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Photocatalytic Potential of Green Synthesized ZnO Nanoparticles in Dye Removal Compared to TiO_2; a Performance Evaluation

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Abstract

The present study focuses on the photo catalytic degradation of Reactive Blue 13 dye using a green synthesis process for the preparation of zinc oxide (ZnO) nanoparticles. In contrast, titanium dioxide (TiO_2) is used as a comparative catalyst. The synthesis of zinc oxide nanoparticles (ZnO-ZNP) was carried out using a green approach employing the grapefruit peel extract and characterized by scanning electron microscopy SEM and zeta potential analyses. SEM analysis showed the spheres and smooth pellets of ZnO nanoparticles with an average size of 62 nm; at the same time, zeta potential (-11.9 mV) was shown to have good dispersion stability properties. The photo catalytic degradation of Reactive Blue 13 (RB-13) was performed under UV light, and different parameters such as pH, catalyst concentration and irradiation time were used to achieve the most effective dye removal. The study revealed that 0.06 g of ZnO nanoparticles exhibited an impressive photo catalytic activity, achieving a remarkable 98% dye degradation efficiency at pH =11 and after an irradiation time period for a few hours (120 minutes). In comparison, TiO_2 performed with a lower efficiency of 86% under the same conditions. These findings indicate the capability of ZnO nanoparticles in intense textile dye removal processes for wastewater treatment. The high degradation efficiency of Reactive Blue 13 in optimum conditions proves the potential of ZnO as a green and economical photo catalyst for environmental remediation, surpassing conventional TiO_2 due to slow dye decolorization. The outcomes of this can be very helpful in advanced nanomaterial exploration for sustainable wastewater treatment purposes.

Keywords: Degradation, grape fruits, nanoparticles, reactive dye, zeta potential

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1. Introduction

The Earth's surface is two-thirds water, but only 0.03% is suitable for human consumption, and with a growing global population and industrial activities, demand exceeds supply. Essential to textile dyeing, water serves as a solvent for chemicals and dyes, washing and rinsing media, a medium for transferring chemicals and colors to fabric. Industry's wastewater contains harmful dyes resistant to biodegradation [1]. Various methods, including physical, chemical, and biological processes, have employed to remove dye molecules from industrial effluents, each has its limitations and drawbacks [2]. Nanoscience and nanotechnology offer potential solutions, as nanomaterials can engage in novel interactions. Effective wastewater treatment methods are essential to address environmental and health impacts of industrial activities. Metal oxide nanoparticles possess distinctive chemical and physical characteristics that make them useful in many industrial and domestic applications like magnetic resonance imaging (MRI), wastewater treatment, energy research, medicine, and catalysis [3].

However, although they have enhanced sensitivity and detection limits, there is a need to deal with their cytotoxic effects and environmental hazards [4]. The nanoparticles of zinc oxide (ZnO-NPs) are versatile inorganic substances that exhibit a high level of photo stability, chemical stability, and UV absorption hence they qualify for use. This is why titanium dioxide (TiO_2) and zinc oxide (ZnO) nanoparticles have special physical and chemical properties that make them useful in many different applications in homes and factories like sewage treatment plants [5]. It is believed that TiO_2 has a great ability to absorb organic molecules, which contributes to their hydrolysis and subsequent photo catalytic [6] reaction. The attractive physical and chemical properties of ZnO nanoparticles have wide applications in many areas such as biotechnology, solar cell technology, and photo catalysis [7].

2. Experimental Materials and Method

Undertaken at Riphah International University's Chemistry lab in Faisalabad was a study focusing on evaluating the photo catalytic capability of green-synthesized

zinc oxide nanoparticles in the degradation of Reactive Blue 13 dye, with titanium dioxide as the comparative catalyst. Employed were various apparatuses, chemicals, and instruments namely zinc sulfate heptahydrate ($ZnSO_4.7H_2O$), NaOH, HCl, distilled water, beakers, a measuring cylinder, hot plate, muffle furnace, UV chamber, centrifuge, sonicator, and UV Vis spectrophotometer. These devices facilitated pH measurement, heating procedures, homogeneous heating, photo catalytic degradation, separation, absorbance determination, and sample preparation. The methodology necessitated synthesizing ZnO nanoparticles via fruit peel extract, followed by calcination at designated temperatures and assessment of photocatalytic activity. The study aimed for an in-depth exploration of the degradation process of Reactive Blue 13 dye utilizing ZnO nanoparticles [8].

2.1. Synthesis of Zinc Oxide Nanoparticles

ZnO nanoparticles' synthesis involved utilizing zinc sulfate heptahydrate as the precursor in conjunction with a fruit peel extract. Additionally, three solutions were prepared, each with a concentration of 0.1 M: NaOH, HCl, and $ZnSO_4.7H_2O$. The NaOH solution's function was to regulate the pH levels, while the HCl solution maintained an acidic environment. The Zn salt solution was created by dissolving zinc sulfate in distilled water [9]. In the synthesis process of ZnO nanoparticles, the peel extract and Zn salt solution were mixed in a 1:1 ratio, then subjected to heating at 70°C with constant stirring, followed by cooling. To achieve a uniform consistency, the solution was gradually added drop wise with continuous stirring and subsequently centrifuged to extract the nanoparticles, which were then dried and stored. Heating was then applied for 30 minutes at 70°C. To precipitate zinc ions into ZnO nanoparticles, the mixture's pH needed to be adjusted to 11 once it had cooled to room temperature [10].

This critical step was carried out by adding sodium hydroxide solution." To allow the nanoparticles to develop, the mixture was left at room temperature for five hours. The produced ZnO nanoparticles were then separated using a centrifugation process that ran for ten minutes at 10,000 rpm [11]. The 40 minutes were spent drying the gathered nanoparticles in an oven set at 80°C. After the nanoparticles had completely dried, they were gathered and kept in Eppendorf for further use. The nanoparticles were calcined at temperatures between 300 and 600°C for 4 hrs as shown in Figure 1. Sonication techniques, including direct and indirect Sonication, were used to reduce the size of the nanoparticles, disperse them in a base fluid, and deagglomerate them. Scanning electron microscopy (SEM) and Zeta potential analysis were used to study the morphology and size of ZnO nanostructures [12].

2.2. Experimental Setup for Photo catalytic Process

The experimental setup involved preparing a stock solution of Reactive Blue 13 dye and adjusting its pH using HCl and NaOH solutions. The dye's properties, and chemical structure, were also described. ZnO nanoparticles were used as a photo catalyst to degrade the dye under UV radiation. A UV-Vis spectrophotometer was used to track the dye elimination process during the photo catalytic reaction, which was carried out in a specially-made chamber with a 40-watt UV lamp. The following formula was used to determine the dye clearance efficiency:

$$\text{Dye Removal Efficiency } \% = \frac{\text{Co} - \text{Ct}}{\text{Co}} \times 100$$

2.3. Reactive Blue Dye 13

Reactive Blue Dye 13 is found in powder form that is dark blue, soluble in water, and insensitive to fighting. At 20°C, the solubility of the dye is 110 g/L, while at 50°C, it is 140 g/L. The water solution is blue-purple. About the white sex media, the reddish-blue staining changes (4–5) in the presence of copper. Used to dye and print continuously on rolls of cotton, viscose, wool, silk, and fabric [13].

2.4. Optimization of Different Parameters

The effect of pH (3-11), photo catalyst concentration from (0.02g to 0.06g), irradiation time 120 minutes, and different dye concentrations from (10ppm to 50ppm) in the photo catalytic degradation process was investigated.

3. Results and discussion

3.1. SEM Analysis

The SEM images reveal that ZnO nanoparticles (NPs) form agglomerated spherical clusters with smooth surfaces, with an average size of 62 nm as shown in Figure 1. ZnO NPs usually aggregate into smooth, spherical clusters that don't seem to have any cracks, based on the image. SEM images show that zinc oxide nanoparticles form large clusters with oval or spherical shapes, likely due to agglomeration during solution precipitation. The observed cluster size is larger than calculated using the Scherer method, maybe as a result of the challenges associated with capturing high-resolution images [13].

3.2. Zeta Potential Analysis

In Figure 2 zeta potential measurements using the Malvern apparatus, it was found that ZnO nanoparticles synthesized from *Citrus paradisi* extract had a zeta potential of -11.9 mV, indicating stability. As shown in Table 1, dispersions prepared with 0.2 mg of zinc oxide in 10 mL of deionized water and ultrasonic sonication with dynamic light scattering (DLS) were used to assess size distribution and zeta potential in distilled water with a pH of approximately 7.36. It was used to look at size distribution and shape of the produced ZnO-NPs. Table 2 illustrates the nearly uniform shape and crystallinity of ZnO NPs, which are noticeable in images due to their homogenous size distributions and crushed-ice morphology.

3.3. Particle Size of Zinc Oxide NPs

Specifically, the Z-average particle size was measured at 723 nm, while the Number Mean parameter was determined in water sample at 101 nm in Figure 3. ZnO-NP has been employed in numerous aqueous solutions, and zeta potential measurement is primary method for determining the surface charge. As a result, biosynthesized ZnO nanoparticles exhibit good stability as shown in Figure 5. This result implies that the active adsorption sites on these nanoparticles, which are capable of attracting dye and heavy metal ions from an aqueous solution, are what make them significant [14].

3.4. Absorption Spectrum for Reactive Dye

The absorption spectrum in Figure 4 was a key measurement to study the photo catalytic properties of reactive Blue 13 and was measured within the UV−Vis region

(300–700 nm) by using a spectrophotometer in this research [15]. The optical absorption of dye in this wavelength interval plays an important role in photo catalysis. The formation of reactive oxygen species under UV light in degradation of organic contaminants shows a sharp peak in spectra [16]. In order to screen electronic structure of material, spectrophotometry is done, and 574 nm peak plays a vital role in judging photo catalytic activity at different conditions, which is necessary for potential applications like ZnO nanoparticle-based applications needed by environment [17].

3.5. Effects of Parameters

3.5.1. Impact of Time

The impact of contact duration was examined at 10 ppm dye concentration, and reactive Blue 13 dye was photo-catalytically breakdown at an ideal pH of 11. As seen in Figure 5, a fixed amount of 0.06g of ZnO catalyst was used. The relationship between the blue dye degradation and the catalyst's contact time was examined [18]. The catalyst's irradiation time was found to rise by 98% when ZnO NPs were used as a catalyst and by 92% when TiO_2 was used as a catalyst for 120 minutes as shown in Figure 6. The rate of degradation of RB 13 dye increases, it reaches equilibrium after 120 minutes, although the graphical representation shows a quicker rate of decline throughout that time. The large number of reaction-available active sites is responsible for this faster deterioration. However, after this initial phase, it is expected that the pace of degradation slowed down, which could impede further advancement of the reaction [19].

3.5.2 Effect of pH

The impact of pH on the degradation rate was studied in photo catalytic reactions catalyzed by a Titanium dioxide standard catalyst. The decomposition of reactive blue was the highest at pH 10, and reduction reached a maximum value equal to 86%. Higher pH values led to generally better clearance rates. The highest decolorization efficiency at pH 10 was found for reactive blue 13 since ion adsorption with titanium dioxide increased, and the reactions of free radicals were easier. For zinc oxide catalysts, pH also affected the ZnO efficiency and photo catalysis of a reactive dye in Figure 7. A pH range of 3 to 11 was predetermined by adjusting the according to environmental conditions for wastewater treatment influence. Under basic conditions (pH 11), the generation of hydroxyl radicals has increased and subsequently improved for Reactive Blue 13 dye degradation. Photo-decolorization of dyes by TiO_2 or ZnO photo catalysts improves with increasing pH because UV light induces growth rate constants due to hydroxyl radicals, which provides for essential dye breakdown [20]. A neutral to slightly alkaline pH is the ideal range that maximizes radical formation, resulting in decreasing degradative rates. This is a meaningful result for wastewater treatment applications.

These data might be interpreted to mean that pH modulation could improve ZnO nanoparticle-based treatments. The maximum degradation of Reactive Blue 13 dye was obtained at pH 11 level [21]. Higher pH shows a greater percent of dye degradation owing to the presence of more hydroxyl radical production than lower values (Figure.8 ZnO nanoparticles produce these radicals upon exposure to UV light, and they attack dye molecules with high efficiency. The common reaction conditions greatly improve the production and reactivity of hydroxyl radicals, which is beneficial in enhancing the decolorization rate. The optimal pH is important for efficient photo degradation, indicating that a strategy to improve the wastewater treatment process could be adopted properly, which will enhance the photo catalytic activity of ZnO nanoparticles [22]. At pH 11, decolorization of dye by ZnO nanoparticles was almost up to 98%, which demonstrates that generating reactive species is dependent on pH conditions. pH in range of neutral to slightly basic enhances degradation efficiency by improving the interaction between ZnO nanoparticles and dye molecules. Low pH levels, on other hand, impair dye degradation. pH-sensitive charge interactions and electrical bonding between dye and nanoparticles govern efficacy of process [18].

3.5.3. Effect of Nanoparticles Concentration

With 0.06 g of TiO_2 catalyst, the color removal improved up to 85% in 120 minutes, the ZnO nanoparticles (0.06 g) reached a dye degradation of up to 98% under the same conditions shown in Figure 9. The higher ZnO concentrations up to 0.06g led to better dye clearance owing to increased hydroxyl radicals and reduced electron-hole recombination within the band gap. At this point, there is no more benefit. Dye degradation is important to maintaining an appropriate catalyst concentration, which will provide economic and catalytic efficiency [23]. Photo catalytic performance of Reactive Blue 13 dye is improved by a large factor due to the high rate, which consequently causes efficient color focus degradation in Figure 10; this leads to a considerable reduction in consumption. The amount of ZnO is crucial for the degradation of dye, which makes optimizing its dosage very significant in a cleaner, economically viable water treatment process.

High ZnO concentrations could not only offer accessible active sites for photoreaction but also contribute to photon absorption and restrain the negative effects resulting from an excess catalyst. The effect of initial pollutant concentration on the photo degradation rate is also discussed in relation to catalyst loading [24]. Efficiency of ZnO nanoparticles as photo catalysts and catalyst dosages If active site saturation occurs or the transparency of a solution suffers, using higher quantities of ZnO may not lead to an increase in efficiency as one might think because that would impede the light's interaction. The experimental data showed that degradation rates increased to 94% (for ZnO contents up to 0.06 g). In contrast, higher concentrations of this catalyst did not increase efficiency, evidencing the point of saturation for catalysis functionality.

3.5.4. Impact of Dye Concentration

The initial dye concentration significantly impacts the removal rate, with higher efficiencies achieved at lower concentrations. At a fixed TiO_2 concentration of 0.06 g, the degradation rate of Reactive Blue 13 increased as initial dye concentration decreased. Figure 13 illustrates this effect, showing in Figure 11 that reducing initial concentration from 50 to 10 ppm led to a significant improvement in color removal efficiency, from 3% to 86% in 120 minutes. At high ZnO concentrations, the penetration of light is limited, and consequently, few photons reach surface of catalyst to partially degrade it. Photo catalytic activity of dye Reactive Blue 13 (10-50 ppm) is higher at lower concern.

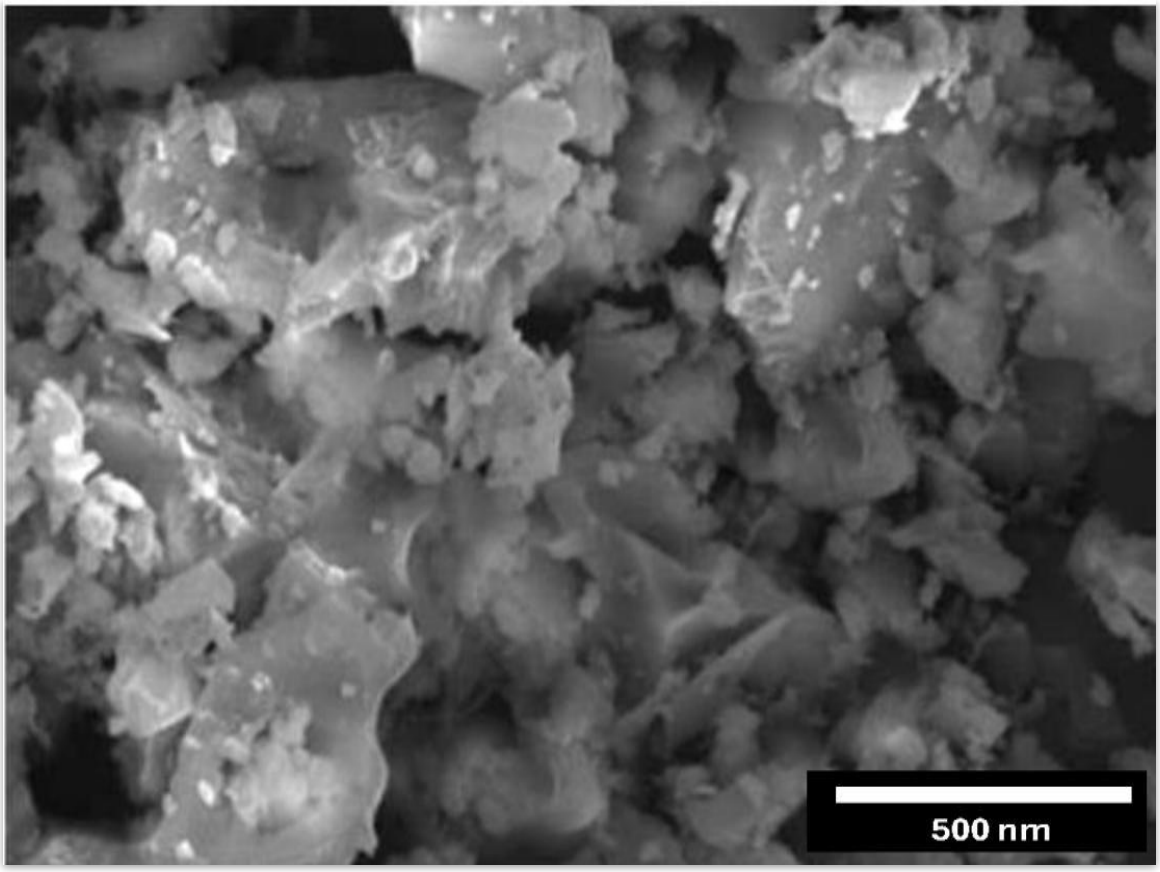

Figure 1. SEM Image of ZnO nanoparticles

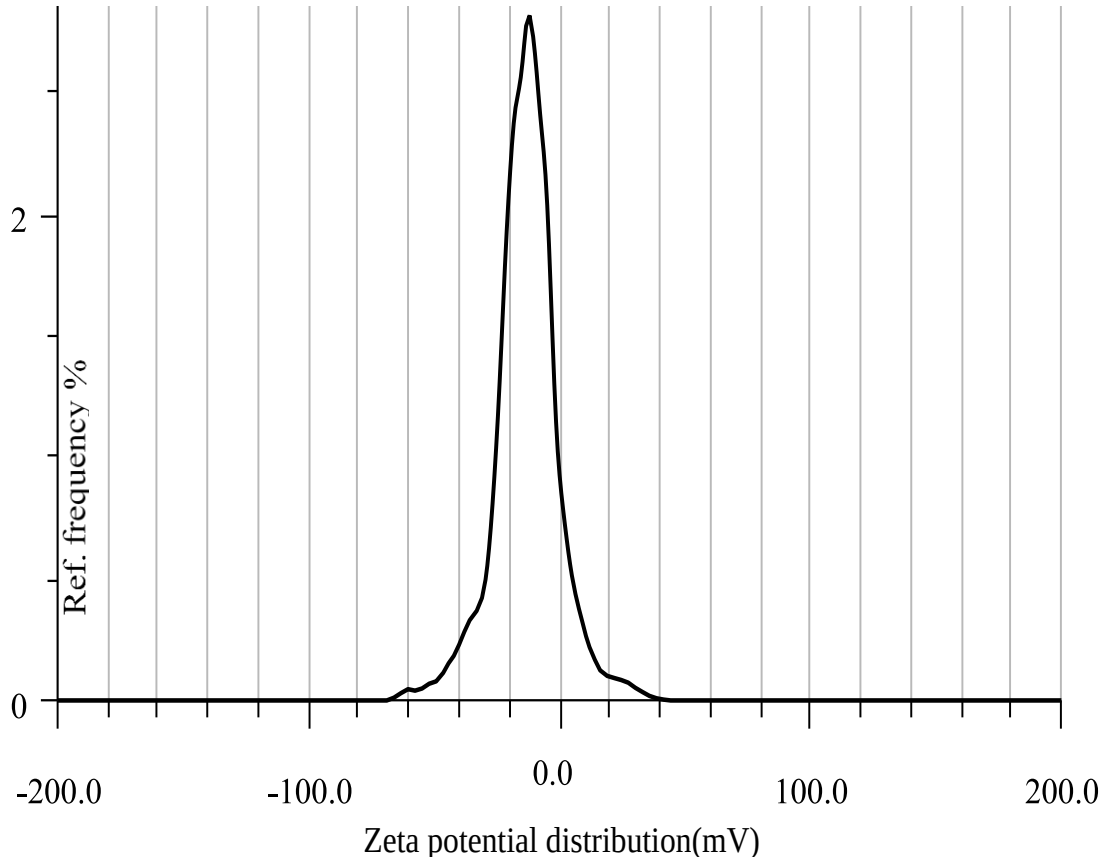

Figure 2. Zeta potential distribution of Zinc oxide nanoparticles.

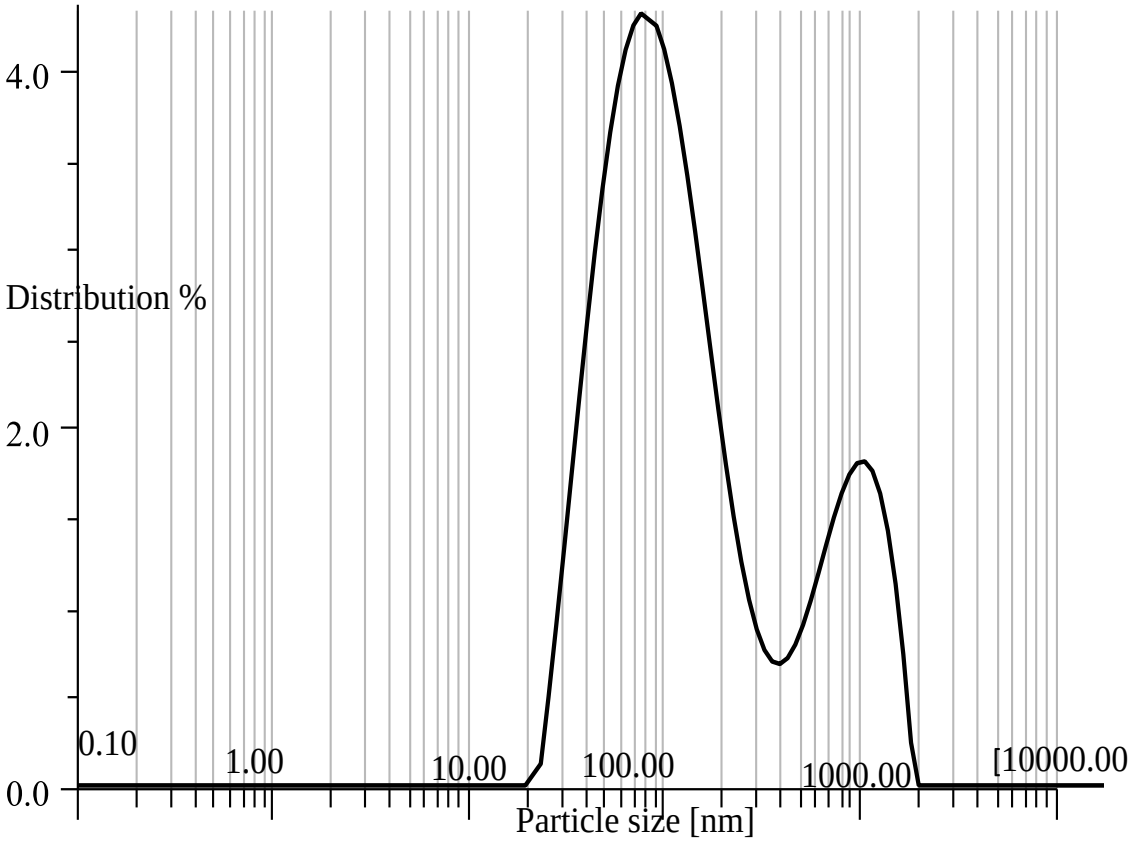

Figure 3. Particle size of Zinc oxide nanoparticles

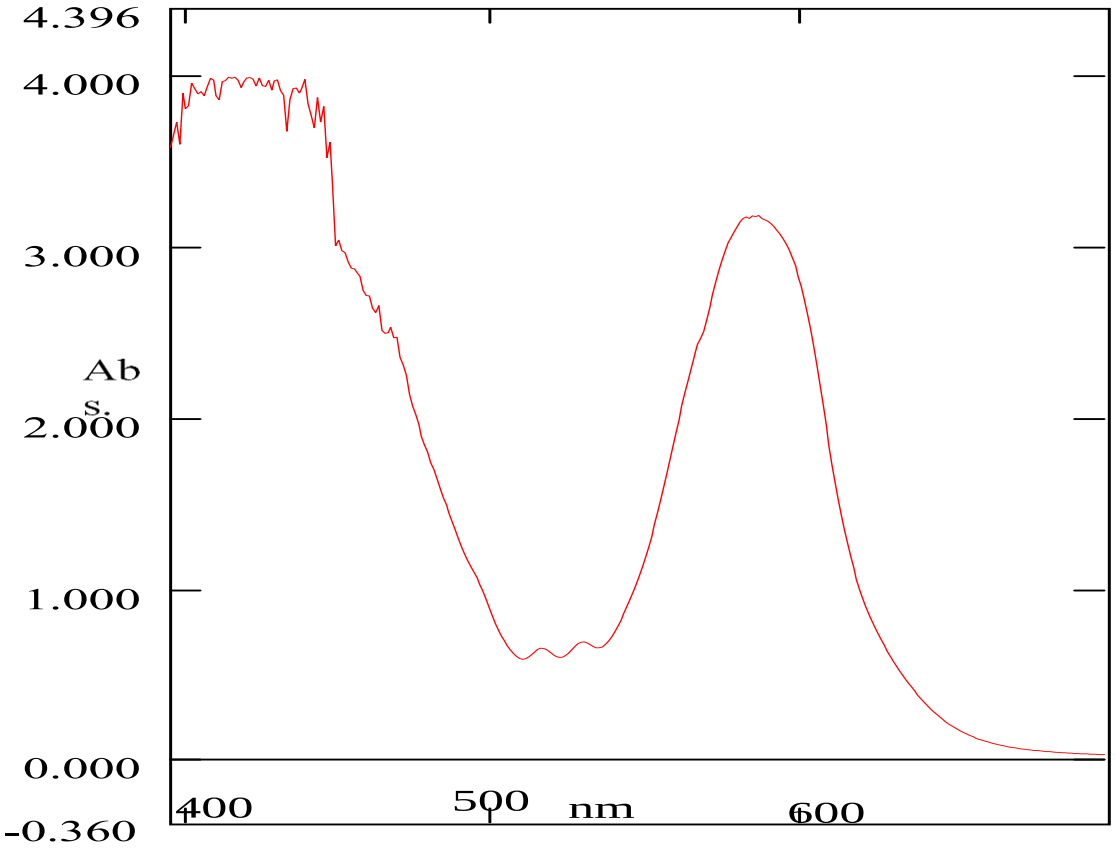

Figure 4. Portion lambda max. Wavelength of Reactive Blue

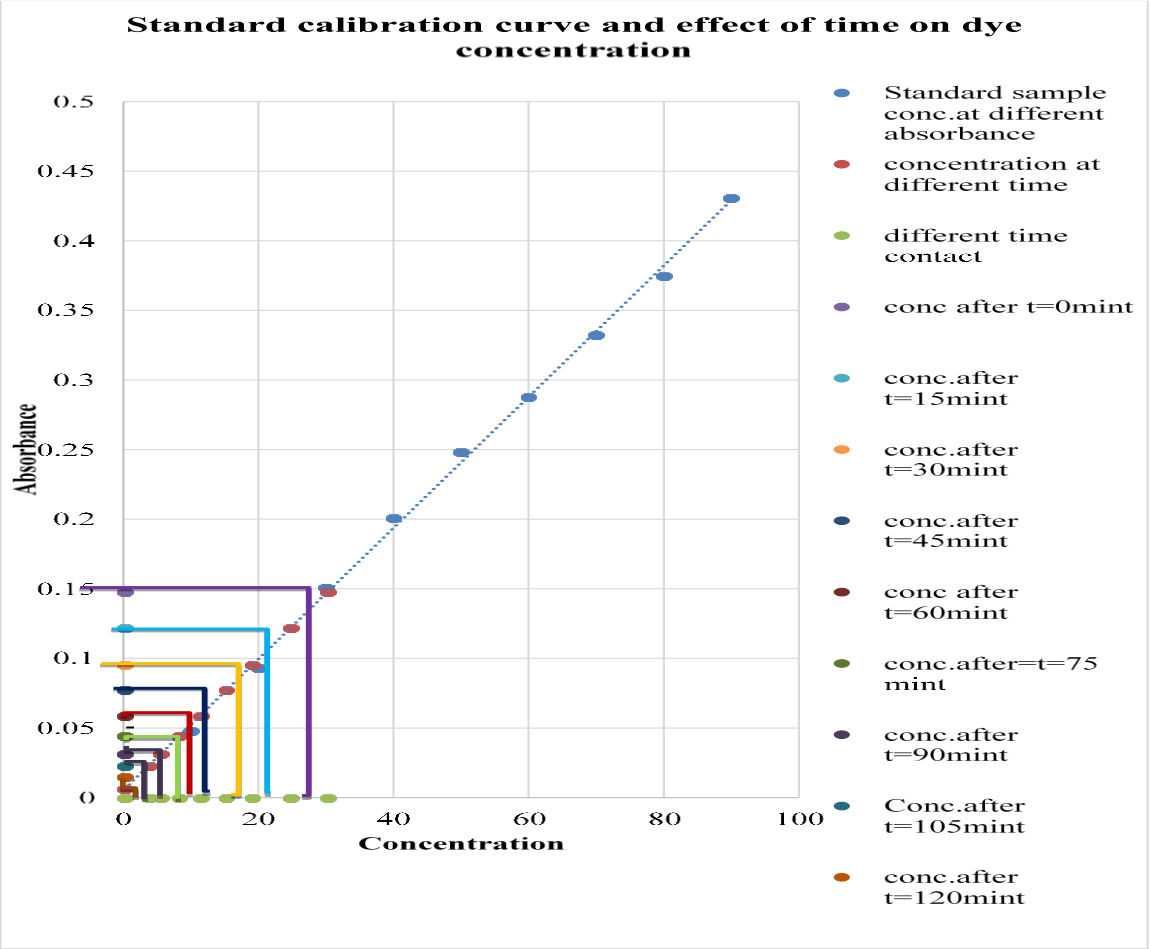

Figure 5. Standard calibration curve and impact of time

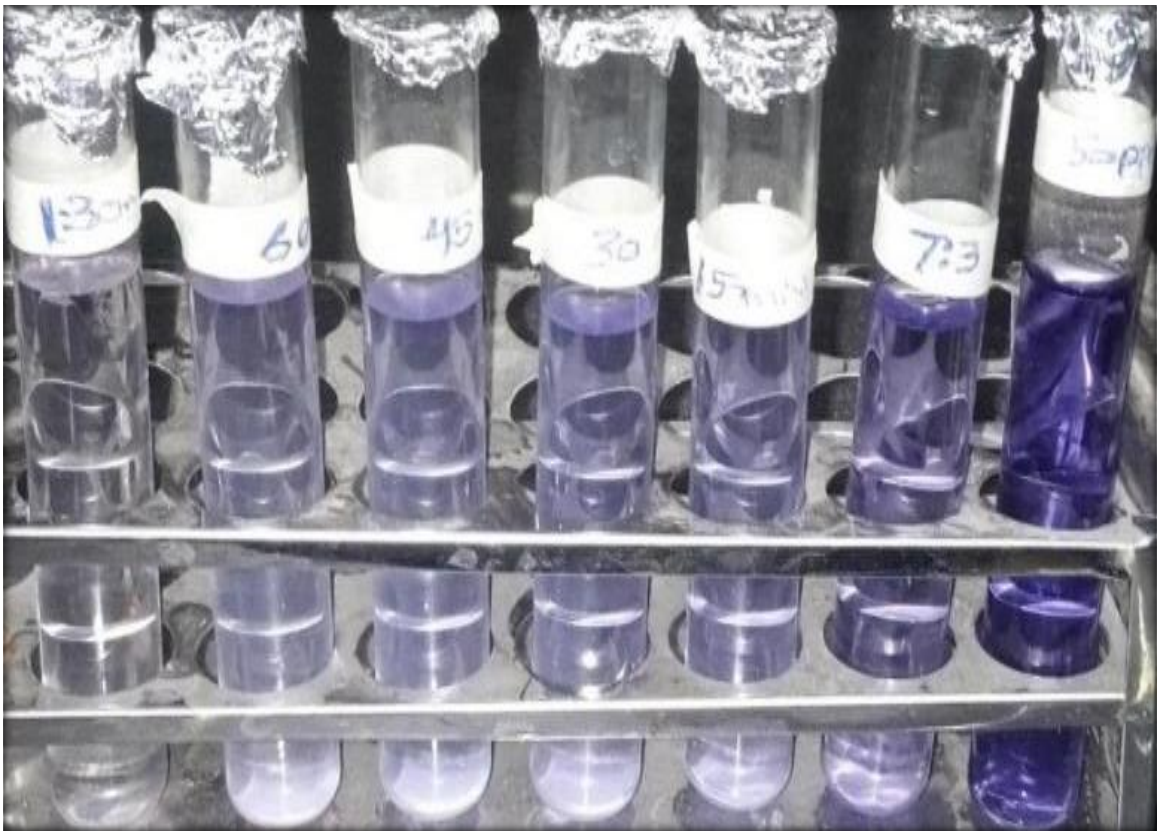

Figure 6. Impact of time on dye Removal by ZnO NPs

Figure 7. Effect of pH by ZnO nanoparticle

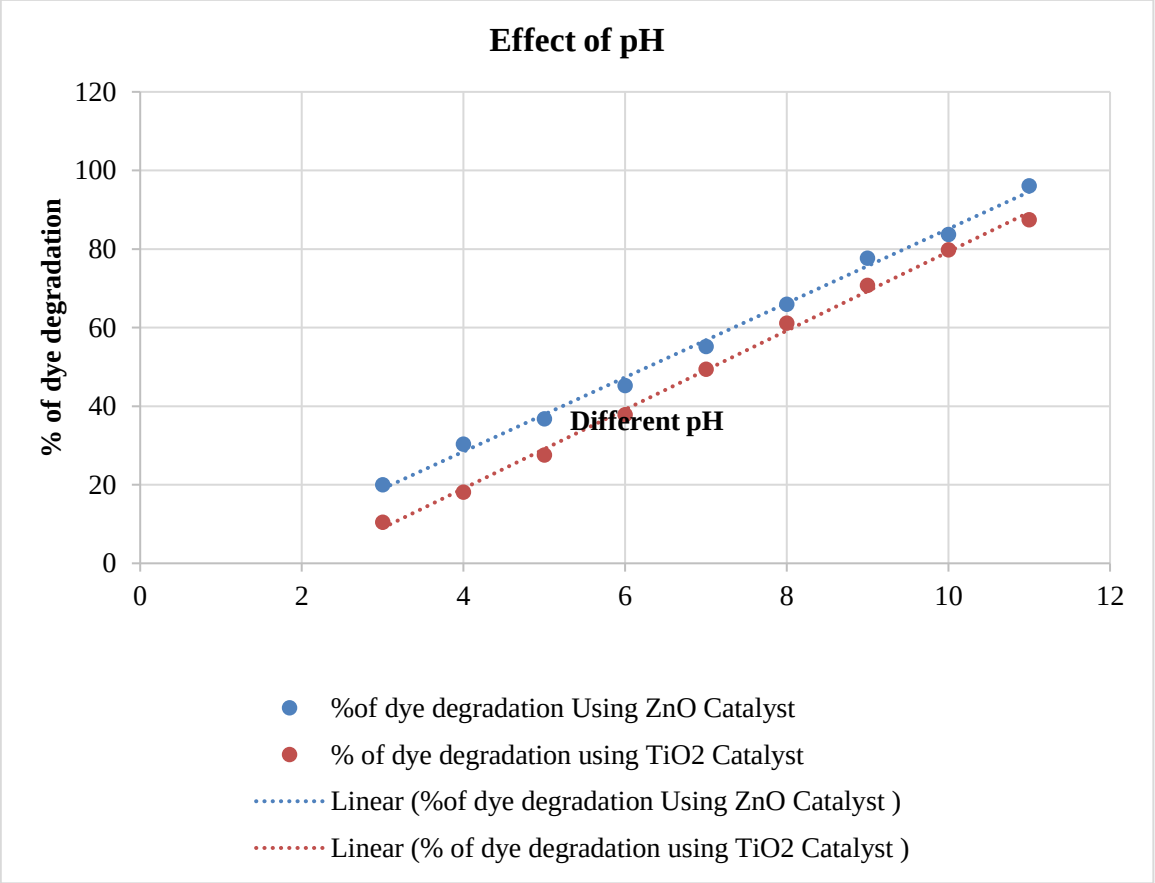

Figure 8. Effect of pH on % dye removal by ZnO and TiO_2

Figure 9. Effect of nanoparticles

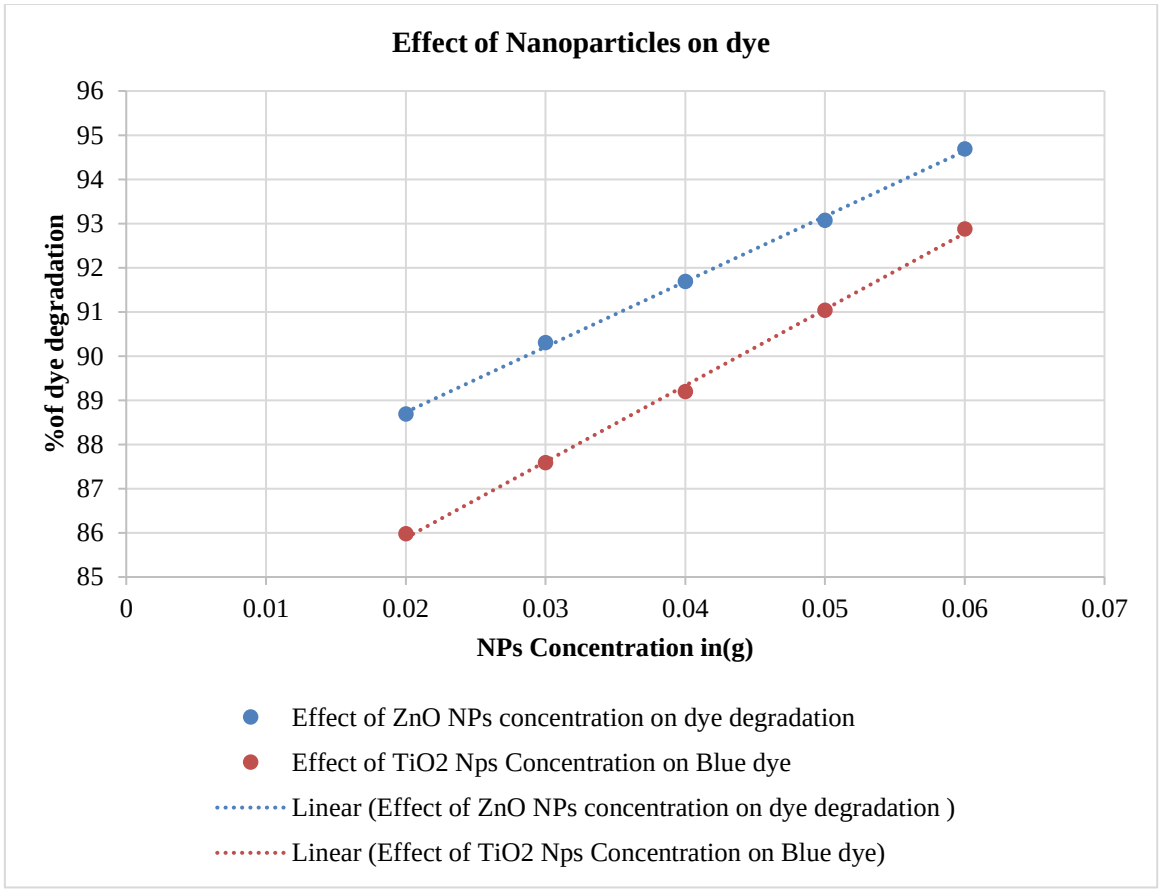

Figure 10. Effect of nanoparticles on dye

Figure 11. Effect of dye concentration

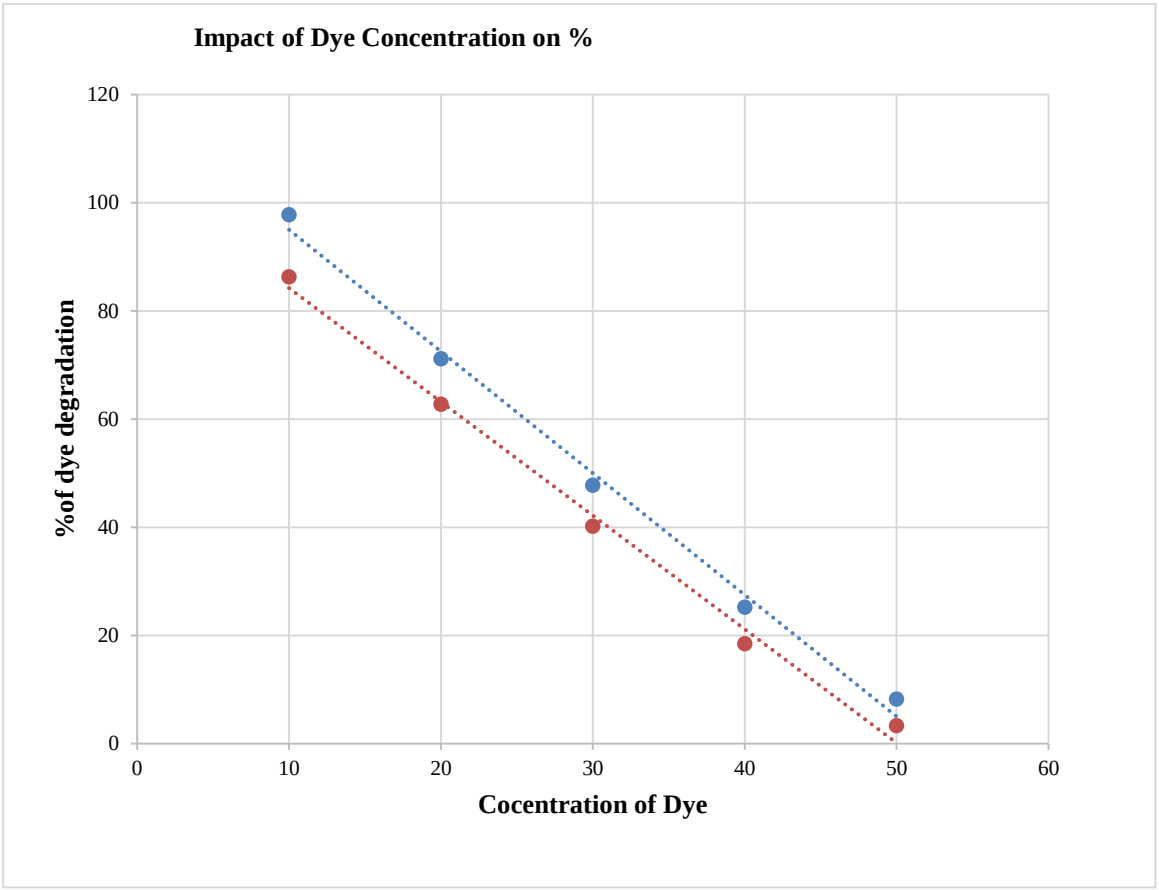

Figure 12. Impact of dye concentration on % removal.

Table 1. Zeta potential characterization

Properties	Values
Target temperature	15.0°C
Equilibration time	0h 01m 00s
Henry factor	1.5 (Smoluchowski)
Adjusted voltage	200.0V (Automatic mode)
Processed runs	Zeta potential distribution
Solvent	Water
Solvent refractive	1.3311
Solvent viscosity	0.001137

Table 2. Results of zeta potential

Properties	Values
Mean zeta potential	-9.9Mv
Standard deviation	0.8mV
Distribution peak	-11.9 mV
Electrophoretic mobility	-0.6353 µm*cm/Vs
Mean intensity	198.7 kcounts/s
Filter optical density	0.5006
Conductivity	0.116 mS/cm
Transmittance	59.8%

ZnO nanoparticles showed ~98% degradation in 120 minutes upon UV exposure at a concentration of 10 ppm, implying the best interaction with dye molecules [19]. To clean more pollutants, increasing dye concentration would make carefully ZnO lose its self-cleaning properties. To obtain a high rate of degradation, percentage removal is 98% at a dye concentration of 10 ppm after 2 h for ZnO as optimum dosage compared to TiO_2. Which achieved only of 16 % when the time was increased shown in Figure 12 [25]. Reactivity also evaluated by photo catalytic activity of ZnO nanoparticles in suspensions with an equally wide range for Reactive Blue 13 dye concentrations (10−50 ppm). Results indicated that higher dye concentrations have a negative effect on color removal efficiency because of high optical density nature of solution, which irradiates UV light and limits catalyst activity. At higher amounts of dye molecules, exchangeable surface sites saturated in ZnO nanoparticles, and thus, complete active site filling becomes significantly reduced for slower rate kinetic removal as observed. Further, process is non-linear, which could potentially suggest that optimizing ZnO nanoparticle concentrations might help improve degradation performance [26].

4. Conclusion

The waste from textile industry contributes a great deal too worldwide water pollution, making up 17-20% of industrial water contamination. Nevertheless, photo catalytic dye decolorization using nanoparticles like ZnO and TiO_2 can provide a cheap and environmentally friendly remedy. Experiments indicated that ZnO and TiO_2 could effectively degrade Reactive Blue 13 dye under various conditions; with ZnO having an efficiency of over 98%. In addition, the study points out that nano-TiO_2 is a photo catalyst for removing reactive dyes 86% from water thus giving an insight into how it may be used in wastewater treatment and environmental remediation. This research looks at efficiency of nano TiO_2 in eliminating reactive dye from water, evaluates its photocatalytic activity, compares it with ZnO NPs resulted that TiO_2 removed the reactive dye is 86% and ZnO degraded this dye is 98% at optimizes conditions for enhanced removal efficiency. The article demonstrates potential application of nano TiO_2 and ZnO as an effective tool in preventing pollution together with emphasizing its possible advantage in wastewater treatment.

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Conflicts of interest: There are no conflicts to declare.

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