from 0.1 to 1.0g/100mlfor raw effluent. Thesolution was kept under sunlight illumination for a period of 240min. The solution was then centrifuged at 12,000 rpm for 10min and the supernatant was used for water quality analysis [27].

III.   Results and Discussion
The study reports that for all the water quality parameters studied for raw effluent, the values were found to be higher than that of SiO₂/Al₂O₃compare with SiO₂/TiO₂.The analyzed data revealed the variation between the test samples, the resultanalysis of variousparameters of effluent are summarized in Table 1 and2.

3.1 Decolorization studies
In the present investigation, the colour of the untreated effluent was dark blue. Colour is a very important factor for the aquatic life for making food from sun-rays. The photosynthetic activity reduced due to dark coloration affecting other parameters like temperature, DO and BOD. Effect of SiO₂ basedTiO₂/Al₂O₃ has been assessed for a photocatalyst load of 0.8g for both the composites with an illuminationperiod ranging from 30-240min. The study showed that with increase in illumination time, percent colour removal increased and attained equilibrium at 150min.This suggests that decomposition increased with illumination time and at constant catalyst load. The hydroxyl radical formed on the surface of TiO₂ and SiO₂ is also constant [28]. Hence the higher concentration, decomposition decreases at sufficiently longer distances from light source or reaction zone due to the retardation of penetration of light.

3.2. Effect of catalyst load
In order to explain the effect of SiO₂ based TiO₂/Al₂O₃ photocatalyst degradation experiments were setup with varying the dose from 0.1 to 1.0mg/100ml.The percentage removal increased from 30 to 78% and reached equilibrium at 150 min. Maximum dye was sequestered from the solution within 150 min.Being a member of metal-oxide semiconductor photocatalysis family, there is a general agreement among researchers that TiO₂ is more superior because of its high photocatalytic activity, large chemical stability and robustness against photocorrosion, low price, and non-toxicity. Decolorization efficiency increased with increase in catalyst load and got saturated at a certain value of loading.The rate of reaction increased with increasing the amount of SiO₂/TiO₂ and SiO₂/Al₂O₃ nanocomposites.

3.3 BOD, COD and TDS
Textile industries use organic substances as raw materials and high levels of dissolved organic matter consume large amounts of oxygen and increase BOD level, which undergoes anaerobic fermentation processes leading to formation of ammonia and organic acids. The COD levels obtained from the industries shows that detergents, softeners and impurities on the fabrics contributes a significant portion of the COD.Textile industries

shows higher TDS value than the other industries mainly due to the fixing, bleaching, dyeing agents, etc., used during the processing of the fabrics on different stages.

In this study, Table 1 shows that in the raw effluent the BOD, COD and total dissolved solids were 240,628 and 5357mg/l respectively, while the treated water showed a BOD, COD and TDS of 115mg/l, 295mg/l and 2175mg/l respectively for SiO_2/TiO_2 nanocompositeand 145 mg/l, 352mg/l and 2360 mg/l for SiO_2/Al_2O_3nano composite which is slightly higher. The table reveals SiO_2/TiO_2 a better photocatalyst than SiO_2/Al_2O_3.

3.4 pHand Electrical conductivity

pH value of the effluent was found to significantlyvary and depends upon the dyes (acidic, basic and reactive dyes) and the materials (cotton, synthetic, etc.) of dying. For example, high pH denatures the protein fibers (such as wool and silks) and acid dyes (such as azo dyes,triaryl methane and anthraquinone). These dyes are applied to the fabrics along with acid solutions. Electrical conductivity is a measure of the ability of water to pass on electrical current and is affected by the presence of dissolved solids. As the level of total dissolved solids (TDS) raises, the conductivity will also increase[29]. The pH and electrical conductivity of the water samplesunder study was foundto be8.9 and 3.5mS cm^{-1}in the raw effluent and in the treated water it ranged from 7.3-7.8 for pH and3.5-3.8mS cm^{-1}for conductivity. ForpH, values werewithin the desirable range for both the composites.

3.5 Magnesium,Calcium and Chloride

Divalent metallic cations particularly Ca^{+2}, Mg^{+2} are responsible for hardness in textile effluents.Chloride in wastewater comes mostly from raw water taken for dyeing and it may add as a fixing agent for some of the dyes. The raw water showed the concentration of magnesium, calcium and chloride to be 8, 98 and 2803mg/l respectively and the treated effluent showed a range of 6, 82-92 and 1055 -2588 mg/l for SiO_2/TiO_2 composite and a range of 7, 85-97 and 1175-2795 mg/lfor SiO_2/Al_2O_3composite.

IV.   Conclusion

The study reports the water characteristics of the collected effluent before and after treatment with the nanocomposites. It reveals that SiO_2/TiO_2 composite is suitable to the dye water problems thereby minimizing the impurities present in it. It also reports SiO_2/TiO_2 as a better photocatalyst in colour treatment compared to SiO_2/Al_2O_3 and may become an effective alternative solution for the textile effluent problem.

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Table 1 Effect of catalyst load on water characterizing profile of textile dyeing effluent

S No	Parameters	Raw effluent	Catalyst load (mg/100ml)									
			SiO₂/TiO₂ nano composite treated effluent					SiO₂/Al₂O₃ nano composite treated effluent				
			0.2	0.4	0.6	0.8	1.0	0.2	0.4	0.6	0.8	1.0
1	Appearance	Blue colour liquid	Blue colour liquid	Blue Colour liquid	Colorless liquid	Colorless liquid	Colorless liquid	Colorless liquid	Colorless liquid	Colorless liquid	Colorless liquid	Colorless liquid
2	Odour	Pungent	Pungent	Pungent	Light Pungent	Light Pungent	Light Pungent	Pungent	Pungent	Light Pungent	Light Pungent	Light Pungent
3	pH	8.9	7.8	7.5	7.5	7.3	7.2	7.8	7.8	7.8	7.8	7.8
4	% removal	-	32	57	78	100	100	20	55	75	100	100
5	BOD(mg/l)	240	230	188	165	138	115	233	205	185	168	145
6	COD(mg/l)	628	602	555	485	378	295	610	595	515	454	352
7	TDS(mg/l)	5357	4340	3805	3287	2285	2175	4845	4118	3598	2880	2360
8	TSS(mg/l)	148	135	130	128	125	108	132	130	128	130	140
9	TH as CaCO₃(mg/l)	320	312	300	278	260	235	315	305	270	260	240
10	Permanent Hardness as CaCO₃(mg/l)	53	53	49	44	43	43	53	53	50	48	48
11	Temporary Hardness as CaCO₃(mg/l)	177	170	163	160	158	155	172	172	170	168	168
12	Magnesium Hardness as CaCO₃(mg/l)	45	35	34	34	33	35	45	42	40	38	38
13	Calcium Hardness as CaCO₃(mg/l)	215	206	200	198	196	195	215	211	210	208	208
14	Total Alkalinity as CaCO₃ (mg/l)	905	895	888	880	876	875	901	899	897	897	897
15	Calcium as Ca⁺(mg/l)	98	92	88	85	84	82	97	96	90	86	85
16	Magnesium as Mg+(mg/l)	8	6	6	6	6	6	7	7	7	7	7
17	Chlorides as Cl⁻(mg/l)	2803	2588	2102	1674	1218	1055	2795	2354	1938	1728	1175
18	Electrical Conductance (mS)	3.5	3.6	3.6	3.6	3.7	3.5	3.7	3.7	3.7	3.8	3.8
19	Sulphate as SO₄²⁻	1450	1410	1360	1322	1195	1070	1425	1396	1352	1335	1316

Table 2 Effect of irradiation time on the water characterizing profile of dye effluent

S No	Parameter	Raw effluent	SiO$_2$/TiO$_2$ nanocomposite treated effluent (0.8g) Irradiation time (min)								SiO$_2$/Al$_2$O$_3$ nanocomposite treated effluent (0.8g) Irradiation time (min)							
			30	60	90	120	150	180	210	240	30	60	90	120	150	180	210	240
1	pH	8.9	7.5	7.5	7.5	7.5	7.5	7.4	7.4	7.4	7.6	7.6	7.6	7.6	7.6	7.6	7.6	7.6
2	% Removal	-	42	60	78	95	100	100	100	100	35	55	72	85	91	96	100	100
3	BOD (mg/l)	240	222	212	205	175	158	123	115	90	232	222	215	205	185	162	140	125
4	COD (mg/l)	628	608	572	540	573	450	393	332	232	622	602	580	565	548	435	355	265
5	TDS (mg/l)	357	335	322	308	297	287	275	262	202	348	335	325	312	297	295	290	219
6	TSS (mg/l)	148	140	135	128	122	118	115	102	88	142	135	129	125	119	117	115	115
7	Magnesium Hardness as CaCO$_3$ (mg/l)	45	43	41	38	34	30	27	24	22	43	41	38	36	32	28	27	26
8	Calcium Hardness as CaCO$_3$ (mg/l)	215	195	185	170	150	125	108	100	95	202	195	187	155	135	128	120	105
9	Chlorides as Cl$^-$ (mg/l)	2803	2575	2150	1828	1545	1320	1115	2255	968	2780	2748	2380	2170	1810	1460	1205	1188