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Research Article

Synthesis of SiO2 NPs from Sodium Bentonite and their Use in Photocatalytic Activity and Adsorption Applications

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ABSTRACT

Silica nanoparticles (SiO_2 NPs) are a material that is used in many areas and are obtained by various methods and attracts the attention of researchers with its many properties such as photocatalysis, adsorbent and non-toxicity. In this study, sodium bentonite was used as a raw material to synthesize silica nanoparticles. In the FTIR results of the prepared SiO_2 NPs, the Si-O-Si band indicating the formation of NPs is remarkable. The morphology structure was determined to be an agglomerated structure similar to a spherical form in SEM images. In the other part of the study, the photocatalytic degradation of methylene blue (MB) dye was investigated with SiO_2 NPs. The results revealed that 80% degradation was achieved in 60 min and the process complied with the second order reaction kinetics. The effect of MB in aqueous solution was investigated for its use as an adsorbent. After 60 minutes, 90% dye removal was achieved and it was determined that the process fit pseudo-second-order kinetic model (PSO).

1. Introduction

Nanotechnology is a multidisciplinary science with synthesis methods and application areas that emerged in the 21st century [1]. The term nanoparticle represents materials with a size between 1-100 nm and at least one dimension. At these small sizes, particles change their properties due to the laws of quantum mechanics [2]. Nanotechnological developments are emerging in many application areas such as drug delivery, sensors, diagnostics, bioimaging, agriculture, food and water treatment [1, 3–5]. Nanoparticles of many metal and metal oxide elements are synthesized and attract the attention of researchers [4].

Silica (SiO_2) is a compound that is abundant and widespread throughout the world, including plants and grains [6]. Silica-based nanoparticles are important materials due to their easy preparation, high surface functionality, particle size adjustable during synthesis, and biocompatible properties [3]. Amorphous silica in particular is widespread in nature and is used in various applications [7]. According to research, silica nanoparticles are the 2nd most produced nano-sized material in the world. For this reason, research on the subject has diversified [6].

Bentonite is known as a clay that is a good source of silica. It contains aluminum and silica together. Acid and alkali exposures are used successively to try to separate these components from each other [8]. It can be divided into two depending on whether it contains Na and Ca ions. Sodium bentonite has a high water absorption capacity due to the Na ion it contains [9].

Water pollution is a problem that deeply affects human life. There are many methods for cleaning water in addition to membrane, adsorption,

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photodegradation, chemical oxidation, precipitation, osmosis and filtration [10, 11]. However, some of these methods take time and cause new waste materials to emerge [11]. The use of photocatalytic properties of nanoparticles for the removal of pollutants in water pollution treatment has attracted attention in recent years. Because the specific surface area is important at this point. A good photocatalyst is expected to have features such as being cheap, stable, inert and easily available [10]. Similarly, adsorption is an effective and low-cost system used in water treatment. It is basically a mass transfer process in which the solid phase in the aqueous solution is transferred to the sorbent surface [12].

Synthetic dyes are harmful chemicals used in a variety of applications such as paper, food, leather and pharmaceuticals. They can also be released uncontrolled as industrial waste. They have serious damage to the ecosystem [13]. Methylene blue (MB) is a cationic and blue colored dye known as methylthioninium chloride [14, 15]. It is generally used in the textile industry and other areas for dyeing purposes. MB, which has toxic properties, is carcinogenic, not biodegradable and has an aromatic ring structure. It is a harmful compound that can affect human health at many points [15, 16].

In this study, sodium bentonite was used as a silica precursor raw material to synthesize SiO_2 NPs. SiO_2 NPs was prepared by treating with NaOH and HCl. The chemical structure and morphological properties of the obtained nanoparticles were examined by Fourier Transmittance Infrared (FTIR), Energy dispersive X-ray (EDX) and Scanning Electron microscopy (SEM) analyses. SiO_2 NPs synthesized for use in water treatment were used in photodegradation and adsorption processes. For this purpose, cationic MB dye was selected as the model dye compound.

2. Material and Methods

2.1. Materials

Sodium bentonite, which was used as raw material to prepare NPs, was purchased from Carben. Sodium hydroxide pellets were supplied from Honeywell Fluka, hydrochloric acid (HCl, 37%) solution was supplied from Merck, MB was supplied from Isolab firm, and hydrogen peroxide (H_2O_2, %30) was supplied from Tekkim company.

2.2. Preparation of SiO_2 NPs

Sodium bentonite clay was used as a silica precursor in this study. The method used in our study was modified from those applied to fly ash and sodium bentonite in the literature [8, 17]. In the literature, the synthesis of sodium silicate by treating the raw material with NaOH and then treating sodium silicate with acid to obtain SiO_2 is also known as the sol-gel method [18]. 1 g of sodium bentonite was mixed with 8 M NaOH at 90 ºC for 90 min. After the mixture cooled, it was filtered with filter paper. 5 M HCl solution was added dropwise to the filtrate. This process was continued until a thin gel layer was formed and the pH was determined as 9.6 at this time. The temperature at this time was 30 ºC. The addition of HCl was stopped when the gel was formed. It was covered and left to age for 24 h. The next day, the suspension was filtered with filter paper and dried in an oven at 40 ºC for 24 h.

2.3. Photocatalytic activity

For photocatalytic degradation experiments, 50 ml of 10 mg/L MB solution was added to a 100 mL volumetric flask and a certain amount of synthesized photocatalyst was added to the solution and the mixture was stirred on a magnetic stirrer. Then, H_2O_2 was added to the suspension to provide oxidation and mixed. Before irradiation, it was stirred in the dark for 30 minutes to reach an adsorption/desorption equilibrium between the MB molecule and the catalyst. Then, the UV lamp (366 nm, UVA) lights were turned on and the solution was irradiated with UV light for 60 minutes, and during this time, samples were taken from the suspension every 15 minutes and absorbance was measured on a UV/vis spectrophotometer. The wavelength at which MB gives maximum absorbance is 664 nm. The equation for calculating the $\%\, degradation$ is given in Equation 1.

$$Degradation\,(\%) = (Co - C)/Co \times 100 \qquad (1)$$

C_o is the initial dye concentration (t = 0) and C is the dye concentration after irradiation for the selected time point.

Various kinetic models have been proposed in the literature to understand the degradation mechanism of azo dyes over time. The most commonly used models include first and second order kinetic models [19]. Photocatalytic degradation reactions are planned to be explained by first order reaction kinetics (Equation 2) and second order reaction kinetics (Equation 3). Here k_1v k_2 are the first and second order reaction kinetic constants, respectively. C_o is the concentration at the initial time and C_t is the concentration at time t. The unit of concentrations is mg/L. Time is indicated by t (min) [20, 21].

$$ln\,(C_t\,/C_o) = -k_1 t \quad (2)$$

$$1/C - 1/C_o = k_2 t \quad (3)$$

2.4. Adsorption process

The usability of SiO_2 NPs as an adsorbent was tested in the removal of MB solution from an aqueous solution. The initial dye concentration was determined as 10 mg/L and the aqueous solution volume (V, mL)) as 100 mL. Adsorbent (m, g) was added to the dye solution at its own pH and the mixture was provided. In order to examine the effect of the contact time, absorbance values of samples taken from the mixture at certain time intervals were determined in UV/vis spectroscopy at 664 nm. $Adsorption$ (%) and adsorption capacity (q_t) are given in Equations 4 and 5. Here m is known as the amount of adsorbent (g). The unit of adsorption capacity is also mg/g. Pseudo-second-order kinetic model (PSO) is presented in Equation 6. Here q_e (mg/g) is the equilibrium absorption capacity and k_2 is the model constant [22].

$$\%\text{Adsorption} = \frac{\text{Co}-\text{C}}{\text{Co}}\text{x}100 \quad (4)$$

$$q_t = \frac{(C_o - C_t).V}{m} \quad (5)$$

$$\frac{t}{qt} = \frac{1}{k_2.qe^2} + \frac{1}{qe}t \quad (6)$$

2.5. Characterization

The chemical structure of the SiO_2 NPs was investigated by Attenuated Total Reflection-Fourier Transform Infrared (ATR-FTIR, Bruker, Tensor II) spectroscopy in the wavelength range of 4000-400 cm^{-1}. Scanning electron microscope (SEM-EDX, Tescan Mira 3 XMU) was used to analyze the NPs formation in terms of size and shape. The elemental content of the structure was determined by Energy dispersive X-ray (EDX).

3. Results and Discussion

3.1. FTIR

The chemical structure of the synthesized SiO_2 NPs was analyzed by FTIR spectroscopy and is presented in Figure 1. The peaks on the spectrum represent the chemical structure of the SiO_2 NPs structure. The band appearing at 3395 cm^{-1} can be attributed to the stretching caused by the binding of water molecules and silanols [2]. This peak additionally represents the asymmetric stretching vibration of the Si-O-Si bond, indicating the formation of silica NPs [23]. The peak at 1641 cm^{-1} can be attributed to O-H bonding from water, the large peak at 1059 cm^{-1} to Si-O stretching, and the peak at 801 cm^{-1} to Si-O-Si symmetric stretching [24]. It has been reported that the region between 400 and 1200 cm^{-1} is the region representing silica. The peaks occurring at 440.97 cm^{-1} can be attributed to the vibration band of Si-O-Si and the peak at 708 cm^{-1} can be attributed to the symmetric stretching vibration of Si-O-Si [17]. The peaks of the nanoparticle are in agreement with the literature and demonstrate the successful synthesis of SiO_2 NPs.

3.2. SEM

The morphological structure of SiO_2 NPs was examined with SEM images shown in Figure 2. The particles show a structure similar to a

spherical form in different sizes in the range of 50-110 nm. It can be said that the nanoparticles

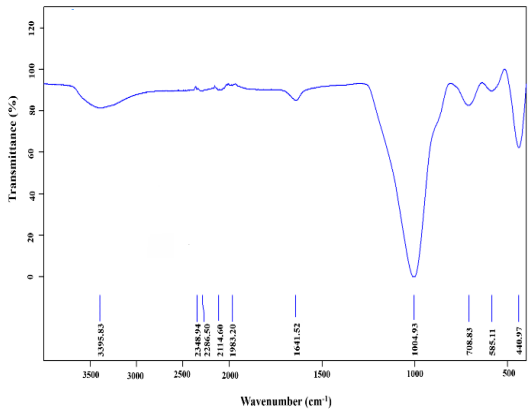

Figure 1. FTIR spectrum of SiO_2 NPs

are agglomerates [5]. According to the literature, these NPs with spherical shape, which are agglomerated, have a significant surface-to-volume ratio and active sites. These active sites increase the absorption of UV light and enable them to be a good photocatalyst [2].

The EDX data graph of SiO_2 NPs is given in Figure 3. The elemental structure of the nanoparticle is understood by weight and atomically. When the percentages are examined here, it is remarkable that there is more than twice the oxygen of silicon. This oxygen may also be excess oxygen coming from sodium bentonite or NaOH that does not react. However, the oxygen element is still at least twice the weight and atomic weight of the silicon element. In this case, it is thought that the synthesized nanoparticle may be SiO_2.

From the results, it is seen that there are elements such as Na, Cl, Al and C. It is thought that the reason for this is the impurities in the raw materials and the HCl, NaOH solutions used [23].

The possible reaction mechanism in SiO_2 NPs synthesis is given below [17, 25, 26]. Here, firstly (Equation 7) it can be assumed that silicates from sodium bentonite react with NaOH to form Na_2SiO_3. In Equation 8, silicic acid, which is the product of the HCl treatment process, appeared when the pH was 9.6 and the temperature was 30 ºC. According to the literature, this reaction is the basis of the sol-gel method. In Equation 9, it is seen that silicic acid can transform into Si-O-Si and silicon hydroxide species [17, 27].

$$SiO_2+2NaOH \rightarrow Na_2SiO_3+H_2O \quad (7)$$

$$Na_2SiO_3+H_2O+HCl \rightarrow Si(OH)_4 \text{ (nano silica)} + H_2O + 2NaCl \quad (8)$$

$$Si\text{-}OH+Si\text{-}OH \leftrightarrow -Si- + H_2O \quad (9)$$

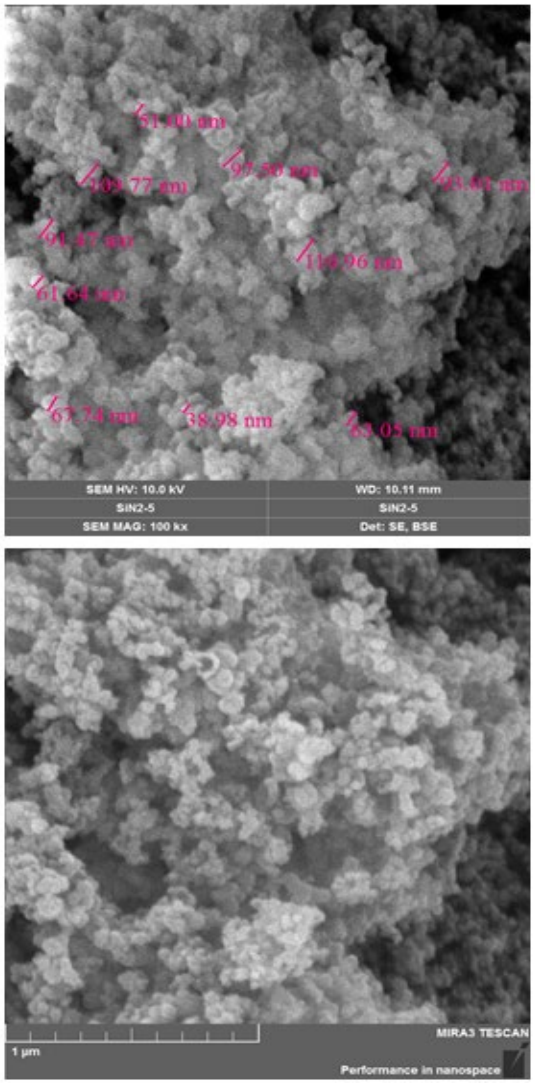

Figure 2. SEM images of SiO_2 NPs

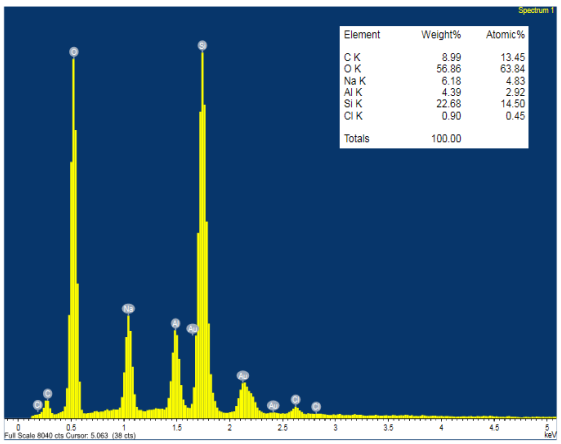

Figure 3. EDX data of SiO_2 NPs

3.3. Photocatalytic degradation

A calibration graph was obtained by plotting the MB solution prepared at different concentrations (0.1-12 mg/L) and the absorbance measurements read in UV/vis spectroscopy at 664 nm. The equation (y=0.1722x+0.1867) obtained from this graph was used to convert the absorbance values to MB concentration values.

The degradation process of MB dye using UV lamp was investigated against time using SiO_2 NPs. The reason for using H_2O_2 in the photodegradation process is to increase efficiency [4].

The amount of photocatalyst and the volume ratio of the dye solution were kept constant as 1 mg/mL. In Figure 4, the UV spectrum of the MB solution at t0 and t60 after the degradation process started is given. Thus, the effectiveness of SiO_2 NPs as a photocatalyst is revealed after 60 minutes. It is seen that the solution shows maximum absorbance at 664 nm as expected and the absorbance peak intensity decreases at the end of 60 min. It was determined that the dye underwent 80% degradation at the end of 60 min in the presence of SiO_2 NPs photocatalyst. According to the literature, photocatalyst properties may increase due to electron-hole pairs that may form on the surface of nanoparticles. This may be caused by oxygen defects in the SiO_2 NPs structure [2].

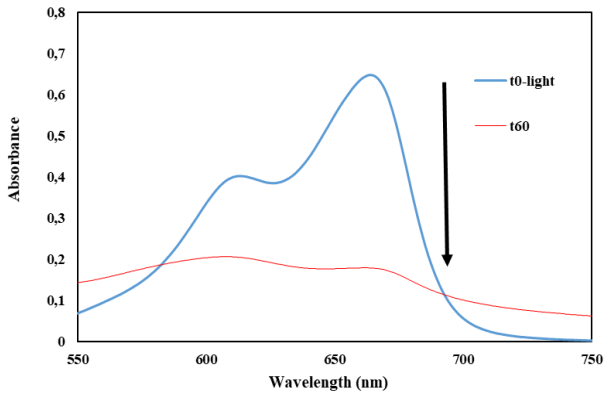

Figure 4. UV spectra of MB at time t0 and time t60

Figures 5 and 6 show the graphs of the first and second order kinetic models. For this purpose, samples were taken from the dye solution at 0, 15, 30, 45 and 60 min for 60 min and their concentrations were calculated. It is seen that the degradation process is compatible with the second order kinetic model. The kinetic model constants k_1 were determined as 0.0379 min^{-1} and k_2 as 0.306 L/mg.min.

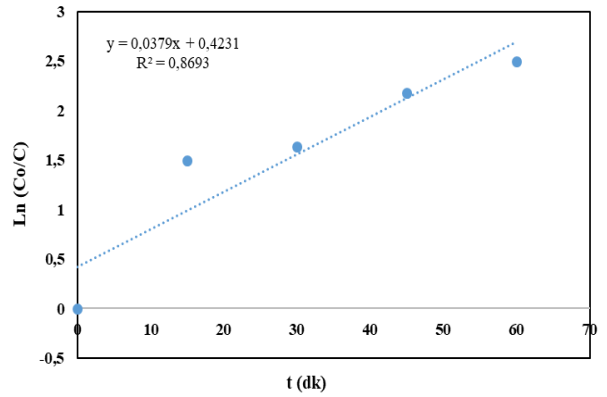

Figure 5. Graph of first order reaction kinetic model

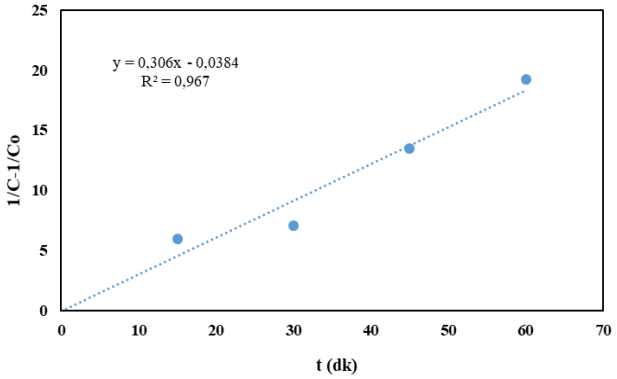

Figure 6. Graph of second order reaction kinetic model

It is well known that crystalline silica has a significant band gap. Therefore, it is difficult to

photoexcite in various UVA, UVB and visible radiation regions. However, structural defects facilitate photoexcitation [28].

Figure 7 shows the degradation rate of MB against time. According to this data, the degradation of MB occurred rapidly after the first 15 minutes. Then, it is seen that the degradation rate slowed down. This result shows that SiO_2 NPs synthesized from sodium bentonite can be used as a good photocatalyst and affects the degradation process in a short time. Among these, SiO_2 NPs is thought to be a photocatalyst that can accelerate photocatalytic degradation and can be easily synthesized. Table 1 shows the comparison of SiO_2 NPs as a photocatalyst with the literature. When Table 1 is examined, it is seen that different dyes or drugs and different working times are used. In addition, the UV lamps and systems used differ. This affects the degradation efficiency. In the light of all these data, it can be said that the SiO_2 we used in our study is a good photocatalyst despite the process not being optimized and reaches a high degradation efficiency in a short time.

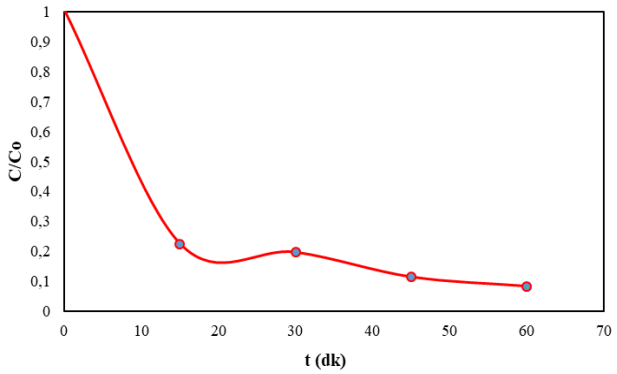

Figure 7. Degradation (%) graph

Table 1. Photocatalysts used in literature and process information

Photocatalyst	Sorbate	Time (min)	Degradation (%)	References
SiO_2-TiO_2	MB	30	85	[29]
Ag/Silica nanocomposite	Ciprofloxacin	180	98	[10]
SiO_2 NPs	Methyl orange	90	95	[2]
SiO_2 NPs	MB	90	98	[2]
SiO_2 NPs	MB	60	80	This study

3.4. Adsorption process

MB from an aqueous solution was removed by SiO_2 NPs synthesized from sodium silicate during the course of 60 minutes of contact time. The effect of contact time on adsorption (%) and adsorption capacity (q_t) is presented in Figure 8. It is seen that the dye adsorbed very quickly onto the adsorbent in the first 15 min. Afterwards, there was no significant change in adsorption and adsorption capacity. At the end of the adsorption process, 90% dye removal and 9.68 mg/g adsorption capacity were achieved.

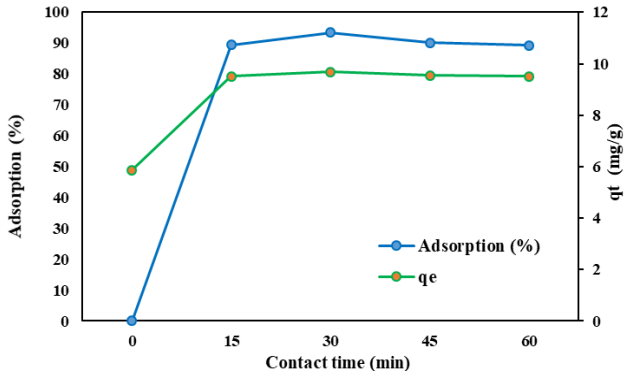

Figure 8. Effect of contact time on adsorption and q_t

When the PSO kinetic model graph given in Figure 9 is examined, it can be said that the MB adsorption of SiO_2 NPs follows the PSO model because the regression (R^2=0.9999) value is almost close to 1. The PSO kinetic model suggests that there is a chemical bond between the dye and sorbent molecules and relates this to the adsorption capacity [11]. In this case, it can be said that there is a chemical reaction between

SiO_2 NPs and MB molecules. It can be said that SiO_2 NPs synthesized from sodium bentonite are not only an effective photocatalyst but also an efficient adsorbent. SiO_2 and nanomaterials prepared with SiO_2 used in various dyes in the literature and their process results are given in Table 2. It is noteworthy that SiO_2 NPs removes the dye from the aqueous solution in a short time. The effectiveness of SiO_2 NPs in the photocatalytic degradation and adsorption parts of the study is promising to achieve good results without optimizing the process.

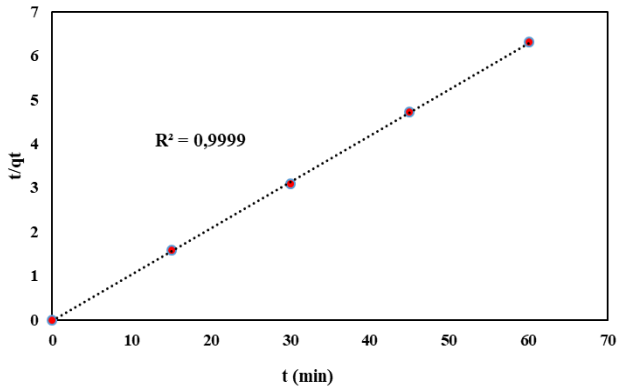

Figure 9. Graph of PSO kinetic model

Table 2. Adsorbents used in literature and process information

Sorbent	Sorbate	Adsorption (%)	Contact time (min)	References
SiO_2 NPs	MB	95.1	180	[27]
SiO_2-TiO_2	MB	88.6	30	[29]
SiO_2	Safranin O	96	60	[30]
SiO_2	Methyl orange	73	80	[30]
SiO_2NP	MB	90	60	This study

4. Conclusion

In this study, it was aimed to prepare SiO_2 NPs by processing sodium bentonite clay with chemical method. According to FTIR, SEM and EDX analysis, spherical nanoparticles in the range of 50-100 nm were obtained. It was revealed that 80% photodegradation was achieved when SiO_2 NPs were used as photocatalysts and 90% dye removal was achieved when used as adsorbents. These results were obtained for a contact time of 60 min and for MB dye for both processes. It was determined that the degradation process was compatible with second order reaction kinetics. It is thought that there is an interaction between the chemical dye and SiO_2 NPs because the adsorption process follows PSO. The study is aimed to be used in the future in process optimization in the removal of pollutants from aqueous solutions or in application areas such as drug delivery and therapeutic effect.

Article Information Form

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The Declaration of Conflict of Interest

No conflict of interest or common interest has been declared by authors.

Authors' Contribution

Conceptualization, N.K; methodology, N.K. and A.K; validation, N.K.; investigation, N.K. and A.K.; data curation, N.K.; writing—original draft preparation, N.K.; writing—review and editing, N.K.; visualization, N.K.; supervision, N.K.; project administration, A.K and N.K. All authors have read and agreed to the published version of the manuscript.

Artificial Intelligence Statement

No artificial intelligence tools were used while writing this article.

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