



Synthesis of Nano-Photocatalyst ZnO-Natural Zeolite to Degrade Procion Red

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Abstract. The development of textile factories has had a significant impact on the environment because dye wastewater is difficult to degrade. The handling of textile wastewater usually involves the adsorption method, in which pollutants only transfer to another phase. As an alternative solution, a photodegradation method was developed using photocatalyst material assisted by ultraviolet irradiation. In this photodegradation method, dye substances are broken down into components that are simpler and safer for the environment. The possibility of using sunlight as a radiation source makes this a particularly promising method for countries such as Indonesia. Procion red, one of the most commonly utilized synthetic dyes in the home textile or Batik industry, was used as a model pollutant in this study. The objectives of the present research were to synthesize and characterize a nano-photocatalyst of ZnO-natural zeolite with the capacity to degrade Procion red. This nano-photocatalyst was synthesized using the sol-gel method, by which $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ was used as a precursor and combined with natural zeolite to increase its photocatalytic ability. The nano-photocatalyst was characterized using X-ray diffraction (XRD), scanning electron microscope/energy dispersive X-ray spectroscopy (SEM/EDX), and Brunauer-Emmett-Teller (BET). The activity of nano-photocatalyst ZnO-natural zeolite was compared to ZnO alone and natural zeolite alone in terms of its ability to degrade Procion red. An average photocatalyst crystallite diameter of 82 nm was achieved, proving that a nano-sized photocatalyst of ZnO-natural zeolite was successfully synthesized. BET characterization showed that nano-photocatalyst ZnO-natural zeolite had a specific surface area of 14.84 m²/g, a pore size of 32.71 nm, and a pore volume of 0.12 cm³/g. A peak Procion red degradation percentage of 96.23% was obtained using nano-photocatalyst ZnO-natural zeolite after 120 minutes of irradiation under sunlight applied to a Procion red concentration of 50 ppm.

Keywords: Nano-photocatalyst; Natural zeolite; Photodegradation; Procion red; ZnO

1. Introduction

The development of the textile industry has certainly had an impact on the environment, as the dye wastewater produced by this industry is difficult to degrade. Dye-containing wastewater usually consists of non-biodegradable organic compounds that can contribute to pollution, especially in aquatic environments. However, the wastewater produced by the textile industry also contains synthetic dyes that are harmful to the environment. Procion red is one of the most commonly used synthetic dyes in the home textile or Batik industry. This synthetic dye is difficult to degrade due to the strong covalent bonds between the carbon atoms of the dye stuff and the O, N, or S atoms of the hydroxy,

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amino, or thiol groups of the polymer (Hunger, 2003).

Degradation methods, such as coagulation-flocculation (Rusydi et al., 2016) and a combination of electrocoagulation and photocatalysis (Sharfan et al., 2018), have been developed for dyes in the textile industry. However, these chemical processing methods apply large quantities of chemicals and produce sludge, which must be separated at the end of process. Membrane applications are quite effective but require significant operational costs and have the disadvantage of introducing new problems, such as the production of compounds with more concentrated pollutant levels. A photodegradation method using photocatalyst materials and ultraviolet light irradiation has been developed as an alternative method (Agustina et al., 2015; Zhao et al., 2015). Among modern methods of dealing with wastewater, photodegradation is relatively inexpensive and easy to implement. Widely studied photocatalysts include metal oxide semiconductors, such as ZnO and TiO₂. The ZnO semiconductor has an advantage over TiO₂ because it is able to absorb the UV spectrum of the sun and quantum of light more readily than TiO₂. In addition, ZnO is an efficient photocatalyst material for the detoxification of wastewater because it produces H₂O₂ more efficiently than other photocatalysts (Hidayanto et al., 2013; Mydeen et al., 2019). ZnO is widely used due to its cost-effectiveness, high oxidation ability, and wide availability. However, electron-hole recombination can block the photocatalytic activity of pure ZnO.

Research has been carried out to investigate the pure photocatalytic activity of semiconductors, such as ZnO, in degrading organic compounds, such as synthetic dyes. Photocatalyst activity can be increased through the development of supporting materials, such as natural zeolites. Natural zeolite, a cheap and locally available absorbent, has been used in the energy field and for many applications such as additives, catalysts, ion exchangers, and absorbents (Millar et al., 2016; Papa et al., 2017; Wulandari et al., 2019). Zeolite is used as a carrier molecular because its crystal structure is porous and has a large surface area, it is composed of a silica-alumina skeleton, it has a high thermal stability, and its presence is quite abundant (Wang and Peng, 2010). The pore structure of zeolite also allows it to be used as a host material (Rahman et al., 2018). Moreover, zeolite is recommended for the degradation of dye through adsorption due to its large surface area (Viswanathan, 2018). The surface area and acidity of natural zeolite could be modified easily (Suhartana et al., 2018; Wulandari et al., 2019). The present study utilized natural zeolite from West Java.

Many efforts have been made to enhance the photocatalytic performance, especially in dye degradation. Such efforts have involved making ZnO-zeolite composites by impregnation methods (Salam et al., 2018), doping ZnO nanoparticles with natural zeolite (Rahman et al., 2018), and adding noble metals, such as Ag nanoparticles (Kusdianto et al., 2019) and Fe₃O₄ (Winatapura et al., 2016). ZnO has a low capacity for adsorption as a photocatalyst, but ZnO photocatalysts can be combined with adsorbents, such as zeolite, to face this problem (Wismayanti, 2015). Therefore, the present study aimed to synthesize photocatalysts of nano-sized ZnO-natural zeolites in order to utilize the adsorbent properties of natural zeolites as well as the photocatalytic properties of ZnO. The obtained photocatalyst material was tested for photocatalyst activity in the degradation of synthetic dyes. Then, this activity was compared with the degradation of Procion red by standard ZnO and natural zeolites alone.

2. Methods

2.1. Materials

Hydrochloric acid (HCl), Zn (CH₃COO)₂ 2H₂O, ZnO, ethanol, and NaOH were supplied by Merck and used without any further purification. Natural zeolite was derived from West Java, Indonesia (300 mesh), and Procion red was purchased from Fajar Setia in Jakarta. The natural zeolite was activated by 1M HCl and then rinsed and dried for three hours at 220°C. The ratio for activated natural zeolite: 1M HCl was 100 g of natural zeolite : 200 mL of 1M HCl.

2.2. Synthesis of ZnO-Natural Zeolite

First, 45 g of Zn (CH₃COO)₂ 2H₂O was added to 3.32 g of activated natural zeolite and then dissolved in 80 mL of ethanol. This mixture was stirred and heated at 76°C in a reflux flask for two hours. Then, 225 mL of 2M NaOH was added to the mixture and stirred for one hour with a magnetic stirrer. The mixture was allowed to stand for 12 hours and then filtered. The resulting precipitate was heated at 60°C for 24 hours and then stored in a desiccator so that it would remain dry. The ZnO-natural zeolite composites were characterized by X-ray diffraction (XRD of Rigaku Miniflex 600), scanning electron microscope/energy dispersive X-ray spectroscopy (SEM/EDX of ZEISS EVO® MA 10), and Brunauer-Emmett-Teller (BET).

2.3. Photocatalyst Activity Test

A UV reactor system furnished with a UV lamp (Evaco 15 W 254 nm) was used in this study. A total of 100 mg of ZnO-natural zeolite photocatalyst was dispersed in 25 mL of 50 ppm Procion red in the reactor and then placed in a shaker equipped with UV light. The photodegradation process was carried out without pH adjustment. The recording time started when the shaker and the UV lamp were turned on. The experiments were repeated using Procion red of different concentrations and carried out in dark conditions as well as under sunlight. For the experiments in dark conditions, the UV lamp was left off for two hours and the shaker was covered with a box coated in black low-density polyethylene (LDPE) plastic. The experiments using sunlight were carried out on a light day between 10:00 a.m. and 2:00 p.m. Samples were taken every 30 minutes for two hours and then filtered. The concentration of the Procion red was analyzed using a UV-Vis spectrophotometer at a maximum wavelength of 539 nm. The Procion red degradation procedure was also carried out using ZnO and natural zeolite alone.

3. Results and Discussion

3.1. Characterization of Photocatalyst ZnO-Natural Zeolite

Characterization of photocatalyst ZnO-natural zeolite was carried out using SEM/EDX, XRD, and BET. Figure 1a shows the SEM image of the photocatalyst. The morphology of the photocatalyst can be seen in fine clumps. The average crystallite size (*D*) was calculated using the highest intensity from the XRD patterns presented in Figure 1b and the Scherrer formula (Equation 1) reported in previous studies (Widiyastuti et al., 2014; Vaiano et al., 2017):

$$D = \frac{k\lambda}{B \cos\theta} \quad (1)$$

where *k* is the constant (0.9), *λ* is the wavelength of the X-ray source (0.15418 nm), *B* is the full width at half maximum corresponding to the XRD peaks, and *θ* is the peak angle. The average diameter of the photocatalyst crystallite was 82 nm, proving that the synthesis of nano-sized photocatalysts of ZnO-natural zeolite was accomplished successfully. Figure 1b

demonstrates the XRD pattern of the ZnO-natural zeolite. This XRD pattern is identical to ZnO peak, which exhibited diffraction peaks at $2\theta = 31.8, 34.5, 36.4, 47.6, 56.8, 62.9,$ and 68.2 . These peaks revealed a hexagonal wurtzite structure (Balcha et al., 2016). Other peaks found at $2\theta = 9.80, 13.6, 22.38, 25.69, 26.66,$ and 27.72 represented the presence of zeolite by potassium sodium calcium aluminium silicate hydrate content (Razzak et al., 2013). The EDX results confirmed that the composite consisted of ZnO and the elements of zeolite, as shown in Figure 2. Based on the EDX results, 10.05% of ZnO was added to natural zeolite.

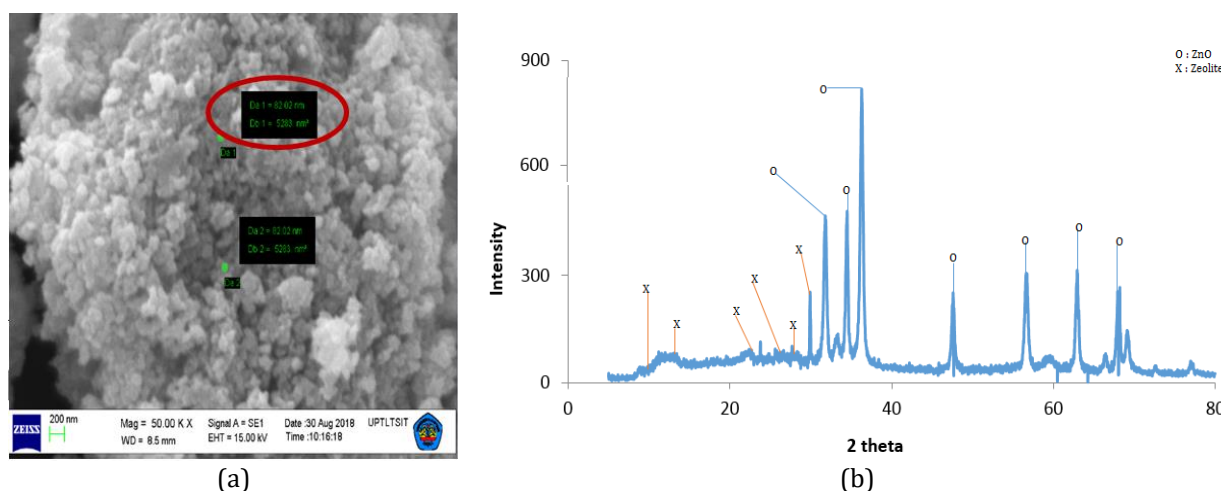


Figure 1 SEM image (a) and XRD (b) of nano-photocatalyst ZnO-natural zeolite

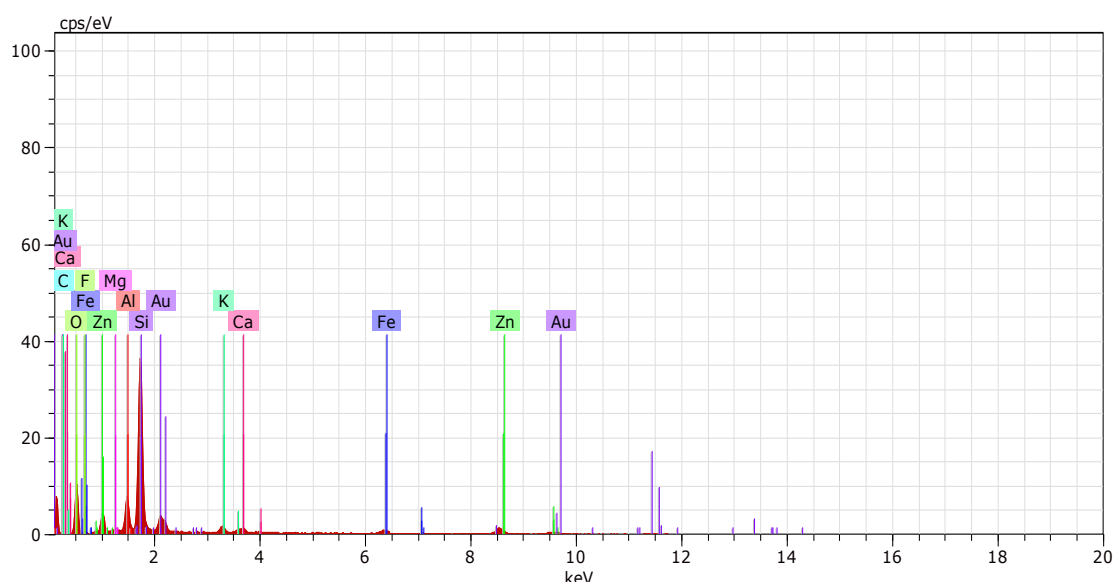


Figure 2 EDX of nano-photocatalyst ZnO-natural zeolite

Table 1 shows the results of the BET characterization. The nano-photocatalyst ZnO-natural zeolite had a lower surface area ($14.85 \text{ m}^2/\text{g}$) than the activated natural zeolite. ZnO incorporated with natural zeolites was unevenly distributed in the nano-photocatalyst ZnO-natural zeolite composite in the form of sintering or clumping. Because of this clumping, it stands to reason that ZnO entering natural zeolite would cover the pores, making the specific surface area relatively smaller (Rakhmawaty and Putra, 2011). However, the pore size of the nano-photocatalyst ZnO-natural zeolite was significantly higher ($32.71 \text{ m}^2/\text{g}$) than that of the activated natural zeolite ($4.30 \text{ m}^2/\text{g}$). Furthermore, the

pore volume of the nano-photocatalyst ZnO-natural zeolite also increased to 0.12 cm²/g. This occurred because the produced ZnO was nano in size. As the size of a particle becomes smaller, its pore size and volume increase and thus its specific surface area decreases (Pakpahan et al., 2017).

Table 1 BET characterization results

Measurement Subject	Natural Zeolite	Activated Natural Zeolite	Nano-Photocatalyst ZnO-Natural Zeolite
Specific Surface Area (m ² /g)	23.40	26.19	14.85
Pore Size (m ² /g)	7.39	4.30	32.71
Pore Volume (cm ² /g)	0.04	0.03	0.12

3.2. Degradation of Procion Red Synthetic Dye using a Nano-Photocatalyst

In order to test the activity of nano-photocatalyst ZnO-natural zeolite, it was used to degrade Procion red synthetic dye. For the sake of comparison, natural zeolite and ZnO were also tested separately for their abilities to degrade the synthetic dye. The concentrations of the synthetic dye ranged from 50 to 250 ppm. The highest degradation percentage of 96.23% was obtained using a Procion red concentration of 50 ppm after 120 minutes of irradiation under sunlight, as demonstrated in Figure 3. At the same time, degradation percentages of 39.31% and 91.21% were obtained using zeolite and ZnO, respectively. High degradation percentages were also consistently obtained using nano-photocatalyst ZnO-natural zeolite at 30, 60, and 90 minutes of degradation (93.20%, 93.47%, and 95.81%, respectively). This finding shows that the catalytic ability of nano-photocatalyst ZnO-zeolite assisted by sunlight irradiation is more effective than natural zeolite or ZnO alone. In terms of particle size, smaller particles have greater surface areas, resulting in greater interaction on the particle surface (Naimah et al., 2011). Therefore, the inclusion of nano-sized ZnO in the photocatalyst improved its function. Even within the first 30 minutes of exposure, the degradation percentage reached more than 90%.

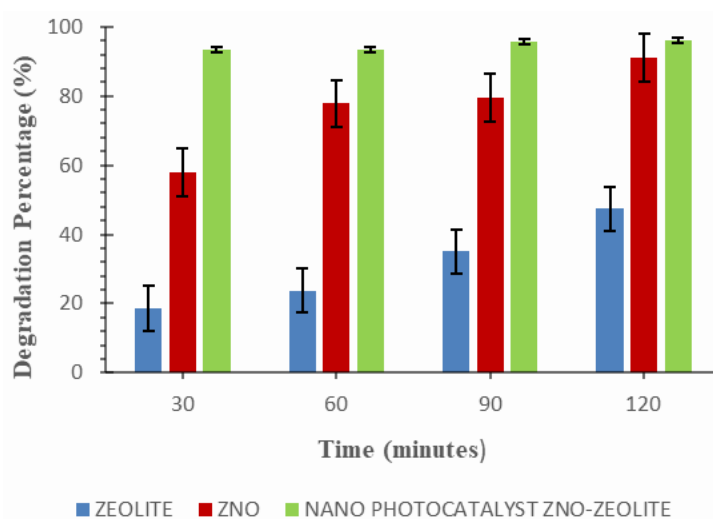


Figure 3 Degradation of 50 ppm Procion red using sunlight

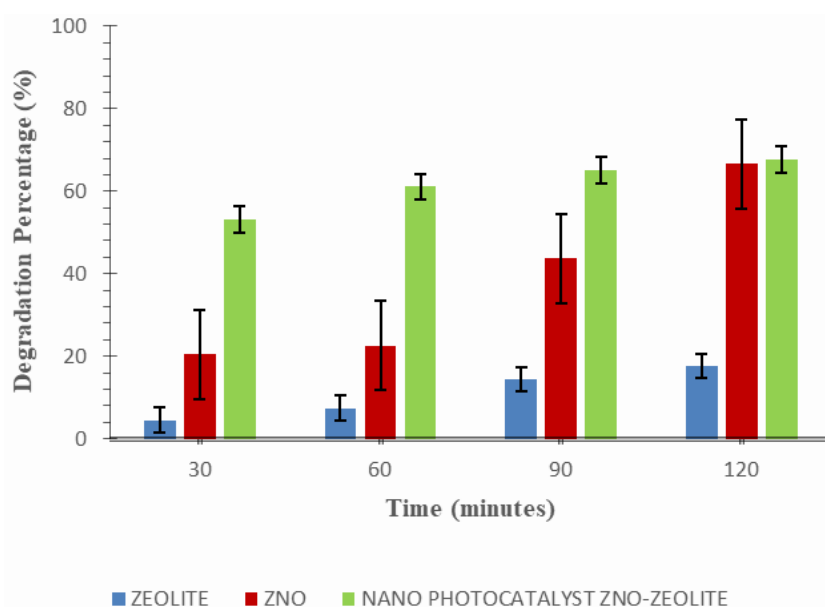


Figure 4 Degradation of 250 ppm Procion red using sunlight

Figure 4 shows that the highest percentage of 250 ppm Procion red degradation using sunlight (67.72%) was achieved at 120 minutes with nano-photocatalyst ZnO-natural zeolite, whereas zeolite and ZnO alone only accomplished 17.61% and 66.71% degradation at this point, respectively. At 30, 60, and 90 minutes, peak degradation percentages of 53.22%, 61.13%, and 65.09%, respectively, were obtained using nano-photocatalyst ZnO-natural zeolite. These results also showed that nano-photocatalyst ZnO-natural zeolite degraded Procion red more effectively than either ZnO or zeolite alone. Overall, nano-photocatalyst ZnO-natural zeolite produced the best degradation results for each test condition.

To determine the effect of the dye concentration, Procion red degradation was studied in the concentration range of 50–250 ppm. However, for the application of a photocatalyst in real conditions under sunlight irradiation, this paper only reported the use of the highest concentration of 250 ppm, which is closest to actual wastewater characters. The percentage of degradation obtained with 250 ppm Procion red using sunlight irradiation is shown in Figure 4. The highest degradation percentage (67.72%) was achieved at 120 minutes using nano-photocatalyst ZnO-natural zeolite, whereas zeolite and ZnO alone only achieved 17.61% and 66.71% degradation at this point, respectively. At 30, 60, and 90 minutes, peak degradation percentages of 53.22%, 61.13%, and 65.09%, respectively, were obtained using nano-photocatalyst ZnO-natural zeolite. However, the percent degradation obtained using 250 ppm Procion red was lower than that obtained using 50 ppm Procion red, likely because 250 ppm Procion red contains a higher concentration of chemicals to be decomposed. As shown by Figures 3 and 4, increasing the Procion red concentration from 50 ppm to 250 ppm caused the degradation percentage to decrease from 96.23% to 67.72% or 28.51% when photocatalyst ZnO-natural zeolite was applied after 120 minutes of irradiation. These results were in accordance with the results of a study conducted by Winatapura et al., in which photodegradation efficiency decreased from 100% to 52% when the Methylene blue dye concentration increased from 10 to 40 ppm (Winatapura et al., 2016). In the present study, increasing the Procion red concentration from 50 to 250 ppm decreased the average degradation percentages by 20.23%, 39.54, and 32.88% for the application of zeolites alone, ZnO alone, and natural ZnO-zeolite photocatalysts, respectively.

The photocatalytic process is less effective for treating high concentrations of waste because the adsorption ability of photocatalysts is limited; therefore, increased waste concentrations cause a decrease in the rate of photocatalytic reactions (Takeda et al., 1995). This principle is in accordance with the results of the present study; for each test condition, greater concentrations of Procion red corresponded to lower percentages of degradation. The rate of dye degradation is related to the formation of OH radicals ($\bullet\text{OH}$), which are the most important species in the degradation process. An increase in the initial concentration of Procion red causes the longer the photon path to irradiate the solution so that the photon reaching the catalyst are reduced, thereby reducing the rate of degradation. Therefore, the concentration of Procion red can affect the rate of dye degradation.

Interestingly, the photocatalyst reached optimum activity within the first 30 minutes of application to the lowest concentration of Procion red (50 ppm) for all test materials. However, the degradation percentages only slightly increased from 30 to 120 minutes of natural zeolite or ZnO application, as shown by Figure 3. In the degradation of Procion red at a high concentration (250 ppm), the use of photocatalyst ZnO-natural zeolite was not optimal because the percent degradation rose only slightly until 120 minutes of reaction time; the same percent degradation was achieved by ZnO alone, as demonstrated in Figure 4. This finding should be taken into consideration in choosing the best material to degrade high-concentration dyes with the aid of abundant sunlight in a country such as Indonesia.

3.3. The Effect of Irradiating Conditions on Procion Red Degradation

To study the effects of irradiating conditions, the nano-photocatalyst was applied under three different types of irradiation. These test conditions were distinguished using UV lamps, sunlight, and a dark place as a control. The results of these tests are illustrated in Figure 5 below. The lowest concentration of Procion red solution (50 ppm) yielded the best degradation results under sunlight. This result is in agreement with the results of Salam et al. (2018), who synthesized ZnO-zeolite composites by the impregnation method and studied the effects of different light conditions (dark room, UV light, and sunlight) on the degradation of Procion red. Salam et al. (2018) found that Procion red degradation by sunlight radiation (83.96% after 120 minutes) was greater than degradation under a UV lamp or in a dark room.

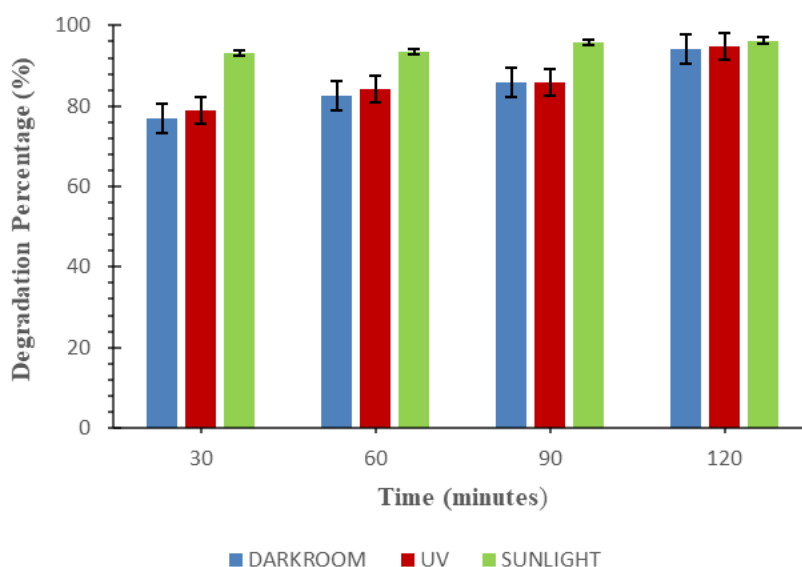


Figure 5 Degradation of 50 ppm Procion red under different light conditions

The degradation of Procion red by the nano-photocatalyst and by ZnO after exposure to sunlight (approximately 555,263.64 lux) was greater than that produced under UV lamps or dark conditions. Exposure to sunlight produces faster color degradation rates, as the high energy levels emitted by UV and light rays appear to be able to excite electrons from chromophore groups (Fraditasari et al., 2015). Sunlight radiation also produces the highest degradation because the intensity of sunlight is much greater than the intensity of reactor-mounted UV light (Chatti et al., 2007). Sunlight also has a wider wavelength, encompassing approximately 40% of visible light and 3% of ultraviolet light. The number of visible rays reaching the active side of the photocatalyst increases the formation of hydroxyl radicals available for the photodegradation of Procion red (Charanpahari et al., 2012).

4. Conclusions

Nano-photocatalyst ZnO-natural zeolite was synthesized using $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ precursors and activated natural zeolite. Characterization of this photocatalyst showed that it was formed with an average crystallite size of 82 nm, a specific surface area of 14.84 m^2/g , a pore size of 32.71 nm, and a pore volume of 0.12 cm^3/g . The use of nano-photocatalyst ZnO-natural zeolite produced the highest synthetic dye degradation percentage (96.23%) in the degradation of 50 ppm Procion red under 120 minutes of sunlight irradiation.

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