

Photocatalytic degradation of industrial dye wastewater in presence of ZnO-PEG nanoparticles

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ABSTRACT

Dye wastewater is one of the very dangerous pollution sources that can harm millions of human lives and caused critical effect to the eco-system. One of the beneficial methods for treating dye wastewater is photocatalysis process due to its quick oxidation of pollutants up to the parts per billion (ppb) level and it is proved that this process does not produce any polycyclic product. Zinc oxide (ZnO) has been claimed as the excellent photocatalyst in photocatalytic degradation process of dyes because of its high photo sensitivity, chemical stability and broad range of radiation absorption. However, agglomeration in the synthesis of ZnO is a serious problem because photocatalytic activity of ZnO is influenced by particle size, shape and surface morphology. Therefore, polyethylene glycol (PEG) is needed to control the shape and particle size of ZnO since it revealed the maximum coagulation concentration and critical stabilization concentration in the stabilizing process of the gold nanoparticles. From the studies, the optimum loading of PEG in precipitation of ZnO nanoparticles had successfully assess by comparing the effectiveness of all type of ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG) in photocatalytic degradation of dye wastewater. Hence, 0.015 g/L PEG loading in ZnO nanoparticles has the most optimum of PEG loading due to the highest degradation percentage of dye removal which is 95.48%. pH 7 was observed to be the optimum condition since it revealed the highest dye degradation percentage which is 95.48%. Hence, it can be concluded that with the presence of ZnO-PEG nanoparticles as a photocatalyst could greatly remove the dye particles in wastewater.

Keywords: Industrial dye wastewater, photocatalytic degradation, ZnO nanoparticles.

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1. INTRODUCTION

Dye wastewater is the major environmental issue of the textile and printing industries besides other minor issues like solid waste, health and safety. The use of synthetic chemical dyes in various manufacturing industrial process has increased considerably over the past decades, resulting in the release of dye-containing industrial effluents into aquatic ecosystem and soil [1]. Wastewater from printing and dyeing industries is frequently rich in color, containing residues of toxic chemicals, reactive dyes and requires proper treatment before all the effluent release into environment [2]. Many approaches have been used including physical and chemical processes in the industrial dye wastewater such as activated carbon and activated bentonites utilizing methods and ozone

treatment. However, almost all these methods are often very costly, generate the large amount of sludge, not environmentally safe and not all the treatment employed give satisfactory outcome especially for several type of dispersed dyes [1].

One of the beneficial methods is photocatalysis process, which is an emerging technology of dye wastewater treatment. This is due to its quick oxidation of pollutants up to the parts per billion (ppb) level and it is proved that this process does not produce any polycyclic product [3]. Various nanoparticles have been used in photocatalysis research including titanium dioxide (TiO₂), zinc oxide (ZnO), tin dioxide (SnO₂), zinc sulphide (ZnS), cadmium sulphide (CdS), iron oxide (Fe₂O₃), etc. for various types of wastewater treatment. However, zinc oxide (ZnO) has been claimed as the excellent photocatalyst in photocatalytic

degradation of dyes because of its high photo sensitivity, chemical stability and broad range of radiation absorption [4].

Zinc oxide (ZnO) nanoparticles were synthesized by various method depending on the control of both physical and chemical properties such as shape, surface state, size, size dispersity, crystal structure, organization onto a support and dispensability [5-9]. Due to the various method that have been implemented, precipitation has been recognized as the great techniques for synthesizing the compound since it provided a simpler route, economic and occurs at a moderately low temperature [4]. However, agglomeration in the synthesis of ZnO is a serious problem because photocatalytic activity of ZnO is influenced by particle size, shape and surface morphology.

Polyethylene glycol (PEG), also known as poly (ethylene oxide) (PEO) or poly (oxyethylene) (POE), is a synthetic polyether of ethylene glycol. PEG is one of the interesting compounds where it is can act as solvent in organic reactions. They are inexpensive and significantly less hazardous compared to other organic solvents. Additionally, PEGs are stable under ambient condition because they have negligible vapor pressure and do not release volatile organic compound (VOCs). Besides, they have great stability in both acidic and basic media and are suitable reaction media for oxidation/reduction reactions [10]. In addition, it has been reported that the polyethylene oxide chains of the polyethylene glycol (PEG) can form complexes with metal cations in solution, resembling crown ethers [11].

According to Parra & Haque (2015), non-ionic polymer PEG with uniform ordered chain structure is easily absorbed at the surface of metal oxide particles. Based on their research, it was clearly observed from the results that polymer adsorbed on the surface of the ZnO nanoparticles as a covering layer for their structural modifications. The changes in length as well as width of ZnO nanorods is controlled by the involvement of PEG in the reaction procedure. They conclude that the addition of PEG in the reaction system will changes the kinetics of the growing process, which is the feature characteristic to the rapid growth of nucleation and cause the aggregation of nanoparticles [12-13]. Therefore, it is believed that the presence of PEG in precipitation of ZnO nanoparticles could reduce the agglomeration which leads to reduce the particle size of ZnO nanoparticles and increase its photocatalytic performances of dye wastewater degradation by using photocatalysis process. Therefore, polyethylene glycol (PEG) is needed to control the shape and particle size of ZnO since it revealed the maximum coagulation concentration and critical stabilization concentration in the stabilizing process of the gold nanoparticles [6].

Previously, Hairom et al., (2014) confirmed that the effluent chemical properties from the industrial newsprint wastewater (UMWW) was successfully treated using membrane photocatalytic reactor (MPR) with utilization of

self-synthesis ZnO nanoparticles via precipitation method. It has been claimed that the chemical properties of effluent were improved in the presence of ZnO-PVP-st, as the photocatalyst in the membrane photocatalytic reactor (MPR). However, recent study has proved that PEG has attracted special attention as a green and inexpensive solvent in various chemical transformation. Therefore, this study aims to investigate the effectiveness of ZnO nanoparticles in presence of PEG for preventing the agglomeration between ZnO nanoparticles. The ZnO-PEG nanoparticles will be used as photocatalyst for industrial wastewater treatment under various parameters in order to obtain the optimum condition of the wastewater treatment process and successfully preserve the environment for future generation.

2. EXPERIMENTS

2.1 Wastewater sample

A dye wastewater sample was collected from the influent sump tank of the central wastewater treatment plant of a printing industry in Selangor, Malaysia.

2.2 Synthesis and characterization of ZnO-PEG nanoparticles

ZnO-PEG nanoparticles was synthesized by precipitation method in a manner corresponding to Hairom and co-workers work [4]. 0.15M of oxalic acid solution and 0.1M of zinc acetate solution will be mixed under stirring condition. Different amount of Polyethylene glycol (PEG), (0.015 g/L, 0.020 g/L and 0.025 g/L) was added into mixture after 5 minutes of reaction. Then, the mixture was stirred for 12 hours under room temperature. The reaction yields precipitate which was filtered and dried at 100°C in oven for 1 hour to remove the excessive water. Subsequently, the precipitate was calcined in furnace under 550°C for 3 hours to remove all the impurities. Eventually, the white powder of ZnO-PEG was formed and ready to characterize. Fourier Transform Infrared Spectroscopy (FTIR) (Nicolet FT-IR Avatar 360) in the wavelength range 400-4000 cm^{-1} in order to analyze its purity and chemical bonds.

2.3 Photocatalytic degradation process

Photocatalytic degradation experiments were carried out by preparing the basic part of the apparatus which is conical flask (1000 ml) used as a photocatalytic reactor in batch method. The UV lamp was placed on top of the photocatalytic reactor to activate the photocatalyst. The dye wastewater was poured into the reactor to react with ZnO-PEG nanoparticles before the photocatalysis process started. The mixture of dye wastewater and ZnO-PEG nanoparticles were well agitated by magnetic stirrer (150 rpm). The photocatalytic reactor was placed in the water bath throughout the experiment to maintain the temperature at

25°C. Sodium chloride (NaCl) and sodium hydroxide (NaOH) (purchased from R&M marketing, Essex, UK) solutions were used to adjust the solution pH that will be measured by pH meter (Eutech Model). At the interval of 5 minutes, 10 ml of degraded wastewater was sampled and the mixed solution (dyes and ZnO) was separated by using centrifuge (Eppendorf, 5804 model) under 300 rpm for 20 minutes. The treated water quality was analyzed by using the analytical method. The colour intensity was observed by using UV-Vis Scanning Spectrophotometer (Spectro UV-2650, Labomed,inc.). Dissolved oxygen and turbidity were measured by using H-series benchtop meters (H280G, HACH). pH value of the treated water was determined with pH meter (Eutech Model).

3. RESULTS AND DISCUSSION

3.1 Synthesis and characterization of ZnO-PEG nanoparticles

According to the experimental procedures, zinc oxide nanoparticles were successfully synthesized by using precipitation method in the presence of polyethylene glycol (PEG) as the capping agent (Fig. 1). In this study, three types of zinc oxide (ZnO) nanostructure were synthesized with different loading of PEG and produce the following nanoparticles:

- i) ZnO-PEG (0.015 g/L of PEG)
- ii) ZnO-PEG (0.020 g/L of PEG)
- iii) ZnO-PEG (0.025 g/L of PEG)

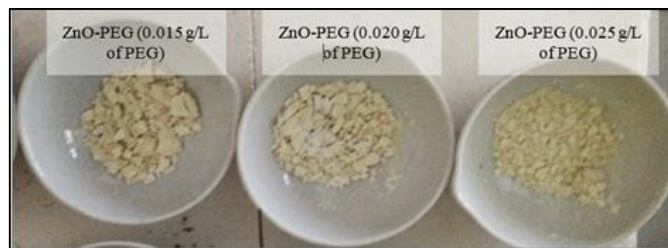


Fig. 1: ZnO-PEG obtained from precipitation methods under different PEG loading.

Characterizations of the ZnO-PEG nanoparticles were conducted by using Fourier Transform Infrared Spectroscopy (FTIR - Perkin-Elmer Model) to ascertain the purity, nature and chemical bonds in ZnO-PEG nanoparticles by producing an infrared absorption spectrum. The purity, nature and chemicals bonds of ZnO-PEG nanoparticles were shown in Fig. 2, 3 and 4. Fig. 2 indicates the FTIR pattern of ZnO-PEG (0.015 g/L of PEG). Prominent absorption peaks for ZnO-PEG (0.015 g/L of PEG) were obtained at 3413.14 and 877.55 cm⁻¹. The peaks at 3413.14 cm⁻¹ are correspond to Zn-O stretching and deformation vibration while the peak observed at 877.55 cm⁻¹ indicates the O-H stretching and deformation due to the

adsorption of moisture from surrounding during synthesizing the photocatalyst [14].

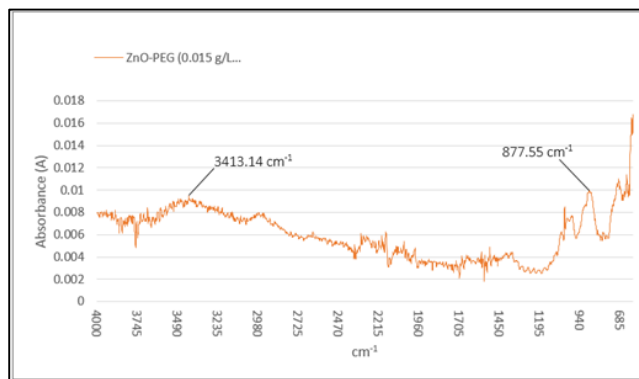


Fig. 2: FTIR pattern for ZnO-PEG (0.015 g/L of PEG)

FTIR patterns for ZnO-PEG (0.020 g/L of PEG) in Fig. 3 shows the prominent absorption peaks at 876.86, 1559.75 and 3390.12 cm⁻¹. The peaks at 1559.75 and 3390.12 cm⁻¹ were appeared to be a very short band Zn-O band. The peak at 876.86 cm⁻¹ indicates the deformation and stretching of O-H. In the case of ZnO-PEG (0.025 g/L of PEG), the several well-defined peaks were obtained at 1033.21, 1653.04 and 3405.49 cm⁻¹ that shown in Fig. 4. The peaks at 1033.21, 1653.04 and 3405.49 cm⁻¹ are corresponding to Zn-O deformation vibration and stretching and there was no other compound detected on the sample ZnO-PEG (0.025 g/L of PEG) [14-15]. In addition to the observed peaks, no other peak related with any functional group was detected in the spectrum which reveals that the synthesized of ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG) were successfully achieved without any impurities [16].

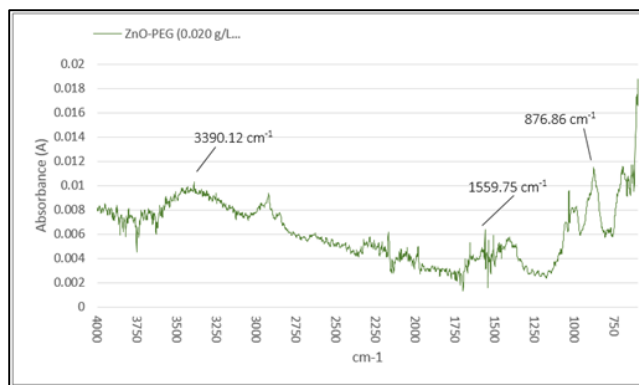


Fig. 3: FTIR pattern for ZnO-PEG (0.020 g/L of PEG)

3.2 Effect of different pH

In industrial dye wastewater treatment, preliminary or primary treatment may include pH adjustment, flow equalization or chemical addition which is extremely

important to the overall treatment process. It is very important to regulate pH since treatment processes can be harmed by dangerous acidic or basic wastes.

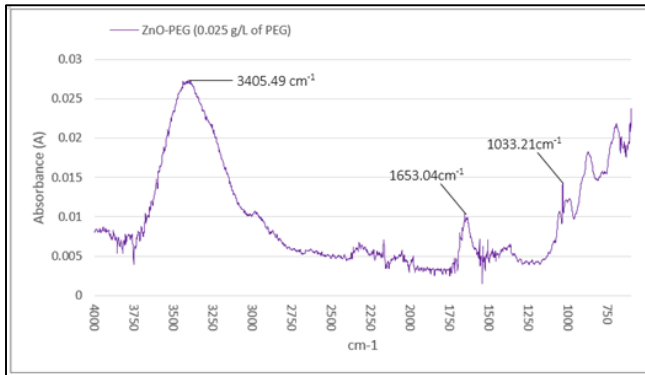


Fig. 4: FTIR pattern for ZnO-PEG (0.025 g/L of PEG).

Regulation of this parameter may be necessary to meet effluent levels specified for secondary treatment. In general, pH is a crucial characteristic of wastewater because it determines degradation efficiency. In order to study the effect of pH on photocatalytic activity of ZnO-PEG (0.015g/L of PEG), the photocatalytic activity has been studied in different pH condition (pH 4, 7 and 9). Fig. 5 shows the pattern of the dye degradation percentage under different pH condition in 40 minutes duration.

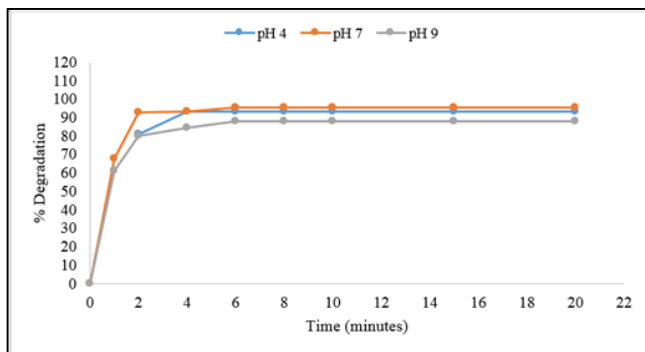


Fig. 5: Percentage of dye degradation versus time for photocatalysis process in presence of ZnO-PEG (0.015g/L of PEG) under different pH.

Based on the result obtained from Figure 5, photocatalysis process under pH 7 indicate the highest percentage of dye degradation which is 95.48% in contrast to pH 4 and pH 9. However, the dye removal become constant after minute 6. In basic and acidic solution (pH 4 and pH 9), the percentage of dye degradation is lower but reaches more than 90% of dye removal and become constant before the minutes reach 10. The results showed that the pH significantly affected the degradation efficiency. Point of zero charge (PZC) on photocatalyst’s surface plays a vital

role in photocatalytic performance where minimum interaction between photocatalyst and dye due to the absence of electrostatic force. For example, at lower pH, the degradation of organic molecules is lower due to the increasing of positive charge on the photocatalyst surface [18]. In the present study, all the reactive dyes have been degraded efficiently at neutral or near neutral conditions. However, alkaline conditions have generally inhibited the rate of photocatalytic degradation. While the lowest dye removal at acidic conditions is due to the photodecomposition of ZnO to Zn²⁺ [15]. Hence, it can be concluded that pH 7 is the optimum condition for degrading dye particles since it show the highest percentage of dye degradation.

Fig. 6 demonstrate the percentage of turbidity removal during photocatalysis process in presence of ZnO-PEG (0.015 g/L of PEG) under different pH in 40 minutes duration. The turbidity of dye wastewater was found to be in the range of 690–700 NTU and 83.9 – 244 NTU for before and after treatment, respectively. Photocatalysis process under pH 7 demonstrate the highest percentage of turbidity removal (87.84%) and become constants at 20 minutes meanwhile, pH 9 shows the lowest percentage of turbidity removal (63.64%). There was a significant positive correlation between the results with the previous explanation of Fig. 5. The highest percentage of turbidity removal that shown in the Fig. 6 was supported by result shown in the highest percentage of dye degradation in Fig. 5. Both of the figure indicates that the photocatalytic degradation under neutral pH (pH 7) is the optimum pH condition for dye degradation.

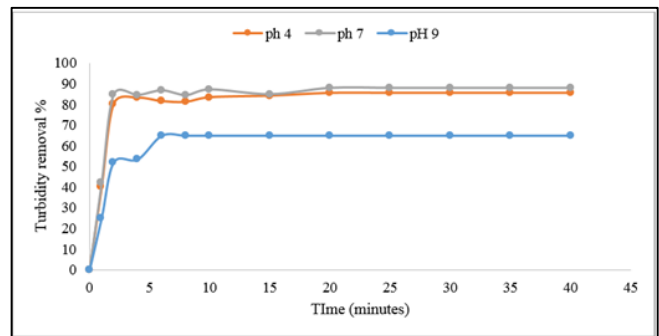


Fig. 6: Percentage of turbidity removal versus time for photocatalysis process in presence of ZnO-PEG (0.015g/L of PEG) under different pH.

Fig. 7 shows the effect of different pH condition (pH 4, 7 and 9) on the concentration of dissolved oxygen (DO) in 40 minutes photocatalysis process. The dissolved oxygen (DO) control is the most widely-spread in real-life, since the DO level in the aerobic reactors has significant influence on the behavior and activity of the heterotrophic and autotrophic microorganisms living in the activated sludge [17]. Based on the Fig. 7, all of the DO reading for all

sample is in the range 0.12 to 7.94 ppm. The DO reading of the samples under pH 4 and pH 9 is in the range 5.17 to 7.94 ppm while the DO reading under pH 7 is in the range 0.12 to 1.18 ppm. DO value under pH 7 had the lowest reading and it is believed that the photocatalysis process under pH 7 were consuming a lot of oxygen molecules to degrade the dye. This statement is supported by Fig. 5 in which it shows that the photocatalytic degradation under pH 7 had the highest percentage of dye degradation. According to the National Water Quality System (NWQS), the acceptable range of DO value after wastewater treatment is in the range 0.02 to 20 mg/L [18]. Therefore, the treated wastewater could be discharged to environment and safe to aquatic life. From the analyzed results, it could be deduced that the treated wastewater is safe to discharge to environment since their DO value is in the acceptable range.

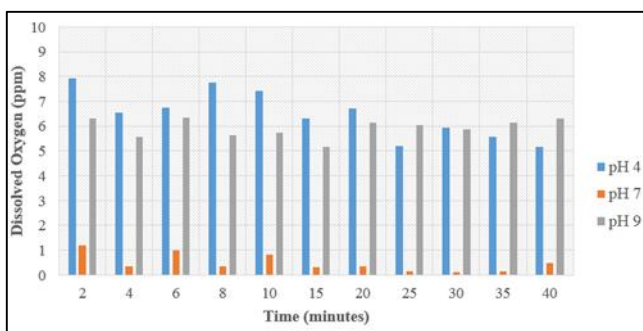


Fig. 7: Effect of different pH on dissolved oxygen versus time in presence of ZnO-PEG (0.015 g/L of PEG).

3.3 Effect of different photocatalysts

Fig. 8 shows the percentage of dye degradation in presence of different photocatalysts, ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG). The amount of ZnO-PEG loading was optimized to avoid waste while ensuring maximum dye degradation. Therefore, the effect of various ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG) on dye degradation was analyzed in Fig. 8.

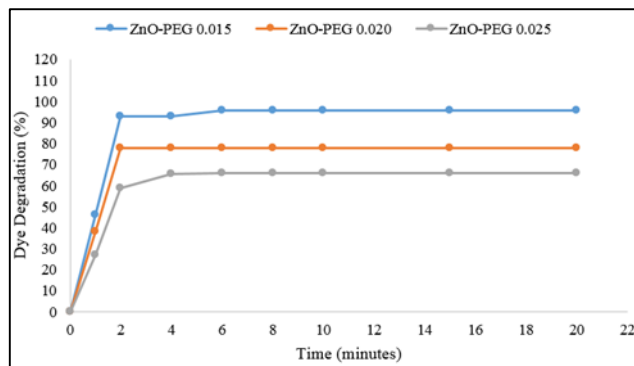


Fig. 8: Percentage of dye degradation versus time for photocatalysis process under different photocatalysts, ZnO-PEG (0.015, 0.020 and 0.025 g/L).

The photocatalysis process was carried out under the most optimum pH (pH 7) to degrade maximum dye particles. Based on Fig. 8, it is obvious that ZnO-PEG 0.015 g/L is the most effective photocatalyst to degrade the dye wastewater since it has the highest amount of dye degradation percentage (95.48%). However, the dye degradation become constant as the time reached 6 minutes. Fig. 8 also shown that the percentage of dye degradation with addition of ZnO-PEG 0.020 and 0.025 g/L are slightly less effective than ZnO-PEG 0.015, 77.85% and 65.88% respectively. Further increase of ZnO-PEG loading decreases the removal rate and this may cause by screening effect and reduce the specific activity of the catalyst. At high loadings of ZnO-PEG, the aggregation between particles may also reduce the catalytic activity. Hence, the optimum amount of ZnO-PEG loading is found to be ZnO-PEG (0.015 g/L of PEG).

Fig. 9 shows the pattern of turbidity removal percentage under different photocatalysts (ZnO-PEG 0.015, 0.020 and 0.025 g/L). Based on the Fig. 9, the highest percentage of turbidity removal for dye wastewater in the presence of ZnO-PEG (0.015 g/L of PEG), ZnO-PEG (0.020 g/L of PEG) and ZnO-PEG (0.025 g/L of PEG) is 87.1%, 58.55% and 48.98% respectively. ZnO-PEG (0.020 g/L of PEG) have the fastest time of turbidity removal which is 2 minute and become constant for the rest of the photocatalytic process. However, the turbidity removal in presence of ZnO-PEG (0.015 g/L of PEG) have the highest turbidity removal and the reading become constant after 10 minutes. This result is supported by previous analysis in Fig. 6. In addition, by increasing the ZnO-PEG loading may cause the screening effect and reduce the specific activity of the catalyst. Therefore, the greatest effect of turbidity was obtained at ZnO-PEG (0.015 g/L of PEG) [16]. According to Fig. 8 and Fig. 9, it was concluded that with the presence of ZnO-PEG (0.015 g/L of PEG), the maximum percentage of dye degradation and turbidity removal were achieved.

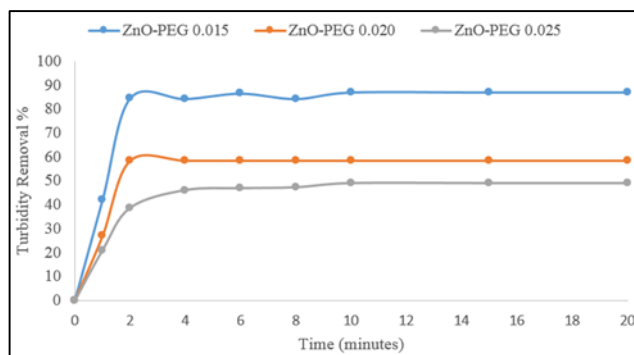


Fig. 9: Percentage of turbidity removal versus time for photocatalysis process under different photocatalysts, ZnO-PEG (0.015, 0.020 and 0.025 g/L).

Fig. 10 demonstrate the pattern of dissolved oxygen against time for photocatalysis process under different photocatalysts, ZnO-PEG (0.015 g/L of PEG), ZnO-PEG

(0.020 g/L of PEG) and ZnO-PEG (0.025 g/L of PEG) in 20 minutes. Based on the Fig. 10, all the reading of dissolved oxygen is in the range 0.32 to 5.42 ppm. The DO readings from all treated dye wastewater sample in presence of ZnO-PEG (0.020 and 0.025 g/L of PEG) almost have the similar value which is in the range 3.27 to 5.42 ppm. However, ZnO-PEG (0.015 g/L of PEG) have the lowest value of dissolved oxygen which is in the range of 0.32 to 1 ppm due to the higher consumption of oxygen molecules in photocatalysis process to degrade dye particles.

Based on the National Water Quality System (NWQS), the acceptable range of DO value is in between 0.02 to 20 mg/L in order to preserve the environment water quality and to ensure the wastewater is not harmful to aquatic life [18]. Hence, it can be concluded that the treated dye wastewater in this study can be discharged into environment since the DO reading is the range 0.32 to 5.42 ppm.

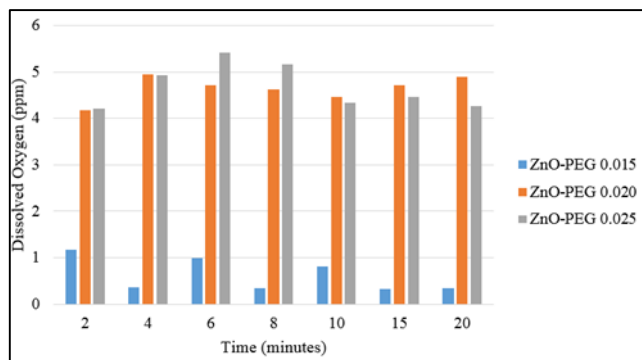


Fig. 10: Effect of different photocatalysts ZnO-PEG (0.015, 0.020 and 0.025 g/L) on dissolved oxygen versus time under optimum pH (pH7)

4. CONCLUSION

The optimum loading of PEG in precipitation of ZnO nanoparticles had successfully assess by comparing the effectiveness of all type of ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG) in photocatalytic degradation of dye wastewater. Hence, 0.015 g/L PEG loading in ZnO nanoparticles has the most optimum of PEG loading due to the highest degradation percentage of dye removal which is 95.48%. The characterization of chemical and physical properties of ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG) were successfully characterized by using Fourier Transform Infrared Spectrometer (FTIR). Accordingly, FTIR results show that the samples ZnO-PEG (0.015, 0.020 and 0.025 g/L of PEG) contain ZnO stretching and deformation vibration. However, OH compound was detected due to the adsorption of moisture from surrounding during synthesizing the photocatalyst. The optimum condition of industrial dye wastewater treatment via photocatalysis process in presence of ZnO-PEG nanoparticles was successfully determined by conducting the photocatalysis process under different type

of pH (acidic, neutral and alkaline condition). Thus, pH 7 (neutral condition) was observed to be the optimum condition since it revealed the highest dye degradation percentage which is 95.48%. Hence, it can be concluded that with the presence of ZnO-PEG nanoparticles as a photocatalyst in photocatalytic degradation, the existence of dye particles in wastewater can be successfully remove.

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