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RESEARCH ARTICLE

Comparative analysis on the photocatalytic activity of titania and silica nanoparticles using dye discoloration and contact angle test

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Abstract

This work was done in order to determine the efficiency of TiO_2 and SiO_2 nanoparticles for photocatalytic activity by using contact angle measurements and dye degradation tests. Covering the mechanism of photocatalysis, which means the promotion of a chemical reaction through the aid of light, the focus here is to apply photocatalysis for the elimination of organic pollutants in water, which is now finding more uses in environmental regeneration. Despite TiO_2-and SiO_2-based nanoparticles may be attractive photocatalysts, there are few reports comparing their efficiency. Both samples were prepared using the sol-gel method; their morphological studies were carried out using a scanning electron microscope attached to an energy dispersion X-ray system. The photocatalytic tests were performed using UV light which was used in the degradation of Rhodamine B and Methylene Blue dyes and the reaction kinetics and efficiency of the photocatalytic process were determined with the help of a UV-visible spectrophotometer. Moreover, the determined hysteresis contact angle described the extent of hydrophilicity or hydrophobicity that resulted from nanoparticles affecting the photocatalytic behavior. As for photocatalytic performance, TiO_2 nanoparticles provided more reactive area and a stronger capacity to generate electron holes when exposed to UV light than SiO_2 so that TiO_2 showed higher photocatalytic activity. The surfaces of TiO_2 NSs coated with a thin layer of TiO_2 showed higher hydrophilicity that enhanced the photocatalytic properties. Not only does this work extend the knowledge on the enhanced photocatalytic performance of TiO_2 in environmental applications, but it also offers valuable guidelines for further research on nanoparticle-mediated photocatalytic applications, by focusing on the connection between surface wettability and photocatalytic efficiency that seems to be underemphasized.

Keywords: Titanium dioxide, Silica dioxide, Photocatalytic activity, Sol-gel analysis, Surface wettability, Contact angle analysis.

Introduction

The amount of industrial waste and toxic pollutants increases day by day which makes the need for unparalleled technology to remedy the damages done increasingly important. In these techniques, photocatalysis has emerged as one of the most effective methods to remove organic toxins from water bodies (Ajith & Rajamani, 2021). This occurs when the energy derived from light, especially ultraviolet (UV) light is used to activate the breakdown of poisonous substances into harmless ones. Photocatalysis has been found to be very efficient in cleaning up the environment and thus, has become a key innovative technology in sustainable development as noted by Ayyar and others 2018(Tahir *et al.*, 2020). Titanium dioxide (TiO_2) and silicon dioxide (SiO_2) nanoparticles have been found to have distinguishable structural and electronic proprieties for photocatalysis according to Ibhadon and Fitzpatrick, 2013; Kang and others 2019. TiO_2 is known for its photocatalytic activity and stability and initiates the processes of generation of electron-hole pairs under UV light that affects the degradation of numerous organic compounds (Broda, Sobczak, & Skumin, 2019). On the other hand, SiO_2, although being less used in photocatalytic systems, has remarkable features like high thermal stability and chemical inactivity, while its photocatalytic activity

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could be improved by the necessary modifications of surface (Nekrashevich & Gritsenko, 2014). Thus, a comparison of the above-mentioned nanoparticles in similar conditions would help determine their photocatalytic efficiency and mechanism. This way of synthesis was chosen because a sol-gel method is effective in obtaining high-purity and uniform particles of the nanoparticles (Filatov *et al.*, 2009). The synthesized nanoparticles with regards to their shape and elemental composition are analyzed using scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) as described by Kim *et al.* (2008) and Noah & Ndangili (2021). Photocatalytic evaluation is done through the degradation of dye and contact angle measurement as discussed by Wang *et al.* (2018). Methylene blue and rhodamine B are the chemicals utilized for dye degradation tests that directly indicate photocatalytic activity (Araújo *et al.*, 2020; Skorb *et al.*, 2007). Meanwhile, the contact angle hysteresis measurements give a relative estimate by determining the wettability of the surface nanoparticles of the photocatalyst that affects the photocatalyst pollutants interaction (Asmatulu *et al.*, 2010). For instance, in the synthesis of anatase phase TiO_2 where it is seen that it has high photocatalytic activity as against to rutile phase because of more surface area of anatase and effective separation between electron and hole (Sun *et al.*, 2018).

Doping with non-metal elements, introducing vacancies, and the formation of heterojunctions that enable TiO_2 to absorb light in the visible light range and with a higher quantum efficiency have been sought after (Li *et al.* 2022). Another method that has been used to control light intensity and rate of photocatalytic reaction is through the integration of PC with photocatalytic materials. It should be understood that the relative speed of the group of light waves can be decreased in the material which will ultimately lead to increased light-matter interaction and improved photocatalytic activity (Likodimos, 2018). The PCs in terms of three-dimensional photonic crystals and one-dimensional Bragg mirrors have been further used to enhance the photocatalytic activity of TiO_2 by trapping light in the active catalytic parts (Raja-Mogan *et al.*, 2020). This paper seeks to extend such thinking by developing a comparative discussion of TiO_2 and SiO_2 nanoparticles conducted under strictly controlled laboratory conditions. In this case, through creating small and excellent nanoparticles and utilizing various analytical methods, this study aims to understand the relationship between the characteristics of nanoparticles and photocatalytic activity. Among the expected outcomes of the work, the higher understanding of the processes that occur during photocatalysis with the help of nanoparticles and the possibility of designing more effective materials to be used for the clean-up of the environment will be of great value.

Materials and Methods

To achieve high uniform and non-encumbered Silica dioxide nanoparticles, the sol-gel technique is employed to facilitate the production of nanoparticles. Esters: 25 mL of ethanol and 2. 8 mL of acetic acid are mixed into the reaction mixture as it is normal to the temperature, stirring the reaction mixture with the help of a magnetic stirrer at a frequency of 700 r/min for 10 minutes. It requires about ten minutes to heat up to boil to produce ethyl acetate sol and make the water molecules evaporate. It is then converted to a gel using a molecular precursor methyltrimethoxysilane is used (Broda *et al.*, 2019). The deionized water is equivalent to 12 mL which was transferred to a container containing 6 mL of methyltri methoxysilane solution then the two solutions are mixed while stirring at room temperature for about 3 minutes. A dropwise addition of 2 mL of the precursor to 10 mL of ethyl acetate is done with constant stirring for 4 hours. Nanoparticles of fine silica dioxide (SiO_2) are obtained after drying the gel and calcination at a temperature of 400°C in a high-temperature hot air oven.

Sol-gel synthesis of titanium oxide nanoparticles was accomplished using the ethanol solvent, spherical hydrochloric catalyst, and titanium tetra iso propoxide (Filatov *et al.*, 2019) as the precursor. First of all titanium tetra-isopropoxide sol was synthesized by dissolving titanium tetra-isopropoxide with 5 mL HCl and 15 mL ethanol and stirring the mixture at room temperature for 30 minutes. To this, 10 mL of deionized water was then incorporated into the above mixture which was further stirred at room temperature for 2 hours. Finally, the solution was dried followed by calcination at 120°C for 1-hour to produce good TiO_2 nanoparticles.

Results and Discussion

Taken under scanning electron microscopy and engaged in energy dispersive X-ray analysis (EDAX analysis) to ensure the size and formation of Silica dioxide nanoparticles and titanium dioxide nanoparticles. Moreover, the percentage of dye removal efficiency of rhodamine B dye and methylene blue is determined from time to time by using the UV-visible spectrophotometer to testify to the photocatalytic activity. In this work, techniques such as the contact angle were used to determine the fluid wettability of the developed nanoparticles.

Scanning Electron Microscopy with EDAX Analysis

An SEM-EDAX analysis of the synthesized SiO_2 and TiO_2 nanoparticles was done using a VEGA 3 TESCAN scanning electron microscope at Anna University, Chennai, Tamil Nadu, India and the morphological and compositional features were revealed. This analysis at higher resolution was intended to establish the size, shape, and composition of elements that are essential while determining the photocatalytic properties of the prepared nanoparticles.

SEM Analysis of SiO_2 Nanoparticles

As shown in Figures 1 and 2, the main rod-shaped tetragonal SiO_2 nanoparticles' average size is about 68.7 nm. This is so due to the size variability of the nanoparticles observed which are attributed to chemical dynamics during the synthesis process with acetic acid and the precursor, tetraethyl orthosilicate (TEOS). The sol stability and the rates of hydrolysis and condensation reactions are affected by these chemicals and they give rise to different particle sizes (Nekrashevich & Gritsenko, 2014). The samples also point to additional variance largely due to the aggregation seen in these samples, and hence optimization of synthesis parameters is necessary to reduce particle size variance.

Elemental Composition by EDAX

Details about the elemental composition of the nanoparticles were given by EDAX. Results plotted in Figure 3 and presented in Table 1 indicate that the SiO_2 nanoparticles fit strongly into a representative carbon, oxygen, and silica analysis depicted in Figure 3 and Table 1. Good interaction of the electron beam with the sample is essential to avoid damage to the sample, which often has high carbon content (of which carbon coating is used in SEM analysis). Synthesized nanoparticles exhibit stable physicochemical properties and are thus suitable for photocatalytic applications based on the high purity as indicated by the measured weight percentage of silica and oxygen at 47.82 and 29.15% respectively.

TiO_2 Nanoparticles SEM Analysis

The TiO_2 nanoparticles have an average size of 69.2 nm, as depicted in Figure 4. The determination of nanoparticle size is consistent, which indicates a controlled synthesis process that is needed for reproducible photocatalytic activity. Figure 5 shows the elemental breakdown which has a primary composition of titanium with a purity level of 55.07% and oxygen with a purity level of 44.93%. As is typical for TiO_2, these proportions validate the successful synthesis of the nanoparticles with no significant impurity inclusion necessary to achieve maximum photocatalytic efficiency.

Photocatalytic Activity Implication

Different surface morphology and elemental purity of SiO_2 and TiO_2 nanoparticles are proved critical for their photocatalytic activity. The photocatalytic activity is influenced by these factors because they change the adsorption of light and reactive species on the surface; among which the surface area, particle size and shape largely determine the photocatalytic activity. Likewise, the materials are highly pure, with little corrupting influence from low-quality impurities in the photocatalytic reaction. It is therefore important to hypothesize these morphological and compositional characteristics for optimizing the nanoparticles for environmental remediation applications as in the degradation of pollutants such as rhodamine B and methylene blue dyes.

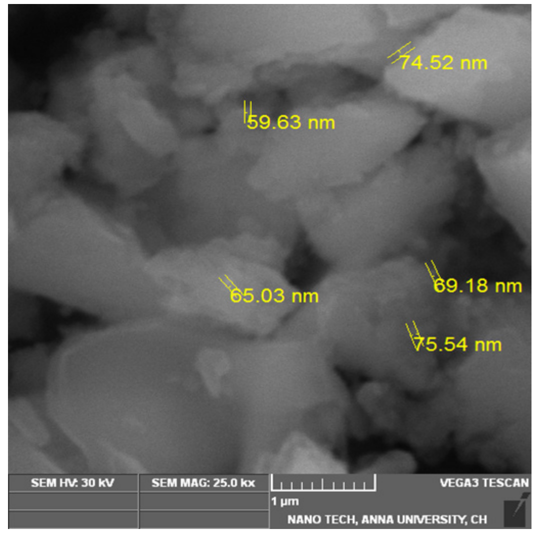


Figure 1: SEM image of Silica dioxide Nanoparticles at 1 μm

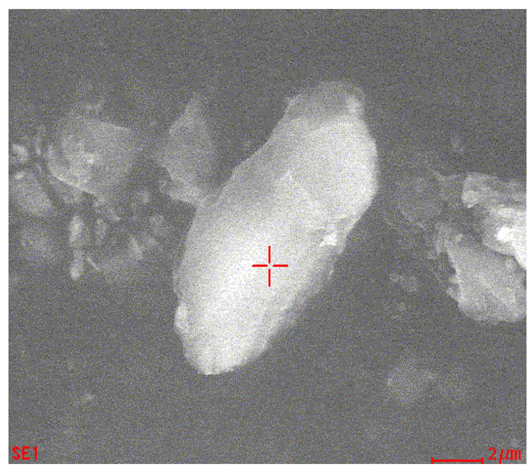


Figure 2: SEM image of rod-like silica dioxide nanoparticles at 2 μm

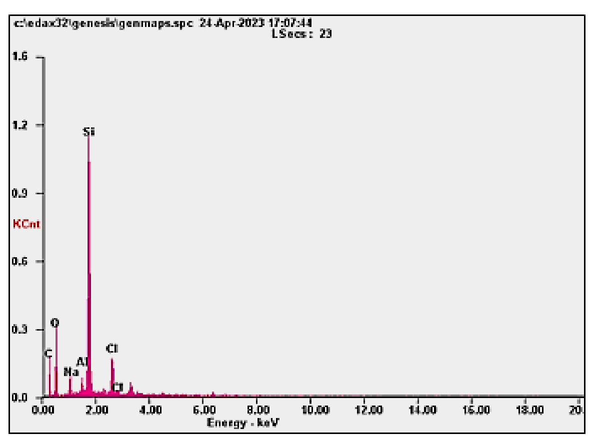


Figure 3: EDAX graph of silica dioxide nanoparticles

Table 1: EDAX weight percentage of silica dioxide nanoparticles

Element	*Wt%*	*At%*
CK	15.91	09.51
OK	29.15	27.90
NaK	02.84	01.89
AlK	01.01	00.57
SiK	47.82	58.71
CiK	03.27	01.41
Matrix	Correction	ZAF

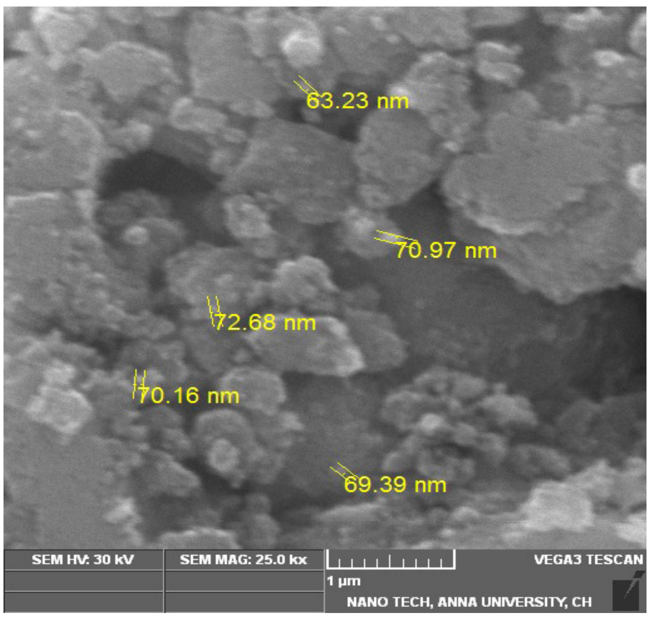
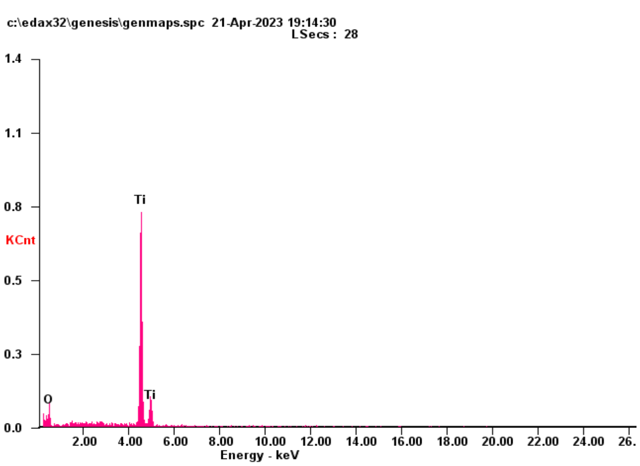


Figure 4: SEM images of titanium dioxide nanoparticles



Element	*Wt%*	*At%*
OK	44.93	70.96
TiK	55.07	29.04
Matrix	Correction	ZAF

Figure 5: EDAX image of titanium dioxide nanoparticles

In conclusion, the SEM and EDAX analyses have provided essential confirmations of the nanoparticles' characteristics, setting a solid foundation for further investigations into their photocatalytic activities. These findings are not only critical for understanding the inherent properties of the nanoparticles but also for tailoring their synthesis for specific environmental applications.

Analysis of Photo catalytic activity using Rhodamine B dye

This paper focuses on the elimination of rhodamine B dye employing photocatalysts including silica dioxide (SiO_2) and titanium dioxide (TiO_2) nanoparticles with a view to dissecting their efficiency in environmental management. In this experiment, 1 g each of SiO_2 and TiO_2 nanoparticles were dispersed in different solutions that contained 0.1 mL rhodamine B dye in 3 mL distilled water. Also, a sample with an equal mixture of SiO_2 and TiO_2 having 20% of its weight in nanoparticle form was synthesized under the same procedure. The above samples were subjected to partial sunlight and absorbance was recorded at intervals of 0, 15, 30 and 45 minutes in a UV-visible spectrophotometer in Anna University, Chennai.

SiO_2 Nanoparticles

The spectra analysis (Figure 6) showed a lack of absorbance peak for plain SiO_2 nanoparticles which justified the material's large band gap of around 9 eV; this material is transparent to UV light and not efficient in UV radiation required for photocatalytic process (Halder & Purkayastha, 2022). With the introduction of the dye, the first peak appeared at 544 nm which shows that rhodamine B was introduced into the system. This gradually declined over time where at 45 minutes there was a clear though slow degradation of the dye by SiO_2 which might be attributed to the surface properties of the nanoparticles and not electronic properties.

TiO_2 Nanoparticles

However, TiO_2 nanoparticles had sharper absorption, giving a diverse peak between 300 and 320 nm which proved to be the best when exposed to UV light suitable for photocatalytic activity (Figure 7). When combined with Rhodamine B, the absorbance peak at 531 nm decreased from 0.75 to 0.12 within 45 minutes after interaction with the photocatalyst showing high efficiency in the photocatalytic degradation of the dye (Li *et al.*, 2021). This fast decline is an indication that TiO_2 has enhanced photocatalytic features similar to the production of reactive oxygen species that act as reducing agents and decompose organic substances.

Photocatalytic Activity of SiO_2, TiO_2 and Mixed Nanoparticle Solution

The photocatalytic activity of the two prepared samples namely SiO_2 and TiO_2, as well as the mixed nanoparticle

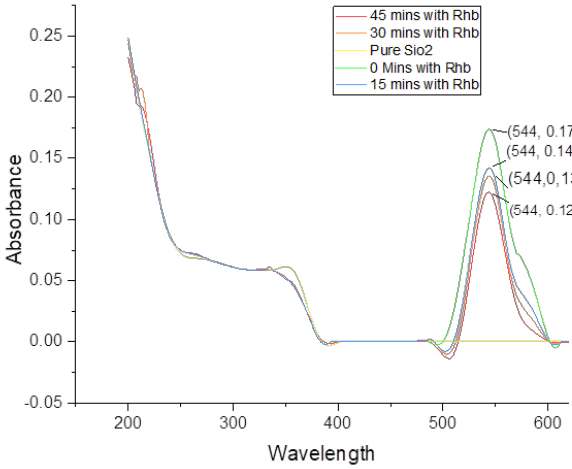


Figure 6: Photocatalytic activity SiO_2 nanoparticles with rhodamine B dye

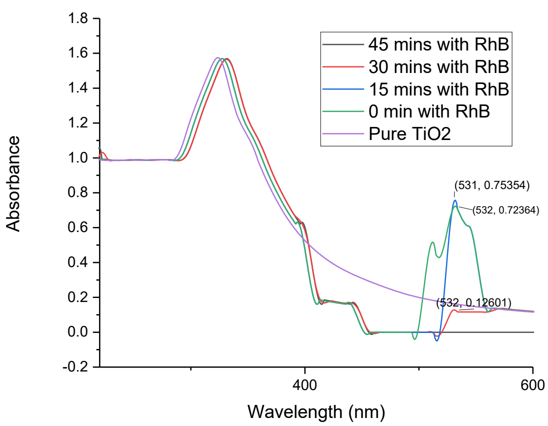


Figure 7: Photocatalytic activity TiO_2 nanoparticles with rhodamine B dye

solution, was also studied (Figure 8). To begin with, the absorbance peak of 531 nm was moderate, but gradually faded as the time elapsed. This result meant that the mix of the two could not possess as poor a degrading capability as the wholly untalented TiO_2 though demonstrated better activity than wholly untalented SiO_2; this showed signs that the impact of TiO_2 is capable of improving on the photocatalytic ability of mix where else the phase of mixed catalytic impact will regime to provoke the generation of electrons and holes respectively that augment over photocatalytic activity.

These results give clear evidence about the efficiency of SiO_2 and TiO_2 nanoparticles in the photocatalytic degradation of organic dyes and it is clearly depicted that TiO_2 has high photocatalytic efficiency. This is in line with the ability to apply TiO_2 on environmental clean-up,

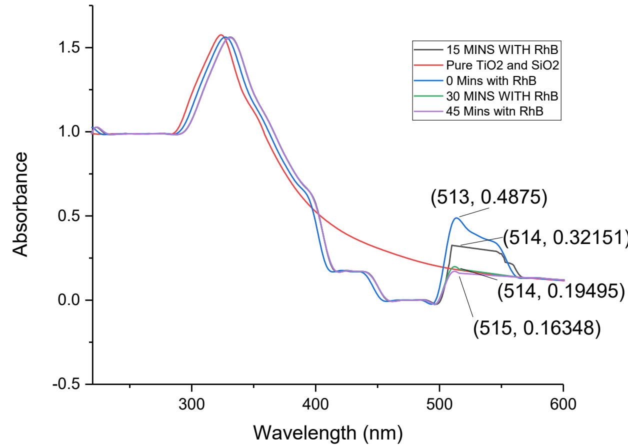


Figure 8: Photocatalytic activity $TiO_2$and SiO_2 nanoparticles with rhodamine B dye

especially in water treatment technologies that require high rates of pollutant degradation. The combination of the two nanoparticles can offer a feasible approach for mediating the cost and the efficiency of photocatalytic systems based on the features of both materials in a composite system.

Analysis of Photocatalytic Activity using Methylene Blue Dye

This section analyses the photocatalytic performance of SiO_2 and TiO_2 nanoparticles in the degradation of methylene blue dye employed as a model pollutant in many photocatalytic experiments. The experiments were performed by adding 1 g of each type of nanoparticle and 0.1 mL of methylene blue dye that was prepared in 3 mL of distilled water. Similarly, a 1:1 suspension of SiO_2 and TiO_2 was also subjected to the experiment. The solutions were exposed to partial sunlight and their photocatalytic activity was determined at different time intervals of 0, 15, 30 and 45 minutes using a UV-visible spectrophotometer from Anna University, Chennai.

The UV-visible adsorption spectra of the prepared methylene blue/TiO_2 nanoparticles system are shown in Figure 9 below; As it is seen in the figure, TiO_2 nanoparticles have its initial absorption onset between 300 to 320 nm, which corresponds to the UV region that is indispensable in photocatalytic process. This high initial absorbance value of 1.31 at 439 nm indicates that the dye that has been used is Methylene Blue when mixed with it. Over the period of 45 minutes, the absorbance gradually decreased, demonstrating a rapid degradation of the dye; values of absorbance were recorded to be 1.1, 0.7, and 0.3 for the TiO_2 nanoparticles, thus exemplifying excellent photocatalytic degradation characteristics [26]. This is consistent with the degradation process of the dye in water, especially the reaction involving the photogenerated reactive oxygen species in the presence of anatase TiO_2 by UV light irradiation.

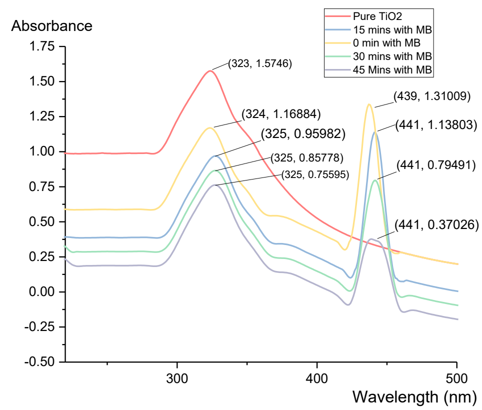


Figure 9: Photocatalytic activity TiO_2 nanoparticles with methylene blue dye

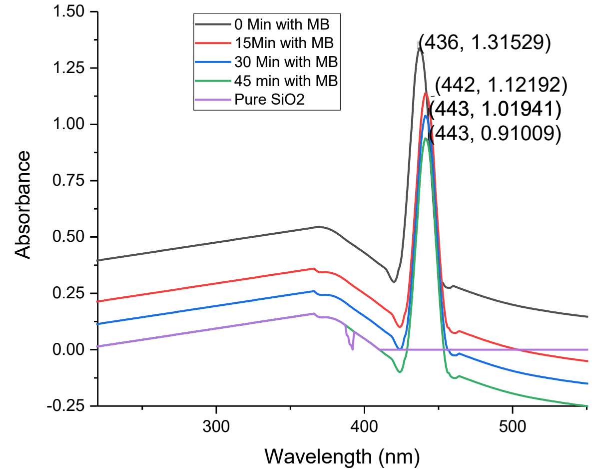


Figure 10: Photocatalytic activity SiO_2 nanoparticles with methylene blue dye

The synthesized SiO2 nanoparticles did not show any kind of absorbance in the UV-visible spectrum showing that they have less photocatalytic activity under these circumstances (Figure 10). A high initial absorbance of the dye at the wavelength of 436 nm was measured, 1.31 and then the absorbance gradually reduced to 1.12, 1.09 and 0.91 at further time intervals. This decrease in dye concentration signifies much slower incorporation of the photocatalyst and dye in the experiment, which would accredit the low surface reactivity and indisputable photocatalytic characteristics of SiO_2 nanoparticles under the current experimental setting.

Merged Solution of TiO_2 and SiO_2 Nanoparticles

When the two nanoparticles were combined, a different behavior was observed regarding photocatalytic activity (Figure 11). These were recorded at 531 nm and initially, they range as 0.48 and after each time interval of 15 minutes reading was taken as 0.32, 0.19 and 0.16. The latter proves that the mixture is not as effective as pure TiO_2 but the photocatalytic activity of the composite is higher than that of pure SiO_2. This implies that the TiO_2 may help in photocatalytic reactions of the mix and thereby increasing the degradation efficiency of the solution.

These results also confirm the fact that TiO_2 nanoparticles possess better photocatalytic activities for the degradation of methylene blue dye as compared to SiO_2. It is noteworthy to add that analysis of the obtained results has revealed the expediency of the use of mixed nanoparticle systems in the tasks of optimizing the costs and characteristics of practical application. The comparative study of the nanoparticle performance helps to draw an important conclusion about optimizing the photocatalytic systems for the degradation of water contaminated with synthetic dyes and other types of organic compounds.

Contact Angle Technique

The data of the contact angle gives information on the nature of the interaction of titanium dioxide (TiO_2) and

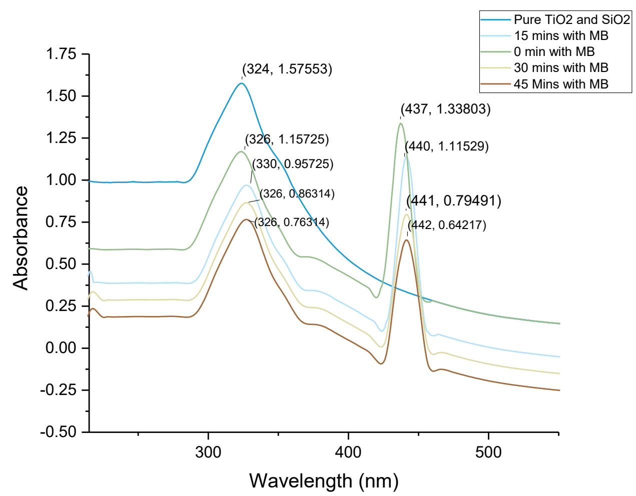


Figure 11: Photo catalytic activity $TiO_2$and SiO_2 nanoparticles with methylene blue dye

silica dioxide (SiO_2) nanoparticles with water and other substances. These properties directly determine the photocatalytic efficiency of the nanoparticles in relation to the interaction of the given pollutant molecules in aqueous solutions. The contact angle values for pure TiO_2 nanoparticles which were determined to be less than 90 degrees are clear evidence of the hydrophilic nature of the material as indicated in Figure 12. This is because the surface chemistry of TiO_2 favors water adsorption by the presence of hydroxyl groups on the surface of the material (Flores *et al.*, 2013). Such of hydrophilic surface exhibits higher rates of photocatalytic degradation because the hydration of water molecules and dissolved organic pollutants' adsorption is more favorable on the hydrophilic surface as compared to the hydrophobic one.

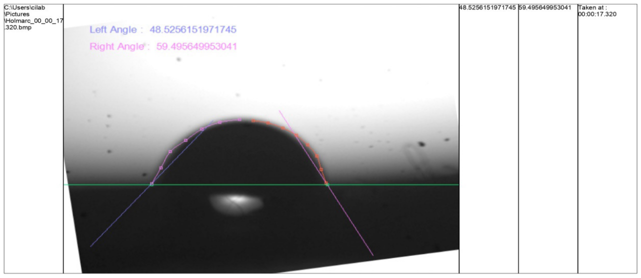


Figure 12: Contact angle of pure titanium dioxide nanoparticles

Other types of nanoparticles that were tested were silica dioxide; nanoparticles that displayed a hydrophobic character with contact angles above 90º as illustrated in Figure 13. It has been noted that the chemical nature of the SiO_2 and the method of preparing the surface of the material will determine the level of hydrophobicity through the contact point angle of the material surface to the water molecule. A non-selective photocatalytic surface is hydrophobic in nature, which may have its disadvantage in repelling water that is essential for aqueous-phase photocatalytic reactions, however can be of significant importance in repelling water or targeting non-polar pollutants.

Nanoparticle Comments on the Mixed Suspension of TiO_2 and SiO_2 Nanoparticles

It was observed that the contact angle of this interface had an intermediate value of both hydrophilic and hydrophobic characteristics as presented in Figure 14. This intermediate wetting property will mean that the mixture could at some point provide a middle ground by offering absorption of water and organic molecules and therefore may increase photocatalytic activity under the right circumstances.

UV Light Effectively

Suspension of all three forms of nanoparticles was exposed to UV light for 10 minutes, and evidence depicted in Figures 15, 16, and 17 showed that the contact angle increased slightly after exposure. This may be due to photochemical modification of the surfaces and the likelihood of creating new surface functional groups that affect their degree of hydrophobicity. Exposure to UV light also produces reactive species that alter surface chemistry and, consequently, alter the hydrophilic or hydrophobic properties of the nanoparticles.

These findings are rather important as they show that the surface properties of the nanoparticles – and these can be deduced from contact angle measurements – are the factors that play crucial roles in photocatalytic activity. Knowledge of these properties allows the synthesis and treatment of photocatalytic materials to be optimized for particular environmental conditions or applications.

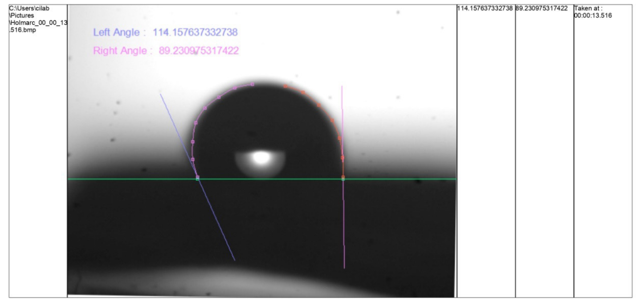


Figure 13: Contact angle of pure silica dioxide nanoparticles

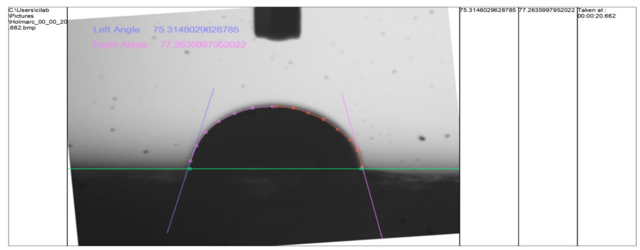


Figure 14: Contact angle of mixture of titanium dioxide and silica dioxide nanoparticles

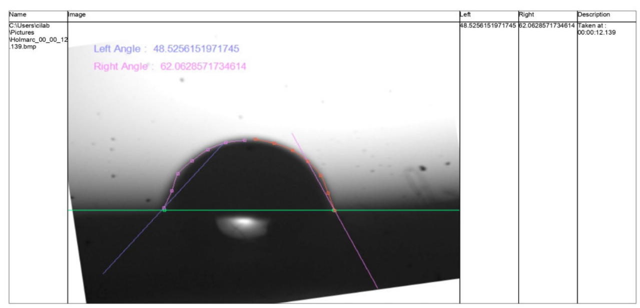


Figure 15: Contact angle of pure titanium dioxide nanoparticles after 10 minutes of exposure to UV light

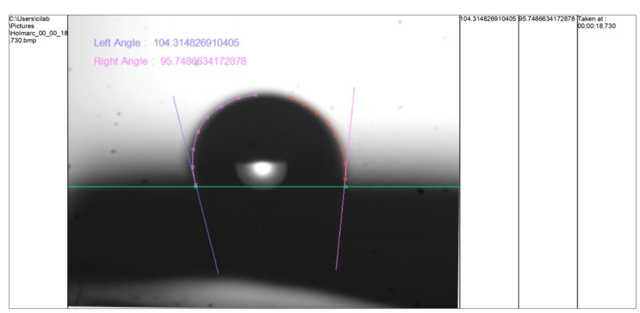


Figure 16: Contact angle of pure silica dioxide nanoparticles after 10 minutes of exposure to UV light

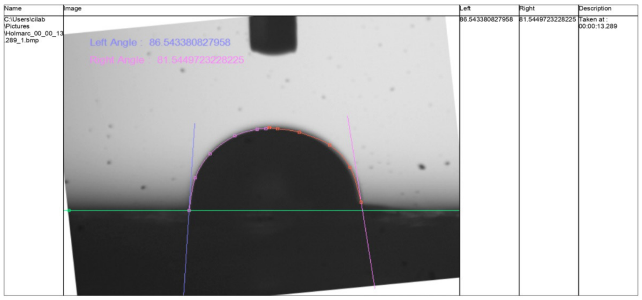


Figure 17: Contact angle of a mixture of titanium dioxide and silica dioxide nanoparticles after 10 minutes of exposure to UV light

Conclusion

Nanoparticles of silicon dioxide and titanium dioxide are effectively synthesized by the sol-gel method. The size and composition were verified using a scanning electron microscope with EDX technique. Photocatalytic efficiency was analyzed with rhodamine B dye, and methylene blue dye and was carefully monitored at different intervals of time using a UV-visible spectrophotometer. It is concluded that the absorbance of titanium dioxide nanoparticles degrades fast in various intervals of time and for silica dioxide nanoparticles there is no major change in absorbance value though it degrades slowly in various intervals of time. The absorbance value for the mixture of silica dioxide and titanium dioxide nanoparticles showcased midway degradation efficiency when compared with titanium dioxide and silica dioxide individually. The contact angle technique was used to analyze the surface wettability. It is seen that pure titanium dioxide nanoparticles are highly hydrophilic in nature and pure silica dioxide nanoparticles are highly hydrophobic. The mixture of silica dioxide and titanium dioxide nanoparticles showed a nearly hydrophobic nature. After ten minutes of exposure to UV light, there is a slight increase in contact angle which may be due to the surface modifications or aggregation in the sample. Hence the present research work confirms the enhanced photocatalytic efficiency of titanium dioxide nanoparticles using contact angle analysis and dye degradation technique.

Acknowledgments

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