

PHOTOCATALYTIC DEGRADATION ACTIVITY OF AROMATIC COMPOUND BY TiO₂ NANOPARTICLES

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ABSTRACT

TiO₂'s capacity as a photocatalyst and its great performance in photovoltaic applications have been the subject of extensive research. In this study, TiO₂ nanoparticles were examined for phenol degradation under UV radiation in a range of pH conditions. SEM and XRD were employed to describe the TiO₂ nanoparticle. The outcomes demonstrated that Titanium dioxide nanoparticles have an anatase-like structure and a 26 nm particle size. Breakdown of phenol by UV light had phenol remove of 77% phenol was destroyed in 2 hours, whereas the degradation of phenol without UV rays had removal efficiency of 15% dye was destroyed in 2 hrs, according to the efficiency of photocatalysis of TiO₂ nanoparticle. It said that UV light could trigger the efficiency of photocatalysis of Titanium dioxide nanoparticles. The breakdown of phenol 15% is no a photocatalysts activity; rather, it is the destruction of phenol by Titanium dioxide nanoparticles without the availability of UV light. Titanium dioxide nanoparticles' photocatalytic activity is pH-sensitive. Titanium dioxide nanoparticles have the efficiency of photocatalysis of 77% in neutral conditions (pH 7.0), the highest photocatalytic activity occurs.

KEYWORD: TiO₂, photo degradation phenol, XRD, SEM.

1. INTRODUCTION

The titanium dioxide's and's photocatalytic capabilities and their great effectiveness in photovoltaic applications have been extensively explored.^[1-5] Because of its high Activity, TiO₂ has recently generated a lot of interest in the field of environmental cleaning.^[6] Additionally, it demonstrates chemical stability, few toxicity, few pollutant load, and low cost.^[7] To enhance the reactivity surface area and boost efficiency, Titania size has been decreased.^[8] Numerous organic contaminants can be successfully degraded by TiO₂ photocatalytic wastewater treatment, according to research.^[9]

TiO₂ has been employed as a photocatalyst extensively because of its high stability and economic availability. TiO₂ has been synthesized in a variety of forms, including nanoparticle, nanotube, nanofiber, and nanosheet.^[10] The utilization of Titanium dioxide in the wastewater treatment process is the subject of numerous initiatives.^[11] The pH of a solution is a crucial factor in the photocatalysts reactions occurring on particle surfaces due to it controls the surface tension characteristics of the photocatalyst as well as the aggregate sizes it generates.^[12] Due to the attained severe circumstances, the dissociation of oxygen and water molecules is a process that is frequently seen.^[13] The pH may have a significant impact on how well drugs are

removed from the environment. Regarding photolysis, different species may have different transformation products, rates of mechanism-based degradation, and photolytic degradation routes.^[14] The facts that the structure orientation of the molecules favors the assault of the oxidation products under all those conditions may possibly be a contributing factor to the higher breakdown rate in acidic or basic pH.

The rate of deterioration for the breakdown and While the rate of the pesticide's degradation and mineralization was shown to decrease with an increase in pH solution, degradation of acid red was shown to rise with an increase in pH solution.^[15] Due to its many functions, including electrostatic attractions between the surface of the semiconductor, charged radicals, solvent molecules, and the substrate produced throughout the reaction, interpreting pH impacts on the photocatalytic activity is a particularly challenging endeavor. There have been many tries, but no satisfactory justification.

We will talk about how a substance's pH is pollutant phenol effect of the solution photoactivity of TiO₂ nanoparticles in this study. It has been proposed that ZnO nanoparticles, which have a higher exciton binding energy than TiO₂ nanoparticles and are used in a variety of catalysts including responsive electronic goods,

ultraviolet (UV) light emitters, chemical sensors, spin electronics, and piezoelectric devices, are more promising UV emitting phosphors because of their unique properties and wide range of applications (60 meV). This characteristic lowers the UV lasing thresholds and results in greater UV.^[16] At ambient temperature, emission effectivenessThe potential exists to increase the efficiency of photocatalysis processes because of the increased surface area of nanosize Zinc Oxide compared to bigger particles.

According to a paper, smaller TiO₂ and ZnO particles have a higher redox potential because of the photocatalytic breakdown of organic toxins when exposed to UV rays. The created TiO₂ and Zinc oxide nanoparticles can be viewed as the optimal photocatalysts for industrial-scale environmental cleaning in a central pH environment.^[17] Numerous researchers have looked into the photocatalytic destruction of inorganic and organic contaminants on various semiconductor types. indirect response at the holes that are positive, which form in the valence band semiconductor after that migrate to surface of catalyst and participate in inter - facial oxidation-reduction reactions, is thought to be the cause of Adsorbed colored materials oxidize in the valence band between the valence band. either organic or inorganic molecules. However, in the nanoparticles dispersed, the band gap was reduced, which decreased the recombination process.^[18] In the past, the Langmuir-Hinshelwood rates model and the pseudo first order reactive kinetic model have been used to explain the photocatalytic color removal process.^[19] This investigation looks at the sol-gel process for producing titanium dioxide, explores effects on particle sizes, and contrasts the photocatalytic activity of the generated nanoparticles that of the base material.

2. Experimental

2.1. Materials and methods

We bought TiCl₄ (99.99%) from the Flucka Company.

%99.98) purity is guaranteed for absolute ethanol, according to the Gainland Chemical Company (GCC.(phenol is a pollutant (supplied from Fluka company.(TiO₂ nanoparticles were generated under normal environmental conditions.

2.2. Preparation of phenol solution

Prepare standard solution of phenol is deferent concentration 10,20,40,60 and 80 ppm to calibration curve and choose 60 ppm in 1 liters for degradation by take 60 mg in 1000 ml .

2.3 TiO₂ nanoparticle preparation

An earlier methodology that was modified was used to create the TiO₂ nanoparticles.^[20]

The following was the method's explanation:

10ml of TiCl₄ and 100 milliliters of pure ethanol were introduced drop - wise to a reaction mixture because the

reaction is exothermic, TiCl₄ is very volatile, and it produces hydrogen chloride as fast as it is added.

Following 15 hours of dried at 85°C inside an oven, the resultant solution went from clear to yellow and formed TiO₂ nanopowder as in figure 1.

TiO₂ nanopowder was eventually formed in a variety of phases by splitting all produced powder into seven sections and heating the obtained powdered in a furnace at heats equal to 400 °C.

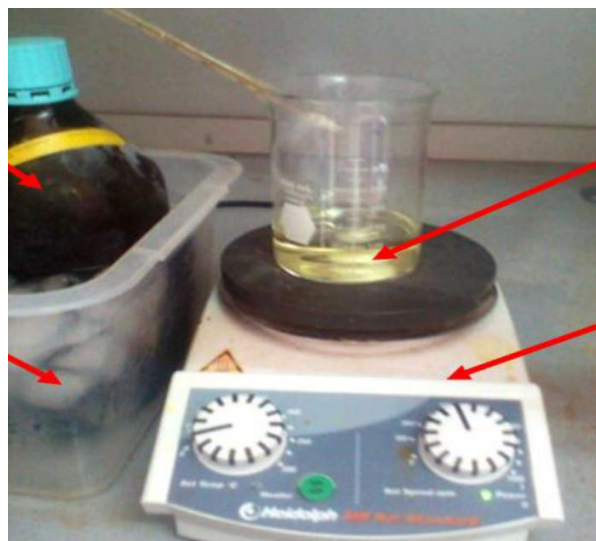


Figure 1: Summarizes this technique.

2.4. Calibration Curve

In order to create the calibration curves, standardized phenol aqueous solutions were used.

At 269 nm, the absorption of each quantity was determined.

Table 1 presents the normal calibration values, which are shown in Figure 2-3.

Table 1: Absorbance of different phenol concentrations.

Con. (ppm)	Abs. in 269 nm
0	0
10	0.19
20	0.36
30	0.55
40	0.73
50	0.91
60	1.09
70	1.24
80	1.47

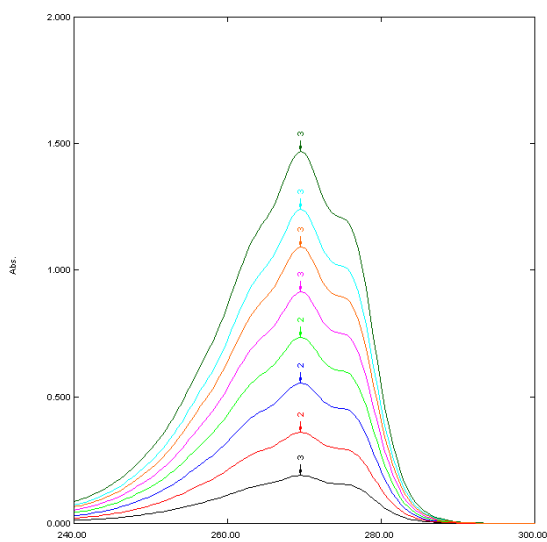


Figure 2: Shows the UV-visible spectra of phenol at various concentrations.

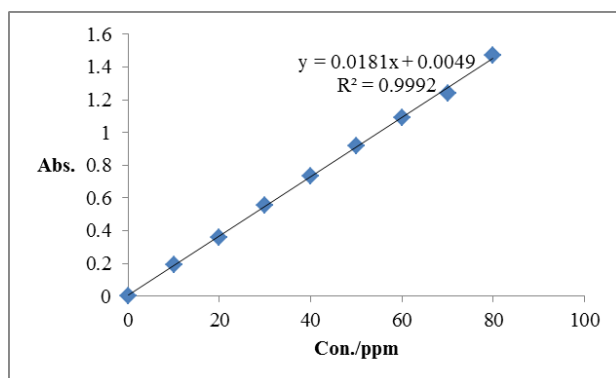


Figure 3: Calibration curve for different concentrations at 269 nm for phenol.

2.5. Characterization

A Philips X-ray diffraction pattern (PW 1730) was employed for X-ray diffraction with automatic data

collection using Cu K radiation ($\lambda = 0.15406 \text{ nm}$) and operated at 40 kV/30 mA. (APD Philips v3.6B.)

The diffract patterns mostly in 2θ to 60° range were gathered using a 0.05° step length and a 2 second capture duration in step.

The diffractometer had to be calibrated before each measurement could be made.

The basis for the XRD calculation of crystallite size was the Scherrer equation.^[1]

2.6. Synthetic Methods for TiO₂ Nanostructures

TiO₂ nanoparticles can be created using a variety of techniques.

Typically, nanoparticles, nanowires, nanotubes, and nanotubes are the main end products. Both gas and liquid techniques can be used to create nano-TiO₂. Due to the need for specialized equipment, gas procedures are typically more challenging than liquid ones. Among the procedures utilized to make nano-TiO₂ are hydrolysis, drying, and calcinations.

The pursuit of affordable, high-quality products has been a constant endeavour. Due to their affordability and high titanium concentration, a number of substances, including titanium tetrachloride (TiCl₄), can be employed as raw materials to produce nano-TiO₂. TiCl₄ has been used in a number of different ways to create nano-TiO₂.^[21]

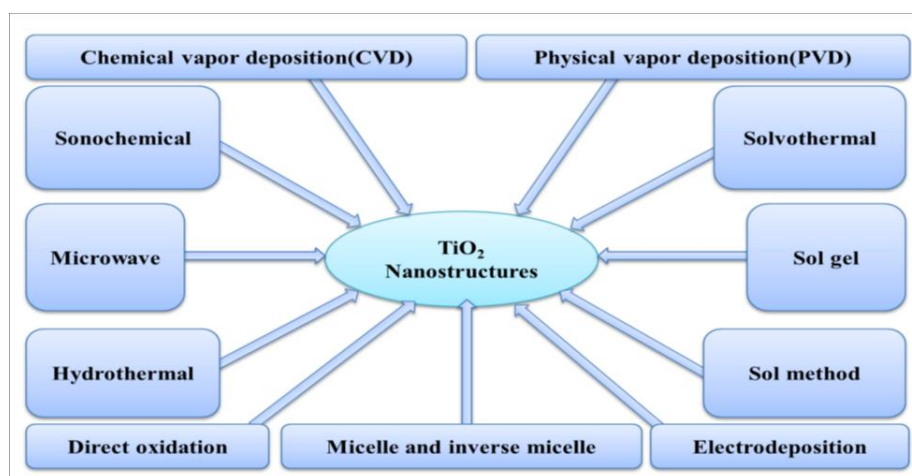


Figure 4: Methods to synthesis TiO₂ nanoparticles.

2.6.1. Sol-Gel Method

Growing numbers of novel materials have been developed using the sol-gel process, including those for

catalyse,^[22] biosensors,^[23] membrane,^[24] fiber,^[25] electro-optic gain surfaces,^[26] photosensitive and nonlinear uses,^[27] and liquid - solid electrochemical devices.^[28]

TiO₂ nanopowder is created using the most significant sol-gel technique.

With this technique, ultrafine powder can be produced, which has highly disproportionate surface to volume and a strong impulse to clump together. By heating, we mean the adherence of particles to one another due to the attraction of van der Waals interactions. Sintering necks form covalent bonds between these particles. As a result, these pressures are strong in the instance of nanoscale, resulting in nanopowder with a large surface area.

Precursors in a typical procedure are often inorganic materials salts or organic metal compounds like metal halides.

These undergo exothermic hydrolysis and polymerization, which results in the formation of a colloidal dispersion or sol.

As illustrated in Figure 5, the solvent is completely polymerized and lost by calcination during the change from the liquid phase to solid gel phase.

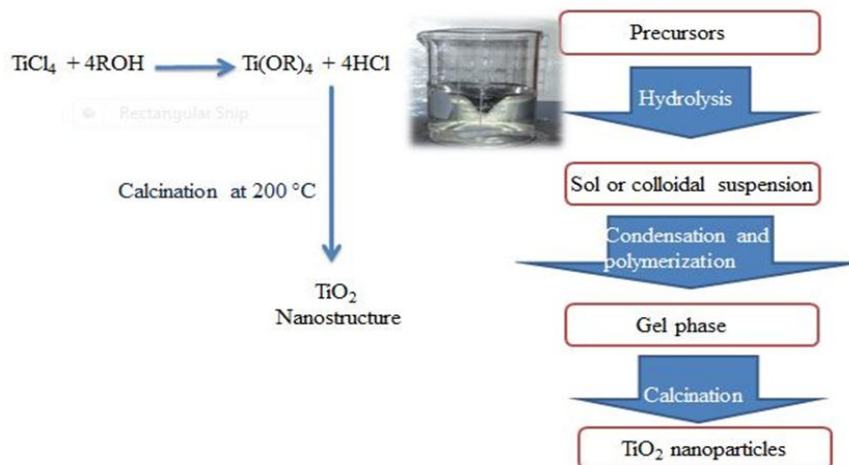


Figure 5: Procurement to synthesis TiO₂ nanoparticles.

2.7. Photocatalytic Activity Experiments

To use a 100 mL photoreactor, the studies on photocatalytic activity were carried out.

The radiation utilized had a typical wavelength of = 269 nm and was produced by a Philips (CLEO, Poland) mercury lamp with four 15W bulbs that produced a 600 mW Hg lamps UV(A) at a strength of 0.7 mW/cm². The suspended solutions were created by mixing 100 mL of a phenol aqueous solution with a 60 ppm concentration with 175 mg of nanoparticle catalysts. The suspensions were magnetically agitated for 30 minutes in the dark before being exposed to radiation to achieve adsorption

equilibrium. The suspension were sampled periodically while they were subjected to irradiation and adsorption. 3 mL aliquots of reaction medium taken, and they were centrifuged for 15 min. A long, flexible needle-equipped syringe was used to carefully collect the supernatant, which was then centrifuged once more at the similar speed for the same amount of time. The catalyst's tiny particles were removed using a second centrifuge.

After the second centrifugation, the phenol's absorbed at its maximum wavelength of 269 nm was measured using a UV-visible spectrophotometer.

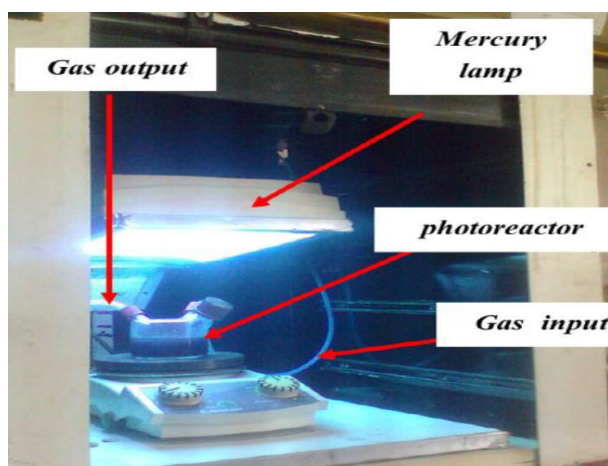


Figure 6: Experimental apparatus of photoreactions.

3. RESULTS AND DISCUSSION

3.1. Raman Spectroscopy

The lengthy maximum at 144 cm⁻¹ (E_g) [177] is one of the key peak mostly in Raman data depicted in the pictures.

Although there are three groups that reference to rutile, the 515 cm⁻¹ A_{1g} maximum is the most intense for the rutile phase.^[29-32]

All of the Raman spectrum for anatase phase in the TiO₂ remain unchanged, with the exception of the composites' anatase Raman spectrum at 144 cm⁻¹ and rutile one at 612 cm⁻¹ being somewhat widened.

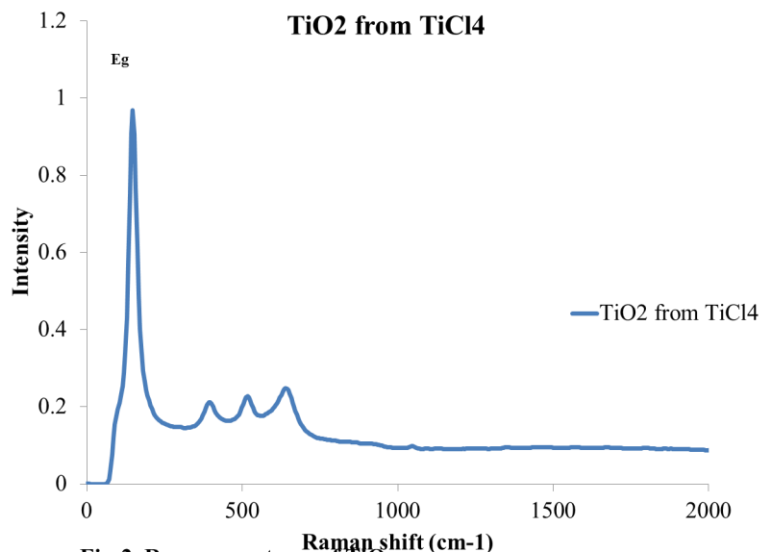


Figure 7: Raman spectrum of TiO₂ synthesis.

3.2. X-ray Diffraction Spectroscopy (XRD)

The average diameters of the crystallites (D) were determined use XRD data and Scherrer's equation in the following formula.^[33-34]

$$D = \frac{k\lambda}{\beta \cos\theta}$$

D is the typical crystalline size, k = 0.94 the fixed crystal lattice, X-ray wavelength of Cu K = 0.154 nm, is the peak's entire width calculated at half maximum intensity, and is the peak's Bragg's angle.

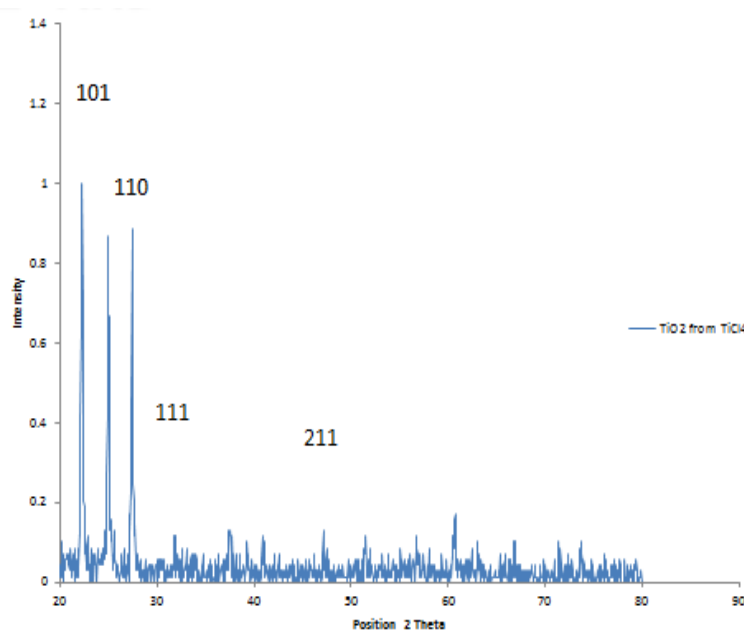


Figure 9: XRD of TiO₂ synthesis.

Table 2: Average crystal size measured by XRD for samples.

Samples	Average crystal size /nm
P25	22.797
TiO ₂ (TiCl ₄)	26.937

3.3. Scanning Electron Microscopy (SEM)

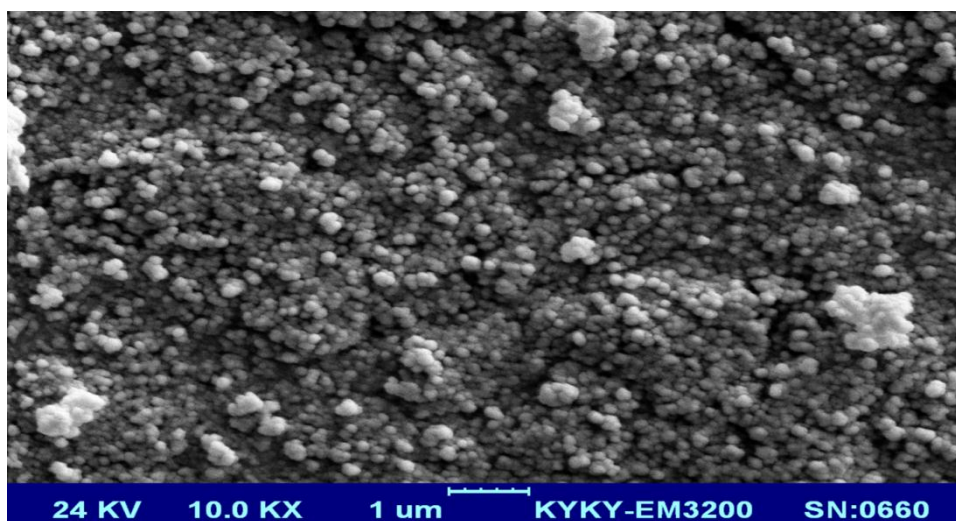
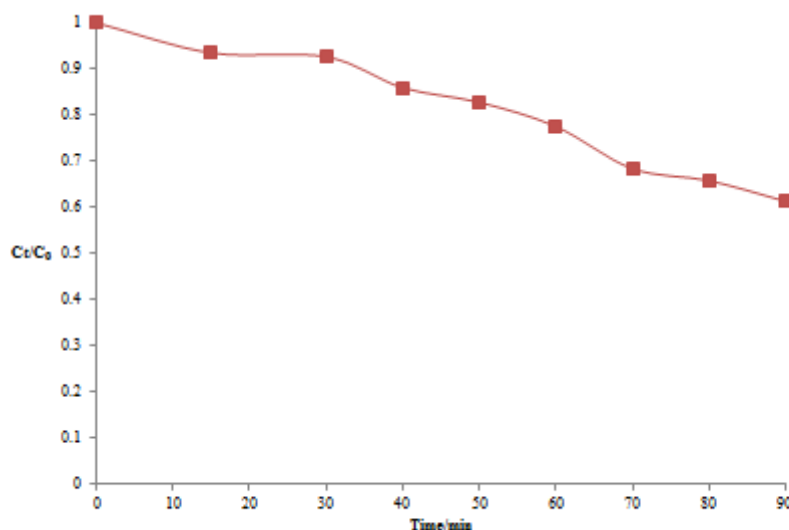
High vacuum was used to operate a SEM. The fundamental ideas are that an appropriate source produces an electron beam. To produce a narrow beam of electrons, a high voltage and passage speed the beam of electrons via a set of electromagnetic lenses and

apertures. The thin beam of electrons was then utilized to scan the sample surface using scan coils.

Different electron image kinds exist.

The backscattering image and the secondary electron imaging are the two types. A high resolution image is primarily produced using the secondary electron imaging.^[35-36] On a JEOL JSM-6700F apparatus, SEM measurements were performed to use a secondary electron detection (SE) at an applied potential of 2.0 kV.

Software known as Spip, or scanning prop image processor, was used to determine the average particle sizes.

**Figure 10: SEM of TiO₂ synthesized.****Figure 10: Photodegradation of phenol by TiO₂ synthesized.**

The Langmuir-Hinshelwood model can be used to describe the photocatalytic reactions occurring on TiO₂ surfaces.

Following the adsorption equilibrium, the reaction rate can be written as: $\ln \left(\frac{C_0}{C_t} \right) = kt$

Where k and t are the actual average speed constant and time, respectively, and C_t and C_0 are the component quantity at instances of time = t and $t = 0$, respectively.

The slope of k is obtained from a plotting of $\ln(C_0/C_t)$ against t .

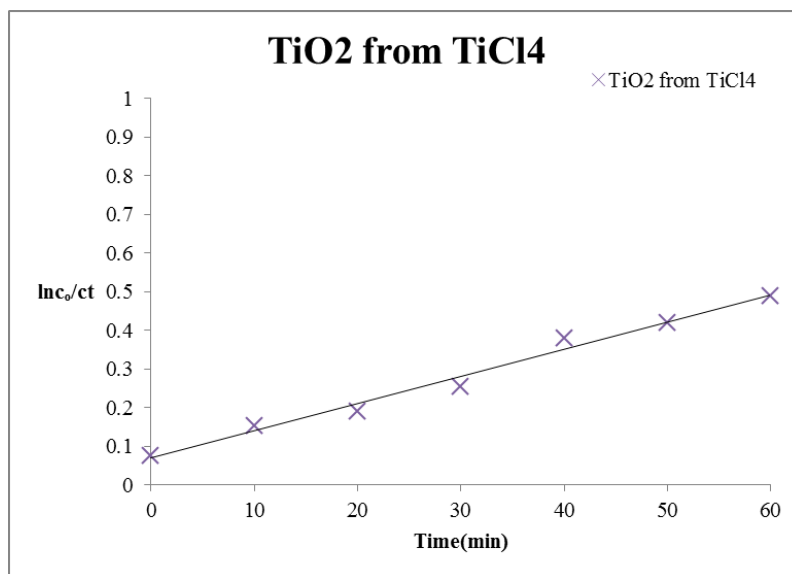


Figure 12: Photodegradation removal of phenol by TiO₂ synthesized.

CONCLUSION

TiCl₄ and absolute ethanol were used as additives in the effective synthesis of TiO₂ nanoparticles utilizing sol gel. Anatase nanoparticle production was verified by XRD analysis. With an average particle size of roughly 26 nm across all samples, the maximum analysis of the XRD pattern revealed only the inclusion of two phases of pristine crystal structure, which may find use as UV shielding material. The findings showed that the top-notch TiO₂ nanoparticles had an annealing temperature of 400°C and a volumetric precursor percentage of 50%.

Sol gel is a great way to create TiO₂ nanoparticles, which are a semiconductor. Sol gel enhances the efficiency of photodegradation for pollutants by increasing surface area and decreasing particle size distribution, and it also produces nanoparticles, which results in high-throughput and economical processes.

The produced catalysts' TiO₂ anatase characteristics were shown by XRD, SEM, and Raman analysis. According to adsorption and photocatalytic degradation of phenol by TiO₂, the greatest elimination percentage was 77%.

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