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# Solar Photocatalytic Degradation of Rhodamine-B Dye Using Lettuce Extracted $TiO_2$ Nanoparticle

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**ABSTRACT**

The present study describes the green synthesis of $TiO_2$ nanoparticles using *Sesbania grandiflora* and *Solanum nigrum* leaf extracts. The characterisation of synthesised nanoparticles is carried out using XRD and FTIR. The photo catalytic degradation of Rhodamine-B dye is carry out under sunlight irradiation by using UV-Vis spectrophotometer. Among the synthesized catalysts, $SG/TiO_2$ was found to be the best for the photocatalytic degradation of dye. The degraded dye water is also tested for the water quality parameters and the results are discussed.

**Keywords:** Photocatalytic degradation, Advanced oxidation process, Rhodamine B,

## INTRODUCTION

Water being the base of existence of life on earth, without which our land would be barren. This life saving liquid is also a universal solvent that also makes it easier to get polluted[1,2]. As surface water resources such as ponds and revers are the main target to get polluted by effluents and it turns to be a headache for chemists to find a suitable solution. As these water resources get polluted could barren the land, leading to infertility, demolish the aquatic life and may even cause death to humans when come across directly or indirectly. This polluted water then goes to water bodies, which is harmful to the life kingdom of plants as well as animals[3]. Dyes are chemical compounds and exhibiting colour with high molecular weights. It is chemically bonded to the substrate. Most of the natural dyes derived from animals, minerals, plant sources such as roots, barks, berries, woods, leaves, fungi and lichens. Synthetic dyes are derived from organic or inorganic compounds. Synthetic dyes are offered a wide range of colours and are man-made from petrochemicals. Pigments are a coloured substance which is not chemically bound to the materials. Usually, dyes are soluble in water but pigments are insoluble. In modern technical world, dyes are broadly used in the textile industry, food industry, photochemical cells, hair colouring, and leather tanning industry, paper production and cosmetics[4].

There are many techniques for the removal of industrial dyes from water or any other sources. It is cost-effective, inefficient and inflexible which

again to be treated. Processes of advanced oxidation (AOP), was a set of chemical treatment to remove organic and inorganic materials in wastewater by oxidation reaction with hydroxyl group (.OH). It is formed by the combination of free radicals. Micro-pollutant was treated with the help of UV radiation with ozone gas, Drinking water containing organic pollutants where treated with systems[5]. The Photo catalysis process was environmentally beneficial approach it's carried out with light, in which light energy converted to chemical energy. The development of novel treatment methods for converting organic pollutants (dye effluents) to non pollutant materials. $TiO_2$ is a naturally occurring mineral. It acts as a semiconductor, highly stable and non–toxic. The other name of titanium dioxide is titanium (IV) oxide, Titanium white, pigment white or titania. Illuminate, rutile and anatase are the different phases of titanium[6]. Green synthesis of $TiO_2$ catalyst prepared by using the plant of *Solanum nigrum* and *Sesbania grandiflora* leafs extract which is used as reducing agent to convert the precursor of Titanium Isopropoxide into nanoparticles. *Solanum nigrum* is commonly known as blackberry nightshade or Manathakkali in Tamil. *Solanum nigrum* naturally occurs in Africa and is used as food as well as a medicinal plant. It acts as capping agent. It belongs to the family of Solanaceae[7]. *Sesbania grandiflora* is a *Agati keerai* in Tamil and humming bird tree in English. It is omnipotent spinach and is capable of curing the psychological problems of human beings. *Sesbania grandiflora* leaves with β–carotene undergo changes upon baking, thus influencing the color characteristics of bread. The brightness and yellowness of the bread may be decreased due to the enzymatic browning[8-12].

Rhodamine-B is a fully synthetic dye. It is a thiazine dye. It was first prepared by German chemist Heinrich Caro in 1876. The INN (International Non-proprietary Name) of Rhodamine-B is otherwise called methylthioninium chloride, is a medication and dye. It is a dark green colour powder but dissolved in water it appear in blue color. Methylthioninium chloride mainly used to treat methemoglobinemia. In malaria treatment Rhodamine-B used is found by Paul Guttmann and Paul Ehrlich in 1891. Rhodamine-B used in textile industries and leather industries[13]. In our present work, we have synthesized $TiO_2$ nanoparticles by using Sesbania grandiflora, *Solanum nigrum* as a natural precursors for the degradation of Rhodamine-B. The characterization of catalyst was carried out using FT-IR and XRD. The photocatalytic degradation of Rhodamine B was carried out under sunlight irradiation[14].

## MATERIALS AND METHODS

### Chemicals and reagents

Leaves of the plant *Solanum nigrum*, *Sesbania grandiflora* are collected from Dindigul District. Ethanol and Double distilled water is used in the Synthesis process. In this study, Rhodamine-B dye is used as a model pollutant. Titanium isopropoxide is used as a precursor.

### Preparation of plant extract

The fresh Leaves of *Solanum nigrum*, *Sesbania grandiflora* were washed with tap water and distilled water to remove the dust particles on their surface separately. The collected leaves were shade dried at room temperature for one week in a normal atmosphere. The leaves were cut into pieces, grained to get the fine powder. 100 mL of ethanol added to the 10 g of leaf powder then mixed well and heated for one hour at 50°C. The ethanoic leaf extract was obtained by filtering using Whatman No.1 filter paper. The ethonoic leaf extract was used for the biosynthesis of $TiO_2$ Nanoparticles.

### Green synthesis of $TiO_2$ nanoparticles

25 mL of Titanium isopropoxide solution was prepared for the synthesis of $TiO_2$ nanoparticles. 25 mL of Titanium isopropoxide solution was added to the 100 mL of ethonoic leaf extract it reacted under stirring at 50°C. After four hours of continuous stirring, titanium dioxide nanoparticles was formed. The Nanoparticles was filtered with Whatman No:1 filter paper and the crude washed with ethanol solution. The separated $TiO_2$ nanoparticles were dried and ground using a mortar then calcined at 500°C in a muffle furnace for about five hours. The as synthesized $TiO_2$ nanoparticles were used for further studies[15].

### Degradation of dye by photo catalytic action

2 mL of Rhodamine-B dye is added as a component in 100 mL of pure distilled water, 100 mg of biodegradable synthesized $TiO_2$ nanoparticles are mixed with 100 mL of Rhodamine dye. A control is continued without the addition of $TiO_2$. To make the working solution of equilibrium, we need to stir for

30 min in order to receive the reaction suspension by mixing. The above must be done before exposing to irradiation. Afterwards, it should be kept under sunlight from morning till evening monitored. At intervals of a few hours, the aliquots of 2 mL of suspension should be filtered and monitored. It is used to evaluate the photosynthetic decay of the dye[16].

The absorption of the dye was recorded by using UV–Visible spectrophotometer. From the UV–Visible absorption spectra, the $\lambda_{max}$ of Rhodamine B dye was found to be at 554nm.

%degradation of Rhodamine B dye = (I.C – F.C)/I.C x 100 (1)

Where,
*I.C*- Initial Absorbance of Rhodamine B (Blank)
*F.C*- Sample Absorbance

## RESULTS AND DISCUSSION

### Fourier transform infrared spectroscopy (FTIR)

FTIR was used to identify the chemical properties of the green synthesized titanium dioxide nanoparticles of Solanum nigrum and Sesbania grandiflora leaves extract. The functional group of the green synthesised Titanium dioxide nanoparticles was identified by using FTIR.

Figure 1 shows the FTIR of sample SG/$TiO_2$. The strong peak is appeared 669 $cm^{-1}$ due to crystal lattice vibrations of Ti-O-Ti band, this showed the anatase morphology of $TiO_2$ nanoparticles. 1271 $cm^{-1}$ is due to C-O-C stretching, 1384 $cm^{-1}$ is due to C-O stretching, 1630 $cm^{-1}$ is due to C=O stretching vibration and 3416 $cm^{-1}$ OH stretching vibrations.

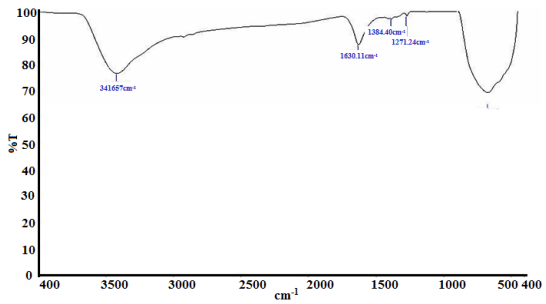


**Fig. 1. FTIR spectra of sample SG/$TiO_2$**

Figure 2 shows the FTIR of SN/$TiO_2$ nanoparticles. The strong peak is obtained around 680 $cm^{-1}$. 1628 $cm^{-1}$ is for C=O stretching, 2121 $cm^{-1}$ for O=C=O stretching. 3411 $cm^{-1}$ to 3768 $cm^{-1}$ is showed the OH stretching vibration[17-19].

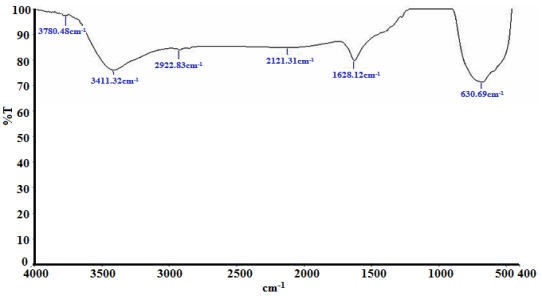


**Fig. 2. FTIR spectra of sample SN/$TiO_2$**

### X-ray diffraction analysis

The 2θ peaks at 25.30° and 48.01° confirm its anatase structure. The intensity of XRD peaks of the sample reflects that the formed nanoparticles are crystalline and broad diffraction peaks indicate very small size crystallite. JCPDS CARD NO. 21-1272. Both SG/$TiO_2$ SN/$TiO_2$ gives almost same values in d-spacing and 2θ values.

**Table 1: XRD peak list of $TiO_2$**

| 2θ | θ | FWHM | Size | d-Spacing |
|---|---|---|---|---|
| 25.270 | 12.635 | 0.109 | 75 | 3.52149 |
| 36.910 | 18.455 | 0.108 | 78 | 2.43337 |
| 37.771 | 18.885 | 0.122 | 69 | 2.37986 |
| 38.528 | 19.264 | 0.128 | 66 | 2.33482 |
| 48.010 | 24.005 | 0.107 | 81 | 1.89350 |
| 53.849 | 26.924 | 0.122 | 73 | 1.70114 |
| 55.037 | 27.518 | 0.114 | 79 | 1.66720 |
| 62.073 | 31.036 | 0.137 | 68 | 1.49402 |
| 62.649 | 31.324 | 0.130 | 72 | 1.48107 |

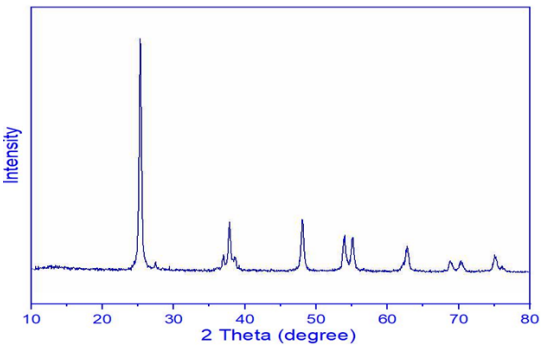


**Fig. 3. XRD Pattern of SG/$TiO_2$**

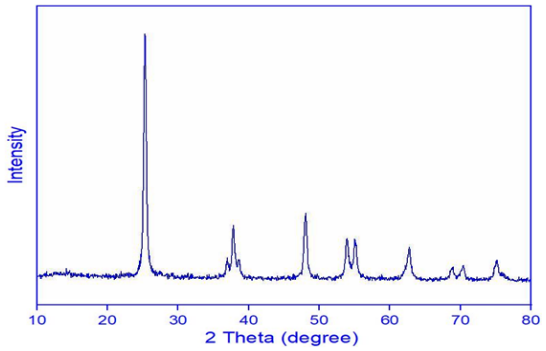


**Fig. 4. XRD Pattern of SN/$TiO_2$**

## Photocatlytic studies of plant extracted $TiO_2$ catalysts

### Photocatalytic degradation of rhodamine b dye over SG/$TiO_2$ catalyst

UV-Visible spectrum was recorded to study the photodegradation of Rhodamine B dye shown in Fig. 5 by using SG/$TiO_2$ photocatalyst at a time interval of 30 minutes. The degradation of Rhodamine-B was carried out, using sun as the main source of light from 0 to 360 min with SG/$TiO_2$. For the purpose of comparison, dark experiment was also carried out in the absence of sunlight and the results reveals only less than 1% degradation was observed. The results showed that the degradation of Rhodamine-B dye was increased with respect to time. The dye was irradiated under sunlight with 100 mg of SG/$TiO_2$ catalyst. The dye was degraded in the presence of sunlight till 360 minutes. The intensity of the peak decreased with increased in exposure time shown in Fig. 5. Thereby the absorbance of the dye was decreased with increased in time.

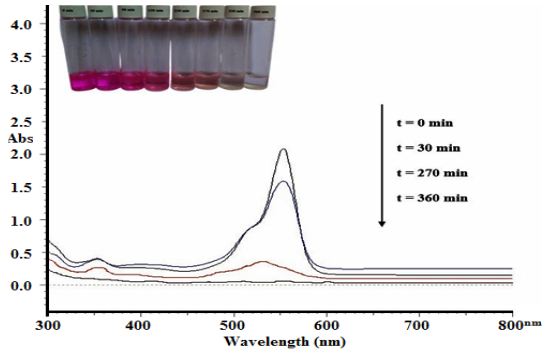


**Fig. 5. UV-Visible absorption spectra of Rhodamine B dye over SG/$TiO_2$ catalyst**

The percentage of degradation of Rhodamine B dye over SG/$TiO_2$ was calculated. From Fig. 6 it has been observed that the percentage of degradation of Rhodamine-B dye was gradually increasing as the time increases in the presence SG/$TiO_2$ catalyst when they are irradiated under sunlight. The catalyst SG/$TiO_2$ shows complete degradation of (100 %) at 360 minutes.

A plot of Absorbance vs Time in minutes shown in Fig. 6 increasing the reaction time, the absorbance value gradually decreases as the time increases and after 6 h the sample shows nearer to zero absorbance.

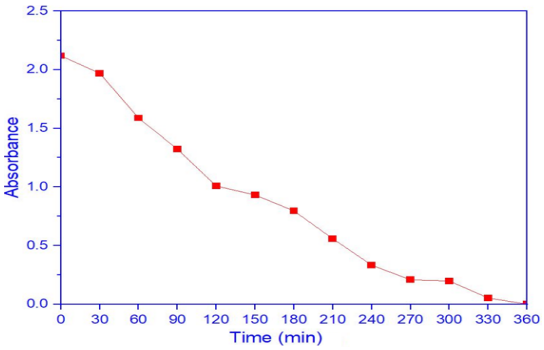


**Fig. 6. Absorbance Vs Time plot of Rhodamine B dye over SG/$TiO_2$ catalyst**

A plot of % degradation Vs Time in minutes is shown Fig. 7. It has been confirmed that as the reaction time increases, the percentage of degradation of the dye also increases and it reaches the maximum degradation of 100% at 360 min for the dye over SG/$TiO_2$ catalyst.

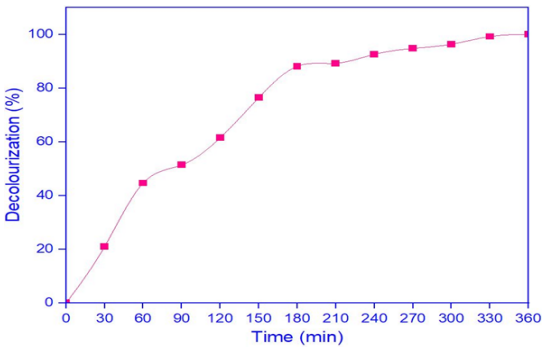


**Fig. 7. Decolourization Vs Time plot of Rhodamine B dye over SG/$TiO_2$ catalyst**

### Photocatalytic degradation of rhodamine b dye over SN/$TiO_2$ catalyst

The decolorization of Rhodamine B dye was carried out by the reaction using SN/$TiO_2$

photocatalyst at a time interval of 30 minutes. The degradation of Rhodamine B was recorded from 0 to 420 min with SN/$TiO_2$. The photocatalytic degradation efficiency of SN/$TiO_2$ catalyst was analysed. The results show that the maximum degradation was observed at 420 minutes. This is evidenced from Fig. 8 that the intensity of the $\lambda_{max}$ at 554nm was gradually decreases as the reaction time increases and also it shows a slight bathochromic 580nm. The dye was irradiated in the presence of sunlight with 100 mg of SN/$TiO_2$ photocatalyst and 100% decolorization was obtained about 7 hours. The absorbance of the dye was decreased with increased in time.

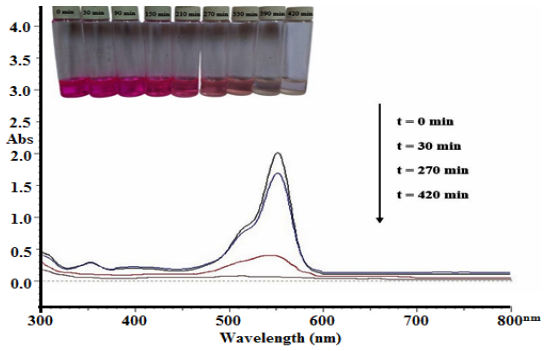


**Fig. 8. UV-Visible absorption spectra of Rhodamine-B dye over SN/$TiO_2$ catalyst**

The percentage of degradation of the Rhodamine-B dye over SN/$TiO_2$ was calculated using equation and it is given in Fig. 9. It has been observed that the percentage of degradation of Rhodamine-B dye was gradually increases as the time increases in the presence SN/$TiO_2$ catalyst when they are irradiated under sunlight irradiation. The catalyst SN/$TiO_2$ shows a maximum degradation of the dye (100%) at 420 minutes.

A plot of Absorbance Vs Time in minutes is shown Fig. 9 increasing the reaction time, the absorbance value gradually decreases as the time increases and after 7 h the sample shows zero absorbance.

A plot of % of degradation Vs Time in minutes shown in Fig. 10. It has been confirmed that as the reaction time increases the percentage of degradation of the dye increases and 100% is the maximum degradation is observed at 420 min for the dye over SN/$TiO_2$ catalyst.

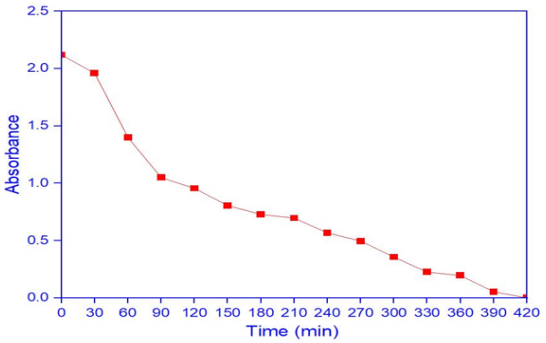


**Fig. 9. Absorbance Vs Time plot of Rhodamine-B dye over SN/$TiO_2$ catalyst**

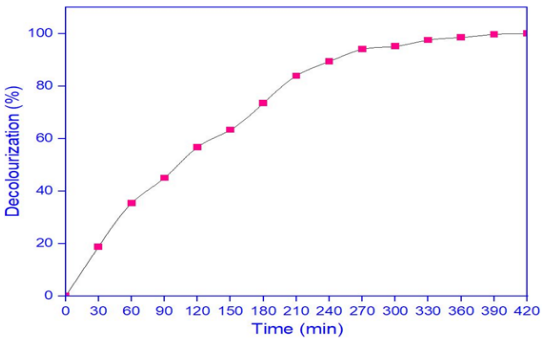


**Fig. 10. Decolourization Vs Time plot of Rhodamine-B dye over SN/$TiO_2$ catalyst**

From the above discussion it is concluded that, 100% is the maximum degradation for the dye over SG/$TiO_2$ catalyst observed at 360 min and for SN/$TiO_2$ catalyst 100% maximum degradation observed at 420 minutes. Among the catalysts SG/$TiO_2$ catalyst exhibited the maximum degradation of 100% in a shorter time of 360 minutes. Percentage of Degradation of Rhodamine–B over catalyst[24-27].

## Analysis of the degraded dye sample

Among the synthesised catalysts (SG/$TiO_2$, SN/$TiO_2$), SG/$TiO_2$ catalyst is degraded the Rhodamine-B dye into more percentage. The decolourised/degraded sample was tested for water quality parameters to confirm the conversion of dye waste water into portable water. Water quality parameters of Rhodamine B dye waste water and degraded water was carried out by experiments on pH, turbidity, total dissolved solids and conductance.

## pH

The dye effluents are highly fluctuating in pH and is important in the dyeing step, limiting chemical factor for aquatic life. Presence of concentration

of hydrogen ion is measured by pH. The acidity or alkalinity of waste water affects both industrial waste water treatment and the environment. Generally we know that below pH 7 it is acidic. Above pH 7 it is alkaline in nature. pH of 7 is neutral. The universal standard pH for waste water discharge ranges lies between[6-9]. Hence the pH of the Rhodamine-B dye sample is 7.13. After complete photo degradation of Rhodamine B sample, have the pH value is reduced to 6.86. From the result it can be concluded that the decolourised water can be used for various purpose.

**Total dissolved solids (TDS)**

A total dissolved solid (TDS) is a measure of the dissolved combined content of all inorganic and organic substances contain in a liquid. TDS is used to study of water quality for industrial waste water samples. It includes both volatile and non-volatile solids. In Water, TDS concentration can be determined using a digital meter.

**Turbidity**

Turbidity imparts an enormous problem in waste water treatment. It is a measure of cloudiness of water and measured by turbidity meter. Turbidity meter measures the scattering of light. Normal drinking water should have less than 5 NTU. It affects the growth rate of algae present in micro aquatic plants because increase the turbidity causes a decrease in amount of light for photosynthesis. It also can increase water temperature and block out the light needed by aquatic vegetation.

**Conductance**

Conductivity is a measure of water's capability to pass an electrical current. It is directly proportional to the concentration of ions in the water. Dye waste water increased the conductivity because of the presence of inorganic dissolved solids of chloride, sodium, iron, phosphate and nitrate. The conductance value of the decolurized water is decreased[28-31].

**Fig. 11. Dye water Vs Decolourised water of Rhodamine-B dye**

The water quality parameters were carried out using various techniques such as pH, Total Dissolved Solids (TDS), Turbidity and Conductance. The quality of the degraded water is under permissible limit and hence the treated water can be discharged into water bodies.

**Catalytic recyclability**

Recoverability and Recyclability are the advantages of heterogeneous catalysts, especially for commercial and industrial applications. The recyclability test of the SG/$TiO_2$ and SN/$TiO_2$ were also carried out. The catalysts can be easily separated from the reaction mixture by brief centrifugation and multiple washings with distilled water followed by drying. The recovered catalysts were evaluated for the decolourisation of Rhodamine-B dye. 100% decolourisation was attained even after the catalyst was used five times and it was monitored by UV-Visible spectroscopy. This demonstrates the high catalytic activity and photo stability of the photo catalysts[32-34].

## CONCLUSION

The purpose of the present study was to investigate the degradation of Rhodamine-B by SG/$TiO_2$ and SN/$TiO_2$ nanoparticles under solar irradiation. The synthesis of $TiO_2$ nanoparticles with faster and purest in a reliable shorter duration period. $TiO_2$ was synthesized using *Sesbania grandiflora* and *Solanum nigrum* leaves. The various functional groups were identified using the FTIR spectrum. XRD showed the crystalline nature of the $TiO_2$ nanoparticles. SEM image shows the morphology of the $TiO_2$. The degradation of Rhodamine B dye was measured using UV-Visible spectrometer. Synthesized $TiO_2$ acts as an active photocatalyst for the degradation of harmful dye as well other as waste water contaminants. The Quality of the degraded dye sample was tested for pH, TDS, Turbidity and Conductance. The recyclability test confirms the stability of green syntitania nano particles.

## ACKNOWLEDGMENT

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**Conflict of interest**

The author declare that we have no conflict of interest.

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