ARUNAI PUBLICATIONS

Asian Journal of Applied Research

DOI: http://dx.doi.org/10.20468/ajar.2018.12.01

Research Article

Economical and Eco-friendly Wastewater Treatment Using Anatase TiO_2

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ABSTRACT

Proliferating awareness of health, economic benefits, and green environment is required in the polluted world. From the peril of water pollution, novel method of wastewater treatment is required. Nanotechnology is found to be a more efficient process while comparing with other traditional process. Among the other nanoparticles, titania is ideal for wastewater treatment. It involves photocatalytic reaction. Nano titania was prepared by the sol-gel method. It was characterized by scanning electron microscopy (SEM) and X-ray diffractometer analysis. Low-cost, eco-friendly activated carbon was synthesized from wood apple (WA) outer shell composite by conventional chemical process. Activated WA was analyzed by Fourier-transform infrared and SEM. Both Nano titania and WA were treated with collected samples. Their parameters were investigated. Nano titania was found to be more potential than WA. Adsorption process was one among the method used for wastewater treatment in removal of dye. Methylene blue was a major pollutant due to its enormous use in the textile industries. Their adsorption capacities were studied by batch experiment. The adsorption efficiency of WA was determined by measuring the percentage removal of color. The effect of contact time, concentration of dye, temperature, stirring speed, and kinetics was studied. Nano titania is more efficient for the treatment of wastewater discharge from household and industries.

Keywords:
Adsorption isotherm, Biosorbent, Nano titania, Photocatalyst examination, Wood apple outer shell

Received: 12th October 2018
Accepted: 20th November 2018
Published: 25th December 2018

INTRODUCTION

Water scarcity is the main problem faced by the world in the 21st century. Usage of water has been increasing twice the population when compared to last centuries. As per the report from the United Nation by 2025, 1800 million people will be facing water scarcity and two-third of world population would be under stress.[1] Wastewater discharged from domestic, commercial, agricultural, and industries are the main source of pollution. Wastewater from industries must be treated before discharge into natural resources. This process must be economical and non-toxic to the environment. From different literature survey field, nanotechnology was one of the most advanced processes for wastewater treatment. The nanomaterials such as metal oxides and semiconductors made attention for scientist in developing wastewater treatment. Nano catalyst which has degradation of organic matters and antimicrobial properties can be used for photocatalytic reaction in wastewater treatment.

Photocatalyst has been proved as a promising technique for purification and treatment of various kinds of wastewater.[2] Nanomaterial catalyst can be used in the chemical oxidation of organic pollutants. It was developed from noble metal which has efficient photocatalytic potential for the degradation of organic and inorganic contaminants.[3] The discovery of photo-induced water splitting on TiO_2 electrode has promoted extensive research on TiO_2 and other semiconductor materials.[4] Catalytic material was activated by light during photocatalyst reaction. Photocatalysis process is a novel method used for wastewater treatment. When it was conducted under proper conditions, the photocatalytic oxidation of most organic contaminants occurs and results in complete oxidation to carbon dioxide, water, and halide ions.[5] Nano titania has chemical stability, large surface area, non-toxicity, inexpensive, and photocatalytic activity. Hence, it is anticipated to play an important role in many fields. Titania exists in three phases, namely anatase, rutile, and brookite. Brookite is uncommon and unstable. Rutile has small surface area and has low photocatalytic activity. However, anatase has larger surface area and photocatalytic activity. Hence, it is used in photocatalysis.[6]

During the photocatalytic reaction, titania takes energy from light to occur chemical reactions, involving the generation of hydroxyl radicals. This hydroxyl radical degrades organic pollutants. Hence, this technology captures the attention for the use of wastewater treatment. Titania is a semiconductor heterogeneous photocatalyst. This reaction occurs by absorption of photon with energy greater than the band gap of semiconductor

which produces negative charge electron on conduction band and positive charge hole in the valence band as shown in equation 1.

$$TiO_2 \xrightarrow{h\nu} e_{cb}^{-}(TiO_2) + h_{vb}^{+}(TiO_2) \quad (1)$$

$$H_2O + h_{vb}^{+} \rightarrow HO^{\bullet} + H^{+} \quad (2)$$

$$O_2 + e_{cb}^{-} \rightarrow O_2^{\bullet -} \quad (3)$$

TiO_2 acts as an electron donor and electron acceptor. Since the valence band hole is strongly oxidizing and the conduction band electron is strongly reducing. Thus, both oxidation and reduction reaction occur at the surface of the photoexcited TiO_2 particle.[7]

Oxygen reduces to super oxide.

$$O_2^{\bullet -} + H^{+} \rightarrow HO_2^{\bullet} \quad (4)$$

$$HO_2^{\bullet} + HO_2^{\bullet} \rightarrow H_2O_2 + O_2 \quad (5)$$

$$H_2O_2 + e_{cb}^{-} \rightarrow HO^{\bullet} + OH^{-} \quad (6)$$

Hydroxyl radical has more oxidizing potential than ozone. Oxidation potential of compound is higher, and therefore, decomposing capacity is also greater.

$$D + h_{vb}^{+} \rightarrow D^{\bullet +} \quad (7)\ D \rightarrow \text{Hole trap}$$

$$A + e_{cb}^{-} \rightarrow A^{\bullet +} \quad (8)\ A \rightarrow \text{Organic molecule}$$

Equation (8) describes the reduction of an adsorbed molecule by the conduction band hole. In back reaction, the electron and hole will recombine, which releases heat.

$$TiO_2[\, e_{cb}^{-} + h_{vb}^{+}] \rightarrow TiO_2 + \text{heat} \quad (9)$$

A complete photoreduction, photooxidation, and adsorption occur on the TiO_2 surface between water, organic molecules, and other metals. Thus, TiO_2 can be used for photocatalytic water treatment.

Extensive use of dyes in the textile, paper, and leather industries causes pollution due to discharge of colored wastewater into the natural resources. This makes an adverse esthetic effect reduces aquatic lives by blocking light passage through water reduces photosynthetic activity.[8] This colored wastewater is hazardous to human health and hence needs to be treated before discharged. Different methods have been recently used for removing some specific colorants. These methods include carbon adsorption, dye reduction, ozonation, and electrochemical techniques.[9] In this study, carbon adsorption method is used for color removal from wastewater. Adsorption process using activated carbon is widely used to remove contaminant in wastewater. However, commercially available activated carbon is expensive; this leads to search for low-cost carbon as alternative adsorbent. In recent years, many researchers have studied production of activated carbon from bagasse,[10] jute fiber,[11] mango seed husk,[12] and nut shells.[13] By utilizing rejected waste as raw material, effective carbon can be made at low cost.

In this process, wood apple (WA) outer shell was used as an adsorbent to remove methylene blue (MB) dye in wastewater. It was used as notable medicine for chronic throat and preventive medicine for cancer. The carbon made from this biomass waste has highly porous structure and high surface area.

EXPERIMENTAL

Chemicals

Commercial available reagents were purchased and used as such without any purification. titanium isopropoxide (TTIP) from Sigma-Aldrich. Absolute Ethanol, Nitric acid and distilled water.

Preparation of titania

Preparation of 20% titanium dioxide (anatase) was done by sol-gel method. Many methods for titania preparation were followed such as e-beam evaporation, sputtering, chemical vapor deposition, and sol-gel method; among these,

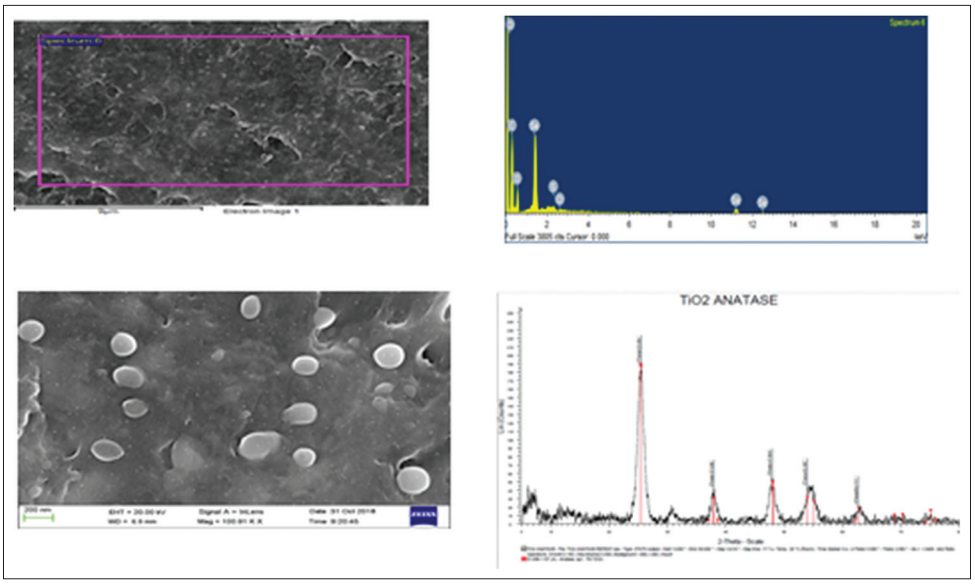


Figure 1 : Scanning electron microscopy EDAX and X-ray diffractometer images of titania

sol-gel method was widely used due to its simplicity and low equipment requirement. 369 ml of TTIP is mixed with 200 ml of absolute ethanol in a separate beaker. This mixed solution was added drop wise into 200 ml of distilled water in a 2 L four necked round bottomed flask fitted with water condenser, thermometer, and guard tube, and then, it has to be continued for about 30 min under vigorous stirring. During the addition of TTIP, an exotherm was expected. The whole mixture leads to a thick precipitate after complete addition. The temperature of the RB flask raised to 75°C and continued stirring for another 45 min to ensure complete hydrolysis. Then, 240 ml of 0.4 M nitric acid was added and the mixture was continuously stirred under 75°C for about 4–5 h in an air proof condition. After 4–5 h of stirring, the obtained solution was cooled to room temperature. The pH of prepared 20% titania found to be <1.

Materials required

WA outer shell, MB dye from Qualigens Fine Chemicals, and distilled water were used for preparing all the solutions.

Preparation of activated carbon

WA was purchased in the market, and its outer shell was washed several times with distilled water to remove impurities and dried in sunlight. The resultant material was grounded well and mixed with boiling solution of $ZnCl_2$ (activating agent) in the ratio of 2:1, filtered, and dried. It was leached with diluted HCl and repeatedly washed with distilled water to ensure the removal of $ZnCl_2$ and dried in an oven. The prepared activated WA was sieved and used for the experiment.

Batch experiment

The experiment was performed in batch mode using MB (molecular formula $C_{16}H_{18}ClN_3S$. $3H_2O$ and molecular weight 319.85 g/mol, λ_{max}= 664 nm) as adsorbate. The stock solution of MB of 250 mg/L was prepared. The effect of contact time, concentration of dye, temperature, stirring speed, weight of adsorbent, and adsorption isotherms were studied. The adsorption of dye was determined by UV spectophotometer. The removal of dye can be calculated by

$$\% \text{ dye removal} = \frac{C_0 - C}{W} \times 100$$

C_0 and C were the concentration of initial and equilibrium concentration of dye. For adsorption isotherm the amount of adsorbed dye at equilibrium was calculated by

$$Q_e = \frac{C_0 - C_e}{W} \times V$$

Where C_e is equilibrium dye concentration (mg/L), W is weight of adsorbent, and V is volume of solution.

Characterization

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

- Field emission SEM (FESEM): The porous structure of titania shows its enlarging specific area favoring photocatalytic activity for promoting the electron transfer process.
- XRD: The sharp and high intensities of XRD peaks have obtained as anatase titania having broadening analysis at (1 0 1) phase with 2Θ = 25.454. No specific impurity peaks were detected.
- Fourier-transform infrared (FTIR): The synthesized adsorbent was characterized by FESEM and FTIR [Figure 2]. The FTIR spectrum of WA shows various functional groups with respect to their peak value. Peak at 3336 cm^{-1} and 2918 cm^{-1}

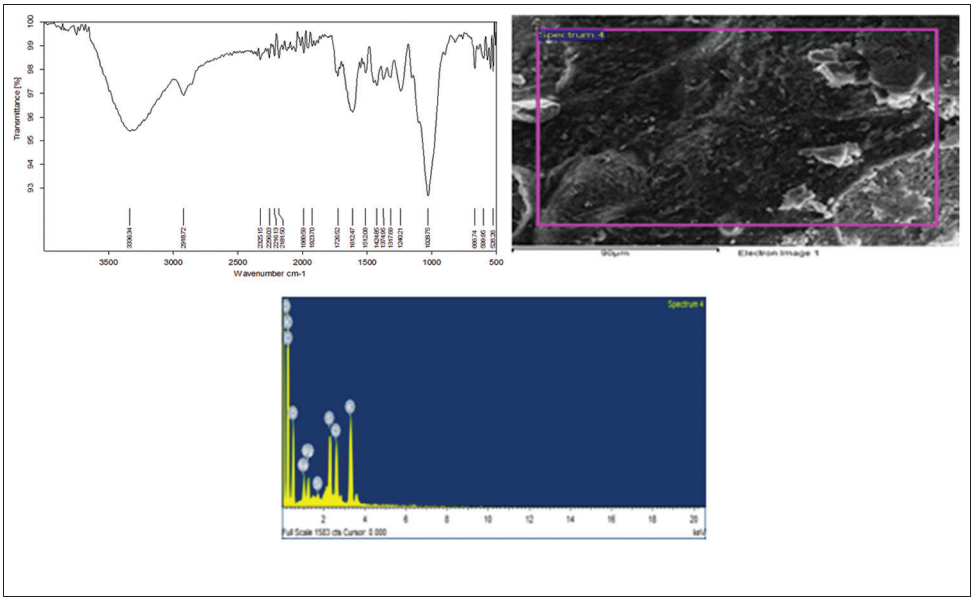


Figure 2: Fourier-transform infrared and field emission scanning electron microscopy images of wood apple

indicates –OH and –CH stretching in $–CH_3$. Peak at 3336 cm^{-1} and 2918 cm^{-1} indicates –OH and –CH stretching in –CH3. Peaks at 1726, 1028, 1512, and 1612 cm^{-1} corresponds to C=O in lactone, -CH bending, C=C, Carboxylate ion and Pyrone respectively.

- FESEM: SEM images of WA show rough, uneven and seem to be homogeneous with porous structure which indicates for adsorption for dyes.

RESULTS AND DISCUSSION

Samples were collected from sewage and industrial effluent from Vellore district; then, its physicochemical examination was examined with and without the presence of synthesized nano titania. About 10 ml of the prepared TiO_2 solution was added to 1 L of collected samples with constant stirring, which was kept under solar light nearly 1 h, and parameters were determined [Figures 3 and 4]. Wastewater treatment by adsorption process was performed with samples using activated WA outer shell composite [Figure 5]. 5 g of WA was added to 1 L of sample and kept in a shaker for 1 h.

Photocatalytic wastewater treatment

No degradation of organic contaminant occurred while treating with WA. Hence, TiO_2 was very efficient for wastewater treatment by the photocatalytic reaction.

After treating with TiO_2, color changed from blackish to colorless, clear, and odorless. The other parameters such as TDS, E.C, T.H, Ca, Mg, Cl, SO_4, and PO_4 obtained values found to be within the permissible limit given by the World Health Organization.

Total nitrogen

After treatment with TiO_2, it was observed that free ammonia value increases. This is due to photocatalyic reduction of nitrate. Protons may begin to dominate the photocatalyst surface, making them available for ammonium formation.[10] The effect of oxygen on nitrate reduction was evaluated to determine the importance of electron availability. For metal-free titania, nitrite is either formed in amounts directly related to the amount of nitrate reduction[11] or is not detected at all but may be a potential intermediate in the selective transformation of nitrate to ammonia.[12] Photogenerated electrons were used in the reduction of protons to yield hydrogen gas and ammonia than leading to nitrate reduction to nitrogen.[13]

D.O and C.O.D

Dissolved oxygen values raise with titania, indicating that dissolved oxygen in sample solution which is a typical electron acceptor reacts with the electrons and forms hydrogen peroxide or water.[14]

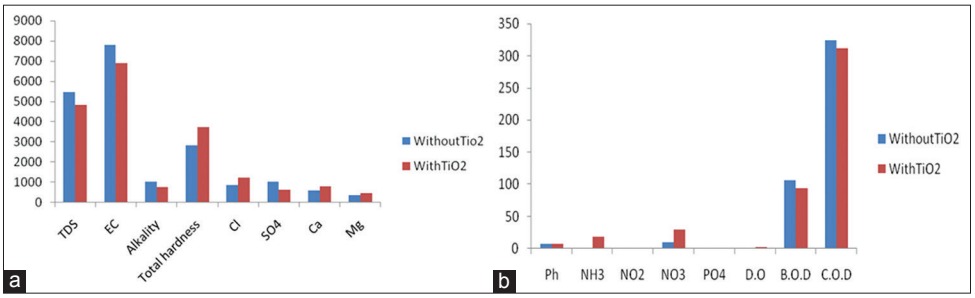


Figure 3: (a and b) Wastewater treatment from industrial effluent with and without titania

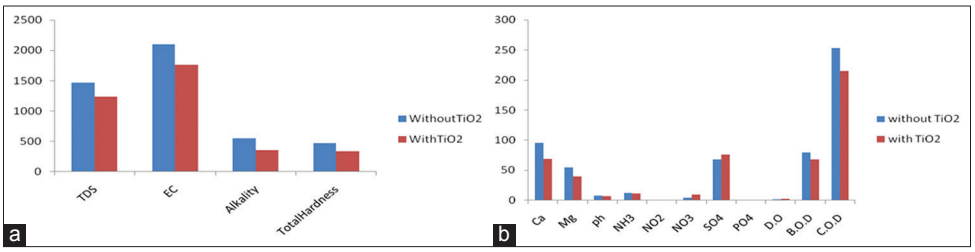


Figure 4: (a and b) Wastewater treatment from sewage water with and without Titania

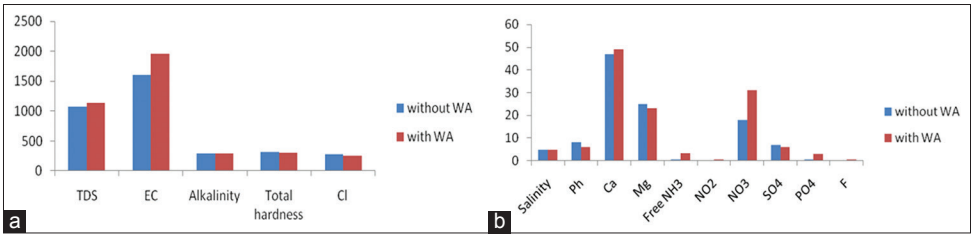


Figure 5: (a and b) Wastewater treatment with activated wood apple

$$2e^-_{cb} + 2H^+ + O_2 \rightarrow H_2O_2 \text{ (or)}$$
$$4e^-_{cb} + O_2 + 4H^+ \rightarrow 2H_2O$$

However, photoelectron in TiO_2 is a relatively weak reducing agent and the solubility of oxygen in aqueous solution is commonly low. When oxygen concentration changes ultimately then it indicates the change in organic concentration. In photocatalytic mineralization, oxygen plays an essential role in conversion to CO_2. Oxygen acts as an acceptor of electrons during the mineralization reaction. Thus, as a reactant, O_2 consumed in the reaction corresponds to consumption of organic compounds, and therefore, O_2 consumed is proportional to the C.O.D concentration.[14] Thus, C.O.D removal was observed to be high.

Bacteriological examination

It was examined by identification of colonies formed on the prepared media (Agar-7) in petri dish. Mostly fecal coliform (blue colour), and Fecal Streptococci (dark & light red) micro organism were present in wastewater. After reaction with titania, no such colonies were found in the Petri dish and this shows its antimicrobial activity [Figure 6]. Furthermore, inactivation of fecal coliforms was achieved in 20 min by exposing water with titania. More recently, nano size titania was also reported to kill virus including poliovirus 1,[15] hepatitis B virus,[16] herpes simplex virus,[17] and MS2 bacteriophage.[18] The antibacterial activity of titania is due to the production of reactive oxygen species, hydroxyl free radical, and peroxide formed during photocatalytic reaction.[19]

Adsorption studies for prepared nano titania

Titania was diluted to 0.3% solution using distilled water and 10 ppm of MB dye was added in a beaker; then, it was kept under solar light. Its absorbance was noted to determine the photodegradation of MB dye at regular interval of time as shown in Figure 7.

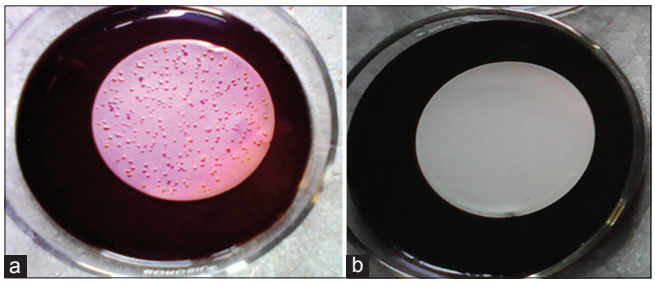


Figure 6: The bacterial examination of sewage water, (a) treatment without TiO_2, (b) treatment with TiO_2

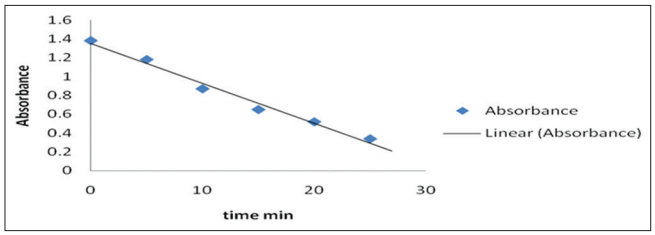


Figure 7: Adsorption of titania with methylene blue dye

The photodegradation of MB dye under visible light should be followed sensitized photocatalyst mechanism.[20] The process involves excitation of MB dye molecules by visible light and electron was transferred from excited dye molecules to the conduction band of semiconductor TiO_2, resulting in formation of unstable dye cation radical and in parallel an active species on the semiconductor surface that attacks to destabilize dye molecule. The decoloring kinetics of MB under solar light in the presence of TiO_2 was faster than in under UV light which was explained by this self-sensitization mechanism.[21] By oversight I have given the citation number as.[20]

Adsorption process using activated WA outer shell:

- The experiment was carried out by the batch adsorption method.

Effect of adsorbent

A different amount of activated carbon was taken in a 250 ml conical flask with 10 ppm concentration of MB dye solution. In each flask, 0.2, 0.4, 0.6, 0.8, and 1 mg of activated carbon along with 100 ml of MB dye solution were kept in a shaker for 60 min. The absorption at 680 nm was determined using UV spectrophotometer. There was an increase in adsorption with increase in activated carbon due to increase in their surface area [Figure 8], but after the optimum amount (0.6 mg), there is no removal of dye.

Effect of concentration of dye

100 ml was taken in five different conical flasks having various concentrations of 100, 150, 200, 250, and 300 ppm, and 0.6 mg of WA was added. It was kept in shaker of 150 rpm for 1 h. At low concentration of MB dye, maximum of dye

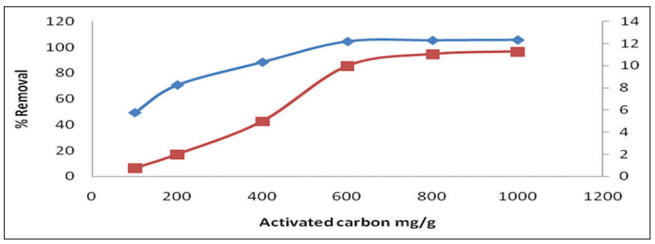


Figure 8: Effect of adsorbent. Volume of methylene blue = 100 ml, time = 60 min, T = 301 K, stirring speed = 150 rpm

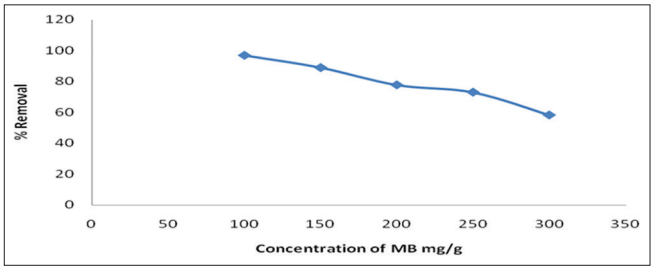


Figure 9: Effect of concentration of dye. Weight of activated carbon = 0.6 mg, time = 60 min, T = 301 K, stirring speed = 150 rpm, volume = 100 ml

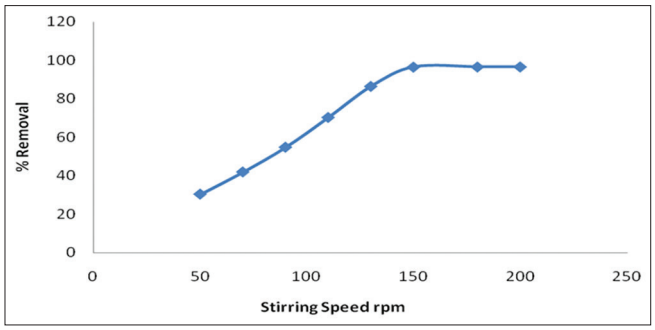


Figure 10: Effect of stirring speed. Weight of activated carbon = 0.6 mg, time = 60 min, T = 301 K, volume = 100 ml

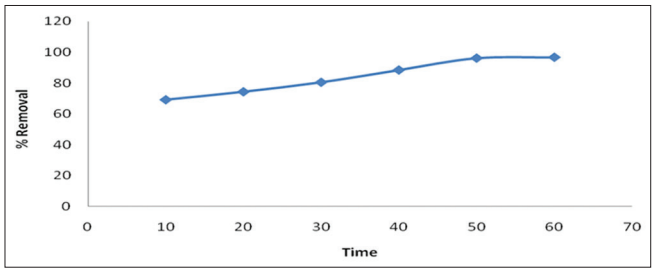


Figure 11: Effect of time. Volume of methylene blue = 100 m, stirring speed = 150 rpm, T = 301 K, weight of activated carbon = 0.6 mg

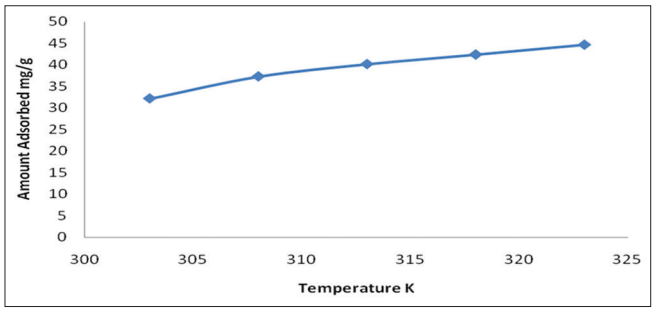


Figure 12: Effect of temperature, volume = 100 ml, time = 60 min, stirring speed = 150 rpm, weight of activated carbon = 6 mg

removal occurs, whereas increase in concentration of MB dye, there found to be minimum percentage of dye removal. This shows that concentration of adsorbent dosage was fixed with the adsorption sites to the low dye concentration, hence with no adsorption occurs with higher concentration of dye [Figure 9].

Effect of stirring speed

The stirring speed varied from 50 to 200 rpm, but after 150 rpm, percentage removal of MB remains constant [Figure 10].

Effect of time

100 ml of MB and 0.6 mg of WA were taken in a conical flask with stirring speed of 150 rpm, the percentage adsorption was determined at regular interval of time, and at certain time reaches, there was a steady equilibrium. The amount of adsorption (q_e) increases from 8.653 to 12.14 mg/g with

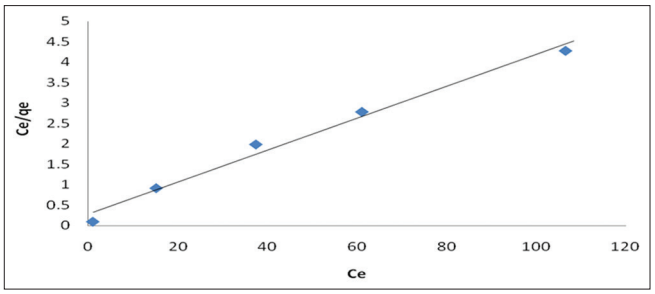


Figure 13: Langmuir isotherm of methylene blue on wood apple. Weight of activated carbon = 0.6 mg, volume = 100 ml, time = 60 min, T = 301 K, stirring speed = 150 rpm

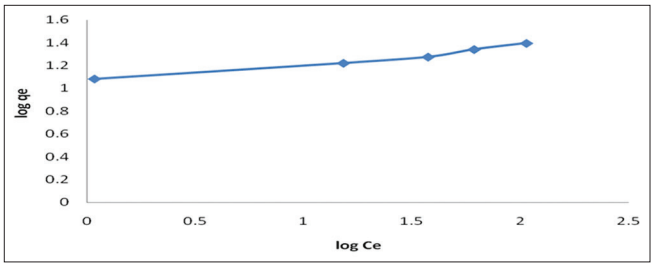


Figure 14: Freundlich isotherm of methylene blue on wood apple

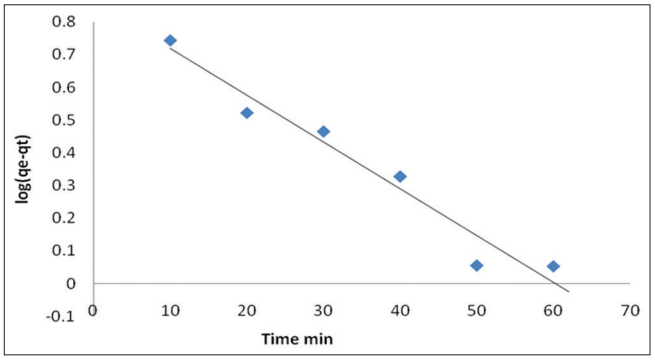


Figure 15: Pseudo first-order kinetics of methylene blue on wood apple

increase in time. From this, it was observed that WA prepared is efficient to absorb MB dye [Figure 11].

Effect of temperature

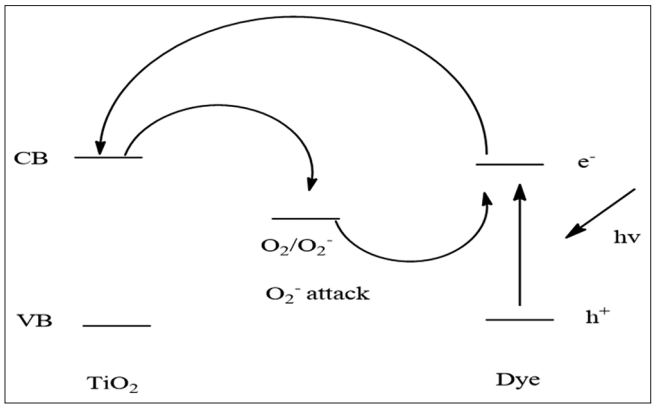
The adsorption process is temperature dependent. As the temperature increases, the adsorption capacity also increases [Figure 12].

ADSORPTION ISOTHERM

It explains about the distribution between the phases when the adsorption process reaches equilibrium state. The Langmuir and Freundlich models were the most common isotherm used to find experimental sorption.

Langmuir isotherm

Langmuir model was assumed that, at a maximum coverage, there is only a monomolecular layer on the surface. Langmuir isotherm is expressed as,



Graph 1: Photocatalysis mechanism between TiO_2 and MB dye

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

Where K_L is Langmuir constant (L/mg) related to the affinity of binding sites and the free energy, q_e is dye concentration at equilibrium onto biosorbent (mg/g), c_e is dye concentration at equilibrium in solution, and q_m is dye concentration when monolayer forms on biosorbent (mg/g). The values of q_m and b were calculated by plotting the graph between C_e/q_e and C_e. Plot of C_e/q_e versus C_e is linear [Figure 13]. Langmuir isotherm can be expressed by equilibrium parameter R_L.

$$R_L = 1/1+bC_0$$

R_L value indicates whether adsorption is favorable or unfavorable. R_L values between 0 and 1 indicate that it is favorable.

Freundlich Isotherm

Freundlich equation for heterogeneous surface is given by

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

Where q_e is amount of adsorption, K_F is Freundlich constant related to sorption capacity, and 1/n is intensity of adsorption.

This gives the exponential distribution of activated sites and their energies. The Freundlich exponents K_F and 1/n can be determined by the linear plot of log q_e versus log C_e which is shown in Figure 14, and the slope 1/n ranging between 0 and 1 shows that adsorption becomes heterogeneous as its value gets closer to zero.[22]

Freundlich constant		Langmuir constant	
K_F	1/n	q_m (mg/g)	b
10.17	0.19	32.14	0.863

Kinetics study

The adsorption kinetics of MB dye is analyzed using Lagergren first-order rate equation.

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

From graph the plot of log $(q_e - q_t)$ versus time for pseudo first-order model, kinetic constant was calculated from slope and intercept, which was correlated with experimental data, indicating that pseudo first order is followed [Figure 15].

Amount adsorbed by WA outer shell composite in a pseudo first order.

Experimental value (q_e) = 12.14 mg/g

Calculated value

From graph (q_e) = 13.05 mg/g

$K_1 = 0.37 \times 10^{-3}$/min

CONCLUSION

The main challenge of using TiO_2 in wastewater treatment could be the development for more efficient and low cost materials to promote sufficient treatment. The prepared nano titania has a higher removal efficiency for organic pollutant when compared with the bioadsorbent WA outer shell composite. It uses renewable sources for the removal of contamination of water. It has antimicrobial activity and high potential of dye removal. All these features expose that nano titania is suitable for the conversion of domestic, commercial, and industrial wastewater into agricultural purposes.

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Cite this article: Vijayalakshmi T, Elango G, Guhanathan S. Economical and Eco-friendly Wastewater Treatment Using Anatase TiO2. Asian J Appl Res 2018;4(12):1-8

Source of Support: Nil, Conflict of Interest: None declared.