

Research Article

Studies on Nanotitania and Betel Leaf Powder-based Water Treatment Collected from Tiruvannamalai District



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ABSTRACT

Several techniques have been practiced for the purification of wastewater, possibly utilization of nanomaterials found to be the recent trend. According to this trend, photocatalytic water treatment using nanotitania is a well-known advanced remedy for acute water scarcity problem. Semiconductor metal oxides such as ZnO, Fe₂O₃, and TiO₂ having photocatalytic property were used. The use of nanotitania was implemented on water treatment, due to its chemical stability, larger surface area, and photocatalytic activity. In this study, nanotitania was synthesized by sol-gel method and its structure was characterized by X-ray diffractometer, transmission electron microscopy, and field emission scanning electron microscopy. Collected wastewater samples were treated with prepared nanotitania and its physicochemical parameters have been studied. Betel leaf was found to be more popularly known treatment for chronic cough and indigestion problem which is relatively natural and biosorbent. Its adsorption isotherm experimental parameters were investigated using methylene blue as adsorbate. Photocatalytic decolorization of methylene blue dye was studied with TiO₂. Nanotitania was found to be eco-friendly, having more efficient, for the treatment of wastewater.

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INTRODUCTION

In 2011–2013, the young chemist professional in the field of chemical science analyzed that, in the year of 2050, our world population will increase to 9 billion so that we have to do more work to reform on the needs of food and water. Water on earth is one of the most natural abundant resources, but only about 1% of that resource is available for human consumption.^[1] It is estimated that over 1.1 billion people lack of adequate drinking water.^[2] Supplying of drinking water stumbles due to overpopulation, industries, and agricultural growth worldwide. Due to this, freshwater resources are mainly contaminated by a variety of organic and inorganic pollutants.^[3] The change of climate and environment occurs with increase of population.^[4] Thus, treatment of wastewater and providing potable water is essential. However, the traditional methods of treatment such as boiling, filtering, and chlorination are not efficient enough to completely erase the contaminant and restrict the water quality standards.^[5] Moreover, the existing wastewater treatment technologies have several drawbacks such as energy consumption, incomplete pollutant removal, and long duration of time and generation of toxic by-products.^[6]

In general, physical and biological processes were practiced for wastewater treatment. Biological treatment processes tend to be usually slow but sometimes cause toxic to microorganism due to the presence of toxic contaminant. In physical processes, contaminant was removed by passing one phase to another, but it produces concentrated sludge which is toxic and perturbed to dispose.^[7] To overcome these shortcomings, there is a need for more efficient and powerful technologies for water treatment.^[8] Advancement in emerging technologies, namely utilization of nanoparticles, plays an efficient role in wastewater treatment and other environmental problems.^[9] Nanoparticles have structural components with one dimension at least <100 nm. In wastewater treatment, a variety of efficient, eco-friendly, and cost-effective nanomaterials have been used for the removal of contaminant from industrial water, river water, and groundwater.^[10]

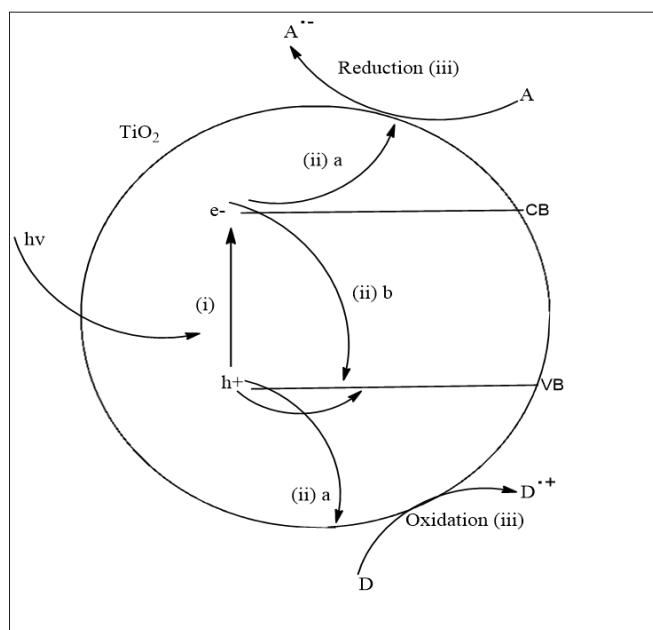
Zinc oxide-based nanomaterials were used for photocatalytic wastewater purification. Similarly, the use of nanotitania in wastewater treatment found to have effective treatment because it is not only used to treat water but it is also a semiconductor photocatalyst.^[11] Titania is an ideal



photocatalyst having long-term stability, relatively inexpensive, non-toxicity, good resistance to electrolytic solution, and holes which are highly oxidizing.^[12] Oxidation potential and energy band gap are very important for ideal photocatalyst to form photo-generated valence band holes [h^+_{vb}] and to form hydroxyl radicals [HO^*] in water which is responsible for the oxidation of organic molecules. Furthermore, the reducing power of excited conduction band electron [e^-_{cb}] must have sufficient energy to reduce molecular oxygen to superoxide.

Titania naturally exists in three crystalline phases such as anatase, rutile, and brookite having a band gap of 3.2 eV, 3.0 eV.^[12] Brookite is photochemically inactive, so it is unimportant. More studies have shown that rutile is less effective than anatase in degradation of many organic molecule due to its closely packed crystal structure. Hence, most of the research works were performed on anatase titania.

On photocatalyst reaction, titania produces a hole and an electron which acts as an active excited species.



Main processes in titania photocatalysis:

- (i) Photon absorption and electron-hole pair generation
- (ii) Charge separation and migration
 - To surface reaction sites or
 - To recombination sites
- (iii) Surface chemical reaction at active sites.



HO^* formed by oxidation of adsorbed water on the surface of catalyst by valence band hole oxygen reduces to superoxide.



Superoxide reacts with proton and electron to form H_2O_2 . H_2O_2 reacts with superoxide to form

OH radical, OH^- ion, and oxygen (20).



Hydroxyl radical has a very high oxidation potential, and it was more oxidizing potential than ozone.

Formed H_2O_2 will be split into two hydroxyl radicals through Aqueous photolysis or to accept an electron.



A is an organic molecule.

D is hole trap.

This reaction describes about the reduction of molecule by the conduction hole titania. Photocatalysis is less studied due to lower reducing power of e^-_{cb} (electron in conduction band) when compared to high oxidizing power of h^+_{vb} (hole in valence band). Superoxide ion ($\text{O}_2^{\bullet -}$) was formed when molecular oxygen reacts with titania. Further, more hydroxyl radical was produced (eq. 3–5). Production of superoxide is found to be more important since this reactive oxygen species (ROS) is responsible for the destruction of cellular membrane walls.

Thus, nanotitania adsorbed on water surface degrades the organic and inorganic substance and serves as an ideal semiconductor nanomaterial for the purification of water. Based on the literature, there was not much report on nanotitania and betal leaf-based water treatment. Hence, the scope of the present investigation clearly involves using these materials to treat the wastewater collected from Tiruvannamalai district.

EXPERIMENTAL SECTION

Materials

Titanium isopropoxide, absolute ethanol, nitric acid, and distilled water were used. Commercial available reagents and solvents were purchased from Sigma-Aldrich and used as such without any purification.

Preparation of nanotitania

In this study, TiO_2 was prepared by sol-gel method. 3 ml of titanium isopropoxide was mixed with 25 ml of absolute ethanol in a separate beaker, and this mixture was added dropwise to 30 ml of distilled water in a four-necked round bottomed flask fitted with water condenser, thermometer, guard tube, and additional flask with constant stirring for about 30 min. The whole mixture turned to thick precipitate after complete addition and then raises the temperature to 75°C with constant stirring for 45 min to ensure complete hydrolysis. After that, 80 ml of 0.4 M nitric acid was added dropwise to the mixture through addition funnel with constant stirring for about 4–5 h. After 4–5 h, the solution was cooled at room temperature. The pH of prepared 20% TiO_2 was found to be <1 .

Preparation of activated betel leaf (BL) and dye solution

BLs were purchased in the local market and washed repeatedly with distilled water to remove dust and other impurities. It was dried then ground well and finally treated with boiling zinc chloride in the ratio of 2:1 (which is a chemical activating agent to produce greater surface area) and then leached out with diluted HCl. Then, it was repeatedly washed with distilled water to remove the zinc chloride, filtered, and then dried in an electric oven. The prepared activated BL was sieved and used for the experiment.

The molecular formula of methylene blue dye is $C_{16}H_{18}ClN_3S \cdot 3H_2O$ (molecular weight = 319.85 g/mol, $\lambda_{max} = 664$ nm) and a stock solution of 250 mg was prepared with distilled water. All working solutions were prepared by diluting 10 ppm of stock solution into 100 ml, and this concentration is followed during the investigation. All chemicals used in this test were of analytical grade.

CHARACTERIZATION

Characterization of TiO_2

About 50 ml of titania solution was evaporated in a rotary evaporator at 60°C using a vacuum pump (50 torr pressure) and pale yellow colored solid was obtained which was kept under vacuum for 1 h at room temperature. Then, the powdered titania was characterized by powdered X-ray diffractometer (XRD) for structure and crystal phase. The morphologies of titania were characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) as shown in Figure 1a-d.

XRD

The sharp and high intensities of the XRD peaks as obtained showed that it is anatase titania having $2\theta = 25.454^\circ$. Phase of anatase was (1 0 1), and confirmed synthesized titania was anatase phase. No other characteristic peak was detected.

Field emission scanning electron microscopy

The porous structure of titania greatly favors the improvement of photocatalytic activity through enlarging the specific surface area and promoting the electron transfer enhancing the photosensitized reaction.

TEM

The morphology of prepared nanotitania has a spherical shape on magnifying at 50 nm.

Characterization of adsorbent

The adsorbent BL was characterized with Fourier-transform infrared (FTIR) and SEM. The FTIR spectrum of BL has shown various functional groups with respect to their peak value. Peak at 3290 cm^{-1} indicates $-\text{OH}$ stretching, 2918 cm^{-1} indicates $-\text{CH}$ stretching of $-\text{CH}_3$. 2850.71 cm^{-1} is due to methylene $-\text{CH}_2$ group, the peak value at 1729 cm^{-1} is C=O group, 1608 cm^{-1} is C=C presence of aromatic ring, and 1023 cm^{-1} is C-H bending of aromatic hydrogen. From Figure 2a and b, SEM Characterization: It has rough, uneven, and heterogeneous surface with porous structure, which indicates the availability of high surface area for biosorption of dyes.

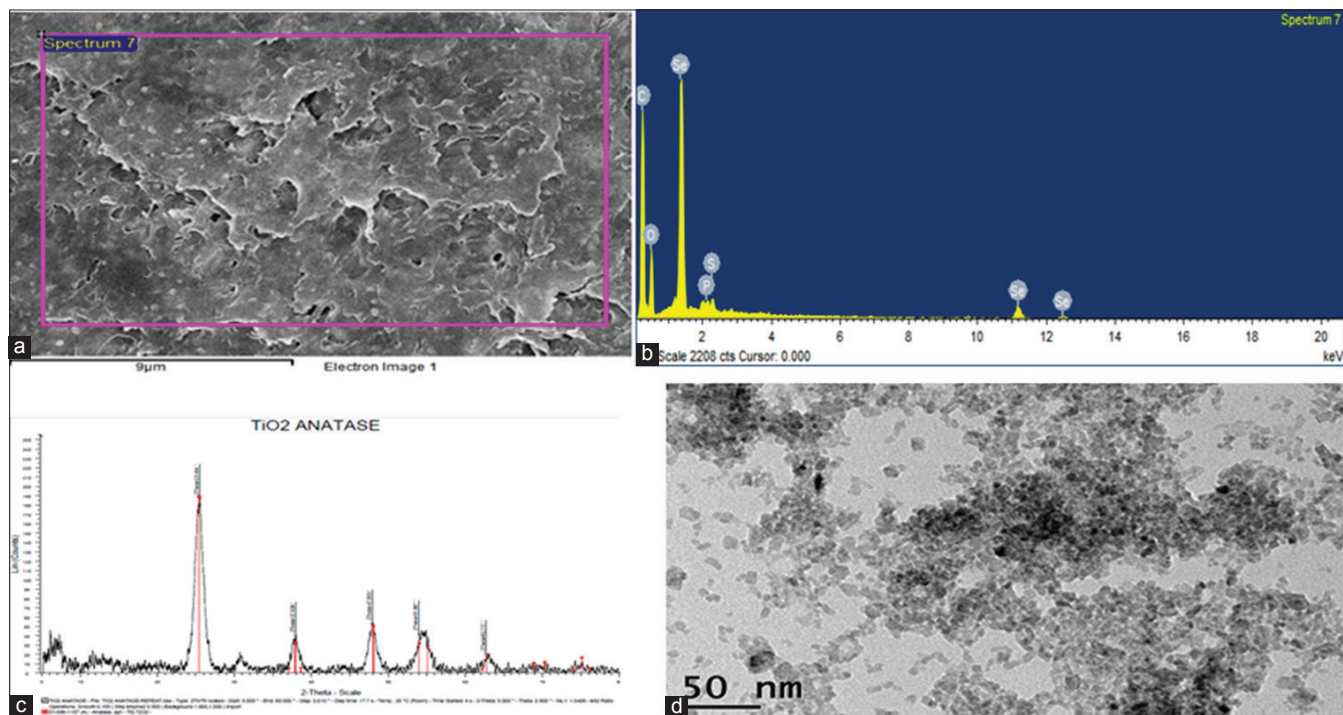


Figure 1: (a) Field emission scanning electron microscopy image of TiO_2 , (b) EDAX image of TiO_2 , (c) X-ray diffractometer image of TiO_2 , (d) transmission electron microscopy image of TiO_2

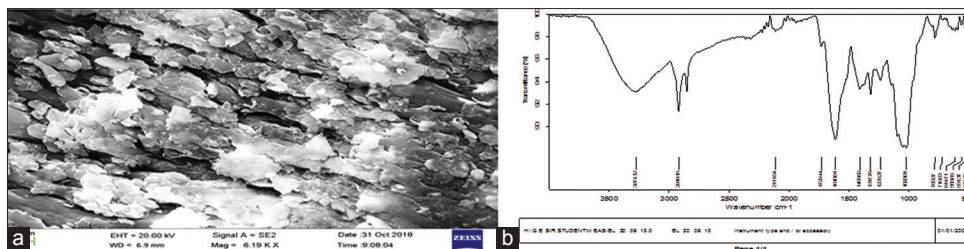


Figure 2: (a) Field emission scanning electron microscopy image of betel leaf (BL), (b) Fourier-transform infrared of BL

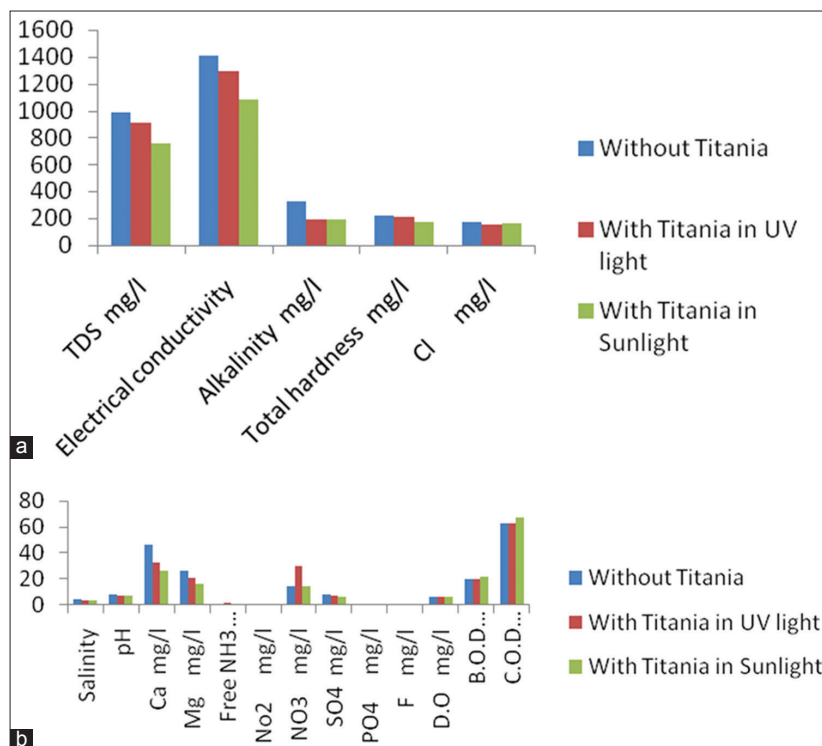


Figure 3: (a and b) Physicochemical examination of pond water, treatment with and without titania

RESULTS AND DISCUSSION

Wastewater is that the water which has been contaminated with organic substances, microorganisms, and industrial effluent. Water discharged from domestic residences and commercial properties causes pollution to both surface and groundwater, and this is led to drainage termed as municipal wastewater. Preliminary treatment was done with samples collected in and around Tiruvannamalai district, and to remove coarse and readily settable solids, the sample was filtered. Prepared nanotitania solution was added to the collected samples with constant stirring and kept for 1 h in the presence of solar and ultraviolet (UV) light. Evaluations of parameters were studied in presence of solar and UV-light and the same work has also been conceded out with activated BL, and their results are presented in Figures 3-5a and b.

After treatment with titania

Parameters

Physicochemical examination values obtained were compared with standard values as per the World Health

Organization 2015 standard specifications. Values of total dissolved salts, electrical conductivity, salinity, alkalinity, total hardness, Ca, Mg, Cl, SO₄, PO₄, and F were found to be within the permissible limit. pH value of solution attains neutral.

Total nitrogen

Standard recommended levels of nitrate, nitrite, and free ammonia concentration in drinking water were found to be 50 ppm, 3 ppm, and 0.5 ppm, respectively. However, after treated with TiO₂ solution free ammonia, nitrate values were increased. These data clearly shows that while photocatalytic reaction takes place in the presence of titania as catalyst photocatalytic reduction of nitrite and ammonia as by products.^[13]

When the energy of incident light is larger than that of semiconductor, electrons are excited photons formed on the valence band h⁺_{vb} and these photons are responsible for the reduction reaction to take place. Ammonium is commonly formed as the Aqueous by-product of photocatalytic nitrate reduction.^[14]

The concentration of both nitrite and ammonium is slowly increased. These are intermediates of the reactions

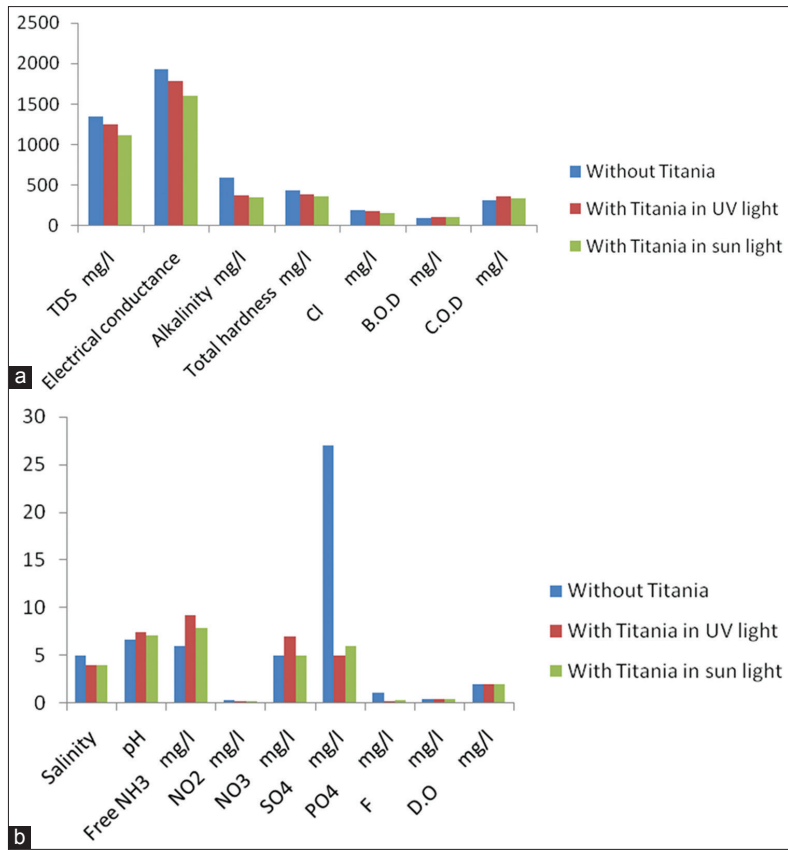


Figure 4: (a and b) Physical examination of sewage water, treatment with and without titania

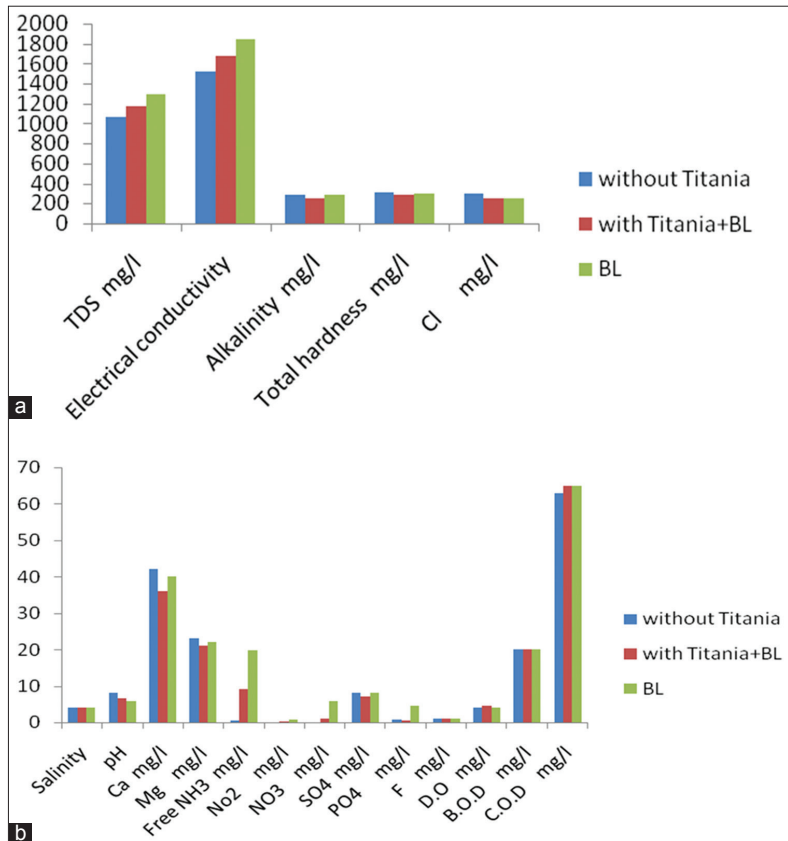
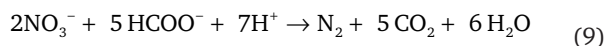


Figure 5: (a and b) Physicochemical examination of pond water, treatment with and without titania, betel leaf

and were readily converted into the final product as nitrogen gas as shown in Equation 9.^[13] Photogenerated electron-hole pairs can recombine within a few nanoseconds, and this can be overcome by adding a hole scavenger (electron donor) to trap holes leaving photogenerated electrons available for nitrate reduction.^[14]



However, the presence of biologically unstable hole scavengers and ammonium is not desirable for drinking water during wastewater treatment.

Bacteriological examination

The presence of microorganism was identified by colonies formed on the media (Agar-7) prepared in Petri dish. Mostly fecal coliform and fecal streptococci were present in the surface of sewage and pond water. After treated with titania, no such colonies were present in the part of titania located in Petri dish, and this shows its antimicrobial activity [Figure 6]. Titania can kill both Gram-negative and Gram-positive bacteria. More recently, nanosize titania reported to kill viruses including poliovirus 1,^[15] hepatitis B virus,^[16] herpes simplex virus,^[17] and MS2 bacteriophage.^[18] To destroy bacteria, concentration of titania is more important. The antibacterial activity of titania depends on the production of ROS, i.e. hydroxyl free radical and peroxide formed during photocatalytic reaction.^[19] ROS generally works by destructing cell membrane, damaging DNA and protein, releasing hazardous ions for cell malfunction, disrupting electron transfer, and hampering respiration process.^[20]

Dissolved oxygen, chemical oxygen demand (C.O.D), and biochemical oxygen demand

Photoelectron in TiO_2 is a relatively weak reducing agent, and the solubility of oxygen in Aqueous solution is commonly low. In photocatalysis treatment, oxygen plays an essential role for the conversion of CO_2 . If oxygen concentration changes, then it indicates the change in organic concentration, and thus, oxygen consumed is directly proportional to C.O.D concentration. From this, it shows that degradation of organic matters takes place.

Treatment with BL

Values of total dissolved salt, electrical conductivity, salinity, alkalinity, total hardness, Ca, Mg, Cl, SO_4 , PO_4 , and F

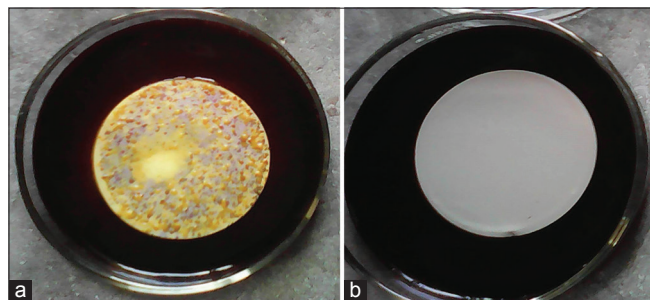


Figure 6: (a and b) Antibacterial activity test by titania

increased and showed no degradation reaction. Free ammonia and phosphate value increase shows their presence in it.

Hence, the activated BL can be used for the removal of dyes by adsorption process instead of using to purify the wastewater.

Dye plays an important role in textile, paper, leather, and print industries. Discharging of these dyes in environment causes contamination in water bodies affecting the water and food cycle of humans. Moreover, these dyes are noxious and hazardous for water organism.

Adsorption process is one of the familiar methods used for wastewater treatment. In general, for the adsorption process, activated carbons were used. However, commercially available activated carbon is expensive and this leads to the search for alternative adsorbent which is having a larger surface area and adsorption capacity. To overcome this setback, research works have been focused on usage of cheap, easily available reusable, and biodegradable adsorbents from natural sources such as rice hull shell,^[21] wood apple shell,^[22] and powdered peanut hull.^[23] From the appraisal of literature, there is no information with BL used as an adsorbent. The objective of this study was to observe the effectiveness of BL for the removal of methylene blue dye.

Batch equilibrium studies

The experiment was carried out by batch adsorption method. The adsorption parameters such as effect of time, dye concentration, stirring speed, and temperature were studied. The pH is maintained to neutral by adding 0.1 N HCl or NaOH. The absorbance of color was measured using EI Model 312 spectrophotometric at $\lambda_{\text{max}} = 680$ nm. The equilibrium adsorption was evaluated using the following equation.

$$q_e = \frac{(c_i - c_e)v}{m}$$

Where q_e is equilibrium adsorption capacity (mg/g), c_i and c_e are the initial and equilibrium concentration (mg/g) of MB dye solution, “v” is volume of dye, and “m” is the weight of adsorbent. The percentage removal of dye was calculated as follows:

$$\% \text{ dye removal} = \frac{C_i - C_e}{C_i} \times 100$$

Effect of adsorbent

The adsorption of MB on BL was studied by varying the dosage of the adsorbent (1–5 mg) at a dye concentration of 10 ppm, and the effect of BL on the adsorption is shown in Figure 7. The removal percentage was increasing with BL amount up to a certain limit (5 mg), and then, it remained approximately constant. Increasing in the percentage of removal with an increase in adsorbent dosage is due to the increase in adsorbent surface area.^[24]

Effect of concentration of dye

About 100 ml of MB solution of various concentration 100, 150, 200, 250, and 300 ppm was taken in five different conical flasks to which 5 mg of BL was added and then shaken at 150 rpm for 60 min. Maximum percentage removal occurred at a lower concentration of MB and decreases percentage

removal with an increase in MB concentration. Hence, with an increase in dye concentration, no further adsorption will occur due to adsorption sites were fixed [Figure 8].

Effect of time

About 100 ml of MB and 5 mg of BL were taken in a 250 ml conical flask and stirred at 150 rpm, for a regular interval of time, and the percentage absorbance was observed. The maximum amount of dye removal took place for 60 min, and then, it remained almost constant and then reached equilibrium [Figure 9].

Effect of temperature

The adsorption process is temperature dependent. As the temperature increases, the adsorption capacity also increases as shown in Figure 10.

Effect of stirring speed

The stirring speed was varied from 50 to 200 rpm but from 150 rpm percentage removal of MB remained constant as shown in Figure 11.

Adsorption isotherm

The relationship between the amounts of substance adsorbed to its concentration in the equilibrium solution at constant temperature is termed as adsorption isotherm. The Langmuir and Freundlich models are the most common types

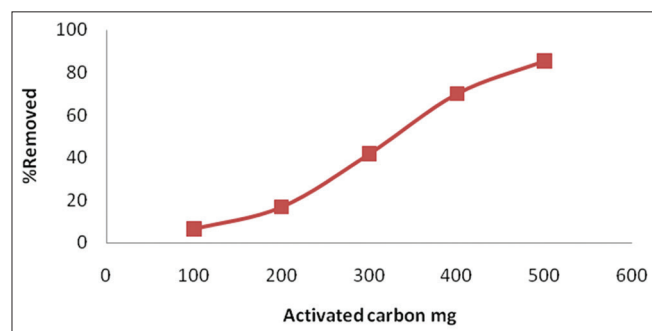


Figure 7: Effect of adsorbent, Volume = 100 ml, Time = 60 min, T = 301 K, Stirring speed = 150 rpm

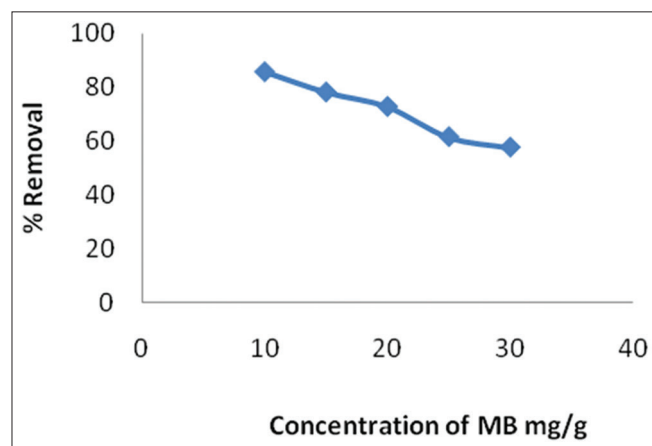


Figure 8: Effect of concentration of dye, volume = 100 ml, Time = 60 min, T = 301 K, Stirring speed = 150 rpm

of isotherm which were used to find experimental sorption data.

Langmuir isotherm

Langmuir isotherm assumes that maximum adsorption happens to a saturated monolayer of adsorbate molecules on adsorbent surface. The energy of adsorption is stable, and there is no migration of adsorbate molecules in the surface place of adsorbent.^[25] Langmuir isotherm is expressed as:

$$1/q_e = 1/q_m + 1/K_L q_m C_e$$

Where q_e is equilibrium dye concentration on adsorbent (mg/g), q_m is monolayer capacity of adsorbent (mg/g), C_e is equilibrium dye concentration (ppm), and K_L is Langmuir adsorption constant. Plot of C_e/q_e versus C_e found to be linear as shown in Figure 12. The values of q_m and K_L were 26.54 mg/g and 0.026 L/mg, respectively. Langmuir isotherm can be expressed by a constant equilibrium parameter R_L .

$$R_L = 1/1 + q_m C_0$$

R_L value indicates that the type of isotherm is favorable or unfavorable in this study ($R_L < 1$), and hence, it was favorable.

Freundlich isotherm

Freundlich isotherm model is an exponential equation considering that the interaction between adsorbed molecules is not restricted to the formation of monolayer. Freundlich model is expressed as:

$$\log q_e = \log K_f + 1/n \log C_e$$

Where q_e is the amount of adsorption and K_f is Freundlich constant related to adsorption capacity and adsorbent.^[26] The

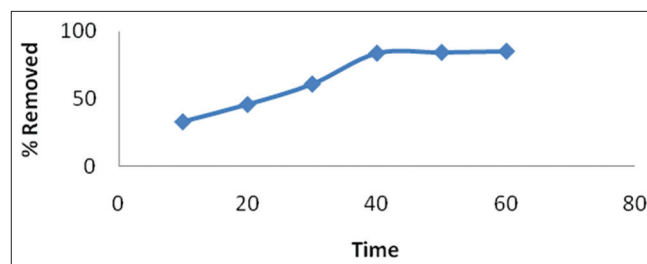


Figure 9: Effect of time, weight of activated carbon = 0.5 mg, volume = 100 ml, T = 301 K, stirring speed = 150 rpm

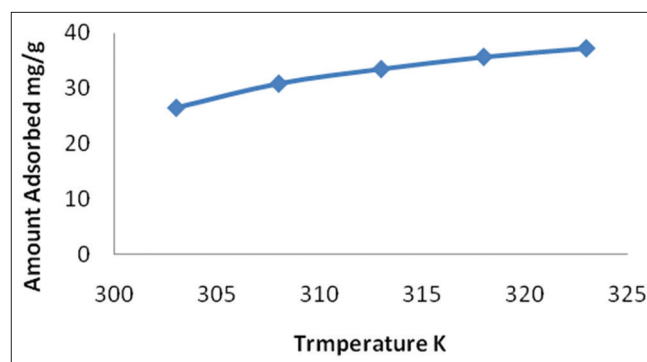


Figure 10: Effect of temperature, volume = 100 ml, time = 60 min, stirring speed = 150 rpm, weight of activated carbon = 5 mg

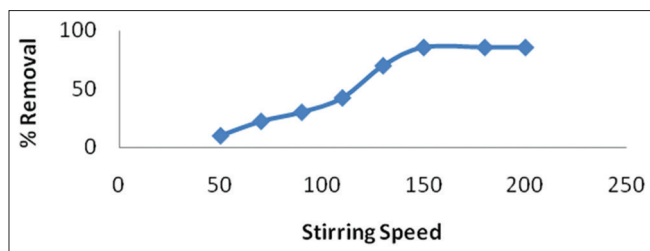


Figure 11: Effect of stirring speed, volume = 100 ml, T=303 K, time = 60 min, weight of activated carbon = 5 mg

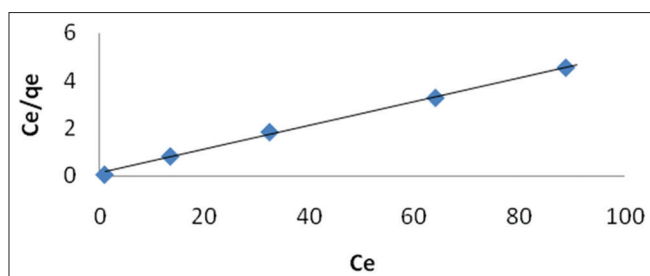


Figure 12: Langmuir adsorption isotherm of MB onto BL

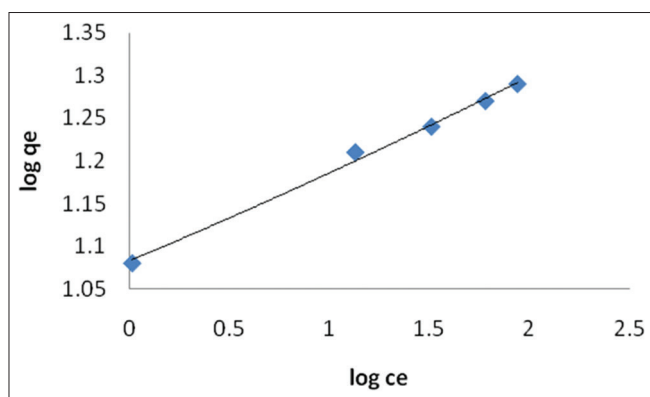


Figure 13: Freundlich adsorption isotherm of MB onto betel leaf

Langmuir isotherm		R	Freundlich isotherm	
qm (mg/g)	K _L (L/mg)		1/n	K _F
26.54	0.026	0.524–0.0178	0.13	16.82

value of Freundlich constant K_F and $1/n$ was 16.82 and 0.13. Figure 13 determines the linear plot of $\log q_e$ versus $\log c_e$; its intercept and slope give the value of K_F and $1/n$. Value of $1/n$ was between 0 and 1 which shows that adsorption is heterogeneous.

Kinetics models for adsorption

The adsorption kinetics data of MB dye are analyzed using Lagergren first-order rate equation:

$$\log (q_e - q_t) = \log q_e - K_1 t$$

Pseudo first-order model was plotted by $\log (q_e - q_t)$ versus t . Kinetics constant were calculated from its slope and intercept of plots; the value of q_e was 11.73 mg/g which was

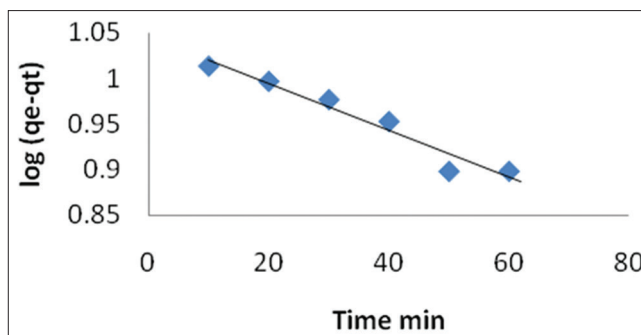


Figure 14: Pseudo-first-order kinetics for adsorption of MB onto betel leaf

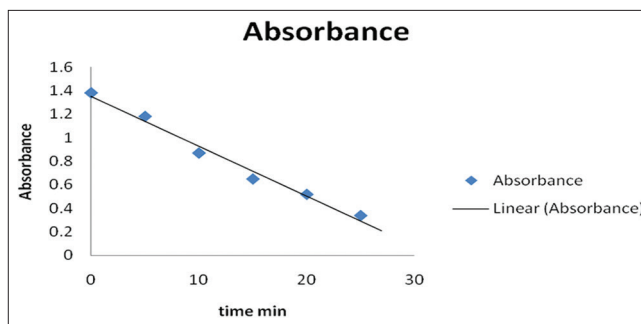


Figure 15: Photocatalyst decolorization of TiO_2

correlated with experimental data and this indicates that pseudo first-order is better fitted [Figure 14].

Amount adsorbed was obtained by:

Experimental value = 11.73 mg/g

Calculated value = 12.43 mg/g

$$K_1 = 0.49 \times 10^{-3} \text{ min}^{-1}$$

Photocatalytic decolorization of MB by nanotitania

TiO_2 was diluted to 0.3% solution using distilled water, to this, 10 ppm of 50 ml MB solution was added in a closed flask, and then, the whole setup was placed in sunlight. The removal of dye was measured by UV spectrophotometer at regular interval of time [Figure 15]. During photocatalytic decolorization of MB dye molecule, the electron was transferred from excited dye molecule to the conduction band of TiO_2 , unstable dye cation radical was formed, and simultaneously, active species on TiO_2 reacts with dye molecule to destabilized it. A comparison of the concentration of MB dye was noted before and after adsorption, which showed a decrease in adsorption of MB dye. A concentration of TiO_2 affects the degradation rate. An increased loading of catalyst increases the quantity of photons adsorbed and consequently the degradation rate.^[27]

CONCLUSION

Nanotitania is one of the ideal choices for advanced wastewater treatment processes. It can be used for the treatment of both toxic contaminant and also municipal

wastewater. It has the capability of purification of wastewater in solar light instead of using expensive artificial UV radiation. When compared with activated carbon from BL, parameters of water given by the WHO were not within the permissible limit. Whereas, nanotitania has antimicrobial activity when compared with BL. Thus, future technologies will focus on efficient processes which need small quantities of nanomaterial. Thus, nanotitania can be used for wastewater treatment which has more efficiency when compared with conventional methods.

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