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Comparison of α-Fe_2O_3 Nanocatalyst Synthesized with Various Surfactants and its Application in Phenol Degradation

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Abstract

Breaking down organic pollutants like phenol is possible with the use of a sophisticated oxidation process called photo-Fenton. The α-Fe_2O_3 catalyst was chosen because it has a small bandgap value so it is effective in degrading organic compounds through a clean photocatalytic reaction mechanism. This study aims to synthesize α-Fe_2O_3 nanocatalysts to be applied in the phenol degradation process through the photo-Fenton reaction mechanism. The catalysts were made using a variety of surfactants in surfactant-assisted coprecipitation. In a batch setup, phenol degradations were performed with an initial concentration of 50 mg/L and reaction times of 120 min where the optimum pH value was searched initially. The concentration of phenol was analysed using a UV-Vis spectrophotometer. The characterization results showed that particle agglomeration of α-Fe_2O_3 catalysts was reduced by adding surfactant resulting in moreordered nanoparticles. The XRD pattern also showed that the α-Fe_2O_3 nanocatalyst with hexagonal structure was successfully formed where the average crystal size based on the Scherrer equation was 38 nm. The BET test results showed that the α-Fe_2O_3-SDS nanocatalyst had the largest surface area of 17 m^2/g compared to the α-Fe_2O_3-CTAB and α-Fe_2O_3-PEG nanocatalysts. The results of the catalyst activity test showed that the α-Fe_2O_3 nanocatalyst could degrade phenol compounds with the largest degradation percentage of 47% obtained with the photo-Fenton process using a catalyst synthesized with SDS surfactant. The results of this study also prove that the photo-Fenton process can degrade phenol better than the Fenton process using the α-Fe_2O_3 nanocatalyst.

Keywords

nanocatalyst, α-Fe_2O_3, surfactant, coprecipitation, photo-Fenton

1 Introduction

One of the hazardous organic wastes from industrial waste is phenol which has a high risk to human health [1]. Therefore, a speedy, effective, and ecologically safe method of breaking down the phenol component is required. An intriguing substitute for the breakdown of a variety of resistant pollutants such as phenol at low temperature and pressure conditions is the use of advanced oxidation techniques, which are based on the production of extremely reactive oxidative radicals [2].

The Fenton process is among the advanced oxidation techniques which use an oxidiser in the form of hydrogen peroxide (H_2O_2) and an iron ion catalyst (Fe) known as the Fenton reagent and produces hydroxyl radicals (•OH) which can strongly degrade organic compounds [3]. According to the concept of green chemistry, using H_2O_2 as an oxidiser and H_2O as a solvent for oxidation reactions is preferred because it is harmless and does not produce by-products other than oxygen and H_2O.

Fe^{2+} or Fe^{3+} species are usually chosen as catalysts in the conventional homogeneous Fenton process because they can catalyze H_2O_2 to produce a strong oxidizer •OH [4]. Despite its high efficiency, the conventional homogeneous Fenton system has several disadvantages, including the excess Fe(II) (or Fe(III)) cannot be recovered. In addition, many homogeneous Fenton-like catalysts that have been reported consist of Fe ion complexes with intricate organic ligands, which can be costly and may break down gradually during degradation [5].

Many efforts have been made to develop heterogeneous Fenton processes using highly active and stable solid catalysts to address the drawbacks of the homogeneous Fenton process. Considerable interest has been shown in

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hematite (α-Fe_2O_3) as a heterogeneous catalyst because it is abundant and low-cost, has a large surface area, a certain morphology, and a well-defined topology [6]. In addition, α-Fe_2O_3 material also has characteristics as a semiconductor that can be applied in the photocatalysis process [7]. α-Fe_2O_3 material as a semiconductor has a small band gap that is efficient in the photocatalysis process. Based on these properties, α-Fe_2O_3 material can be applied in a combination of the Fenton process and photocatalysis known as the photo-Fenton process [3]. This work examined the manufacture and use of α-Fe_2O_3 nanocatalyst in the degradation of phenol. Nanoparticle materials are very interesting materials because materials at the nanoscale are superior to bulk materials in a variety of chemical and physical aspects [8]. Nanomaterials have active sites with high density and larger surface area so that they help promote substrate breakdown on the catalyst surface during the catalytic advanced oxidation process [9]. Different techniques have been identified to synthesize α-Fe_2O_3 nanoparticles, such as coprecipitation, microemulsion, hydrothermal phase, inductive heating, and sol-gel procedures [10]. Among these methods, the coprecipitation method is attractive because it is simple, low-cost, and an industrially feasible method [11]. The wide range of particle sizes that this synthesis process produces, ranging from 50 to 300 nm, is a major disadvantage, so, for technical applications, nanoparticle dispersion without aggregation is crucial according to Kushwaha and Chauhan [10]. Therefore, the addition of surfactants during the synthesis of nanomaterials is very important to prevent the aggregation of nanoparticles so that nanoparticles with high surface area and increased catalytic activity are obtained [12].

Cationic, anionic, and nonionic surfactants can all aid in the synthesis of nanomaterials with a variety of intriguing morphologies [10]. Therefore, in this study, three types of surfactants will be used for the synthesis of α-Fe_2O_3 nanoparticles via coprecipitation and their effects on the characteristics of the resulting α-Fe_2O_3 and its performance in the phenol degradation process are studied.

In this research, the cationic surfactant cetyltrimethylammonium bromide (CTAB), the nonionic surfactant polyethylene glycol 4000 (PEG), and the anionic surfactant sodium dodecyl sulfate (SDS) are proposed as capping agents that will reduce the surface tension of the precursor solution used in the synthesis of α-Fe_2O_3. The crystallinity characteristics of the catalysts made from these three types of surfactants will be determined using X-ray diffraction (XRD), the morphology and elemental composition will be determined using scanning electron microscopy-energy dispersive X-ray (SEM-EDX), and the surface area will be determined using the Brunauer–Emmett–Teller (BET) model. The effectiveness of α-Fe_2O_3 nanocatalysts with varous surfactant in degrading phenol through the photo-Fenton process will be investigated in this study.

2 Materials and methods

2.1 Materials

Materials utilized in this investigation include those needed for catalyst synthesis, such as demineralized water, ferric chloride ($FeCl_3 \cdot 6H_2O$), ethanol, sodium hydroxide, CTAB, PEG, and SDS. The materials for phenol degradation include phenol and hydrogen peroxide.

2.2 Preparation of nanocatalyst with various surfactant

In this study, the surfactant-assisted coprecipitation method was carried out for the synthesis of α-Fe_2O_3 nanomaterials based on the procedure reported by Debnath et al. [13] which was modified. In a typical experiment, a surfactant was added to the $FeCl_3 \cdot 6H_2O$ precursor solution under continuous stirring to form a mixed precursor solution (solution A). NaOH was dissolved in demineralized water to create the precipitant solution. Furthermore, the precipitant solution was added dropwise into solution A accompanied by stirring and the pH was maintained at mildly alkaline conditions (pH: 7.5–8.5) during the synthesis. The resulting mixture was stirred continuously for 1 h at room temperature to produce a brown precipitate. The formed precipitate was filtrated and washed several times with demineralized water until the pH approached 7 and then dried in an oven at 85 °C for 2 h. After that, the solid was calcined at 500 °C for 4 h and allowed to cool slowly to room temperature to obtain α-Fe_2O_3. Lastly, the iron oxide product (α-Fe_2O_3) formed was ground and sieved to produce a homogeneous powder.

2.3 Characterization of nanocatalyst

To ascertain whether the produced α-Fe_2O_3 crystallinity and crystal structure of the nanocatalyst were appropriate based on existing literature, an XRD examination was performed using D8 Advance (Bruker, Germany) with wavelength applied 1.54060 Å. The catalyst's surface area was measured by BET analysis using NOVA 3200e (Quantachrome, USA), and its elemental composition and surface morphology were examined by SEM-EDS analysis using SEM SU3500 (Hitachi High-Technologies, Japan). The large surface area of the catalyst, its morphology that demonstrates the properties of nanoparticles with the proper elemental composition, and the suitability of the synthesized catalyst's crystallinity and crystal structure with the literature are the expected achievement indicators in this study.

2.4 Phenol degradation using α-Fe_2O_3 nanocatalyst

The performance of α-Fe_2O_3 nanocatalysts was examined in the phenol degradation process. The phenol degradation process was carried out through the photo-Fenton process and compared with the Fenton process.

The effectiveness of the degradation process was followed by the concentration of degraded phenol expressed in % through Eq. (1):

$$\%\text{Degradation} = \frac{C_i - C_f}{C_i} \times 100, \tag{1}$$

where C_f is the phenol concentration at completion and C_i is the phenol concentration at the start of photo-Fenton degradation for the duration of 2 h.

The concentration of phenol was determined using a UV-Visible Spectrophotometer (Shimadzu UV-1800, Japan) by interpolating the absorbance data on a linear regression curve. The regression curve was obtained from the measurement results of phenol solutions at the maximum wavelength of 510 nm where its concentrations were varied between 0–10 mg/L.

3 Results and discussion

In this study, α-Fe_2O_3 catalyst can be synthesized at room temperature by coprecipitation method. Ethanol was added into NaOH precipitant solution to help the precipitation process of catalyst material. The addition of alcohol also can reduce the particle size as reported in the study of Reddy et al. [14]. Several surfactants, including cationic CTAB, nonionic PEG-6000, and anionic SDS, were employed to decrease the aggregate size and enhance hematite nanoparticle dispersibility.

3.1 Morphology of nanocatalyst particles

To ascertain the morphology of the produced catalysts, a SEM-EDS examination was performed. While elemental composition is detected via EDS, SEM allows the imaging. The samples analyzed by SEM were Fe_2O_3-CTAB, Fe_2O_3-SDS, and Fe_2O_3-PEG catalysts. Fig. 1 displays the SEM pictures of the α-Fe_2O_3 catalysts obtained from our experiments.

The results of the SEM study showed the uneven agglomeration morphology and relatively uniform particle size in all catalysts with surfactant additions. According to Liu et al. [15] this phenomenon was likely caused by an imbalance between the rate of Fe precipitation and the surfactant-assisted grain growth rate. Fig. 1 shows that particle agglomeration occurs in the α-Fe_2O_3 catalyst synthesized without the addition of surfactants resulting in larger particle size. Whereas particles of α-Fe_2O_3 with the addition of surfactants resemble spheres with only a few agglomerated particles when compared to α-Fe_2O_3 catalyst produced without surfactants. This phenomenon can be caused by the stabilization and steric hindrance properties of the surfactant, thus preventing particle agglomeration [16]. The addition of surfactants reduces the surface energy of nanoparticles through adsorption at the edge of the crystal core, thus preventing them from sticking together [17]. Furthermore, EDS analysis was carried out to determine the elemental composition in the synthesized catalysts. As shown in Fig. 2 the main components in the catalyst are elements of iron and oxygen.

3.2 XRD analysis of nanocatalyst

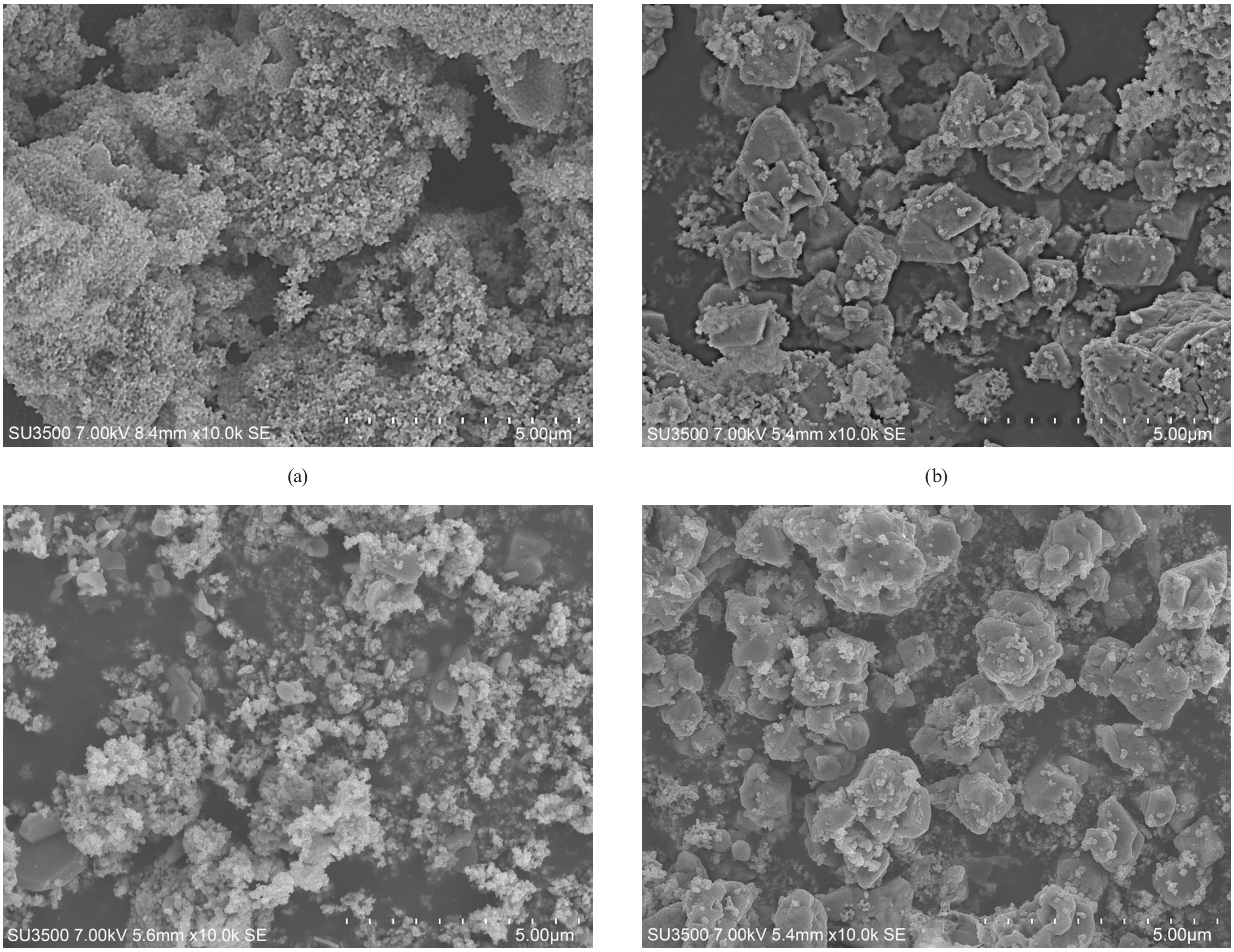
The characterization of the structure or crystal phase of Fe_2O_3 powder was carried out using XRD pattern analysis processed using software namely "Match!", a computer program by Crystal Impact, Germany [18]. The XRD patterns of the three α-Fe_2O_3 catalysts with variations in surfactants are shown in Fig. 3.

Based on Fig. 3, there are sharp narrow peaks in the XRD pattern indicating that all α-Fe_2O_3 products are highly crystalline. The main peaks that appear prove that the sample belongs to the alpha (α) phase of iron oxide or hematite phase, which corresponds to the reflection of the Miller planes (102), (104), (110), (113), (204), (116), (108), (214) and (300). The α-Fe_2O_3 catalyst sample has a crystal structure that is included in the trigonal (hexagonal) crystal system with a rhombohedral lattice system. The Scherrer equation is utilized to determine the crystallite size (D) based on the X-ray diffraction data of the hematite structure [10]:

$$D = \frac{0.94\lambda}{\beta \cos\theta}, \tag{2}$$

where λ is the X-ray wavelength, 0.94 is the Scherrer constant, β is the full width at half maximum (FWHM) in radian, and θ is the diffraction angle. Table 1 displays the crystallite size of the synthesized catalyst derived from Eq. (2).

The XRD pattern also shows that in addition to the main peak of the hematite structure, other peaks related to impurities were found, namely NaCl (halite) and Na_2SO_4 (thenardite). NaCl appeared in Fe_2O_3-CTAB and Fe_2O_3-PEG catalysts which could originate from the reaction between



(a) (b)

(c) (d)

Fig. 1 SEM images (magnification ×10000) of α-Fe_2O_3 nanocatalyst with the addition of surfactants: (a) without surfactant; (b) CTAB; (c) SDS; (d) PEG

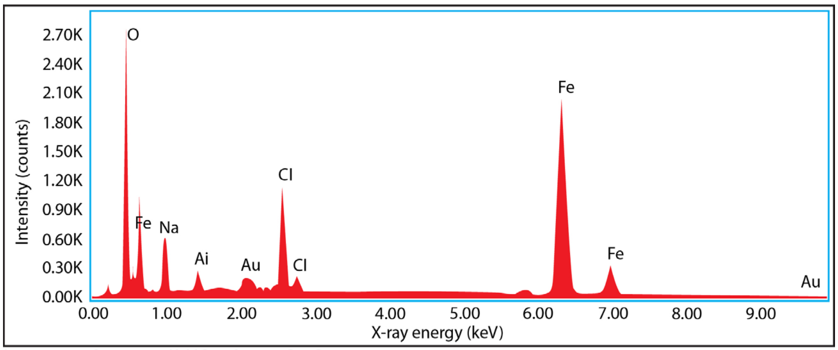


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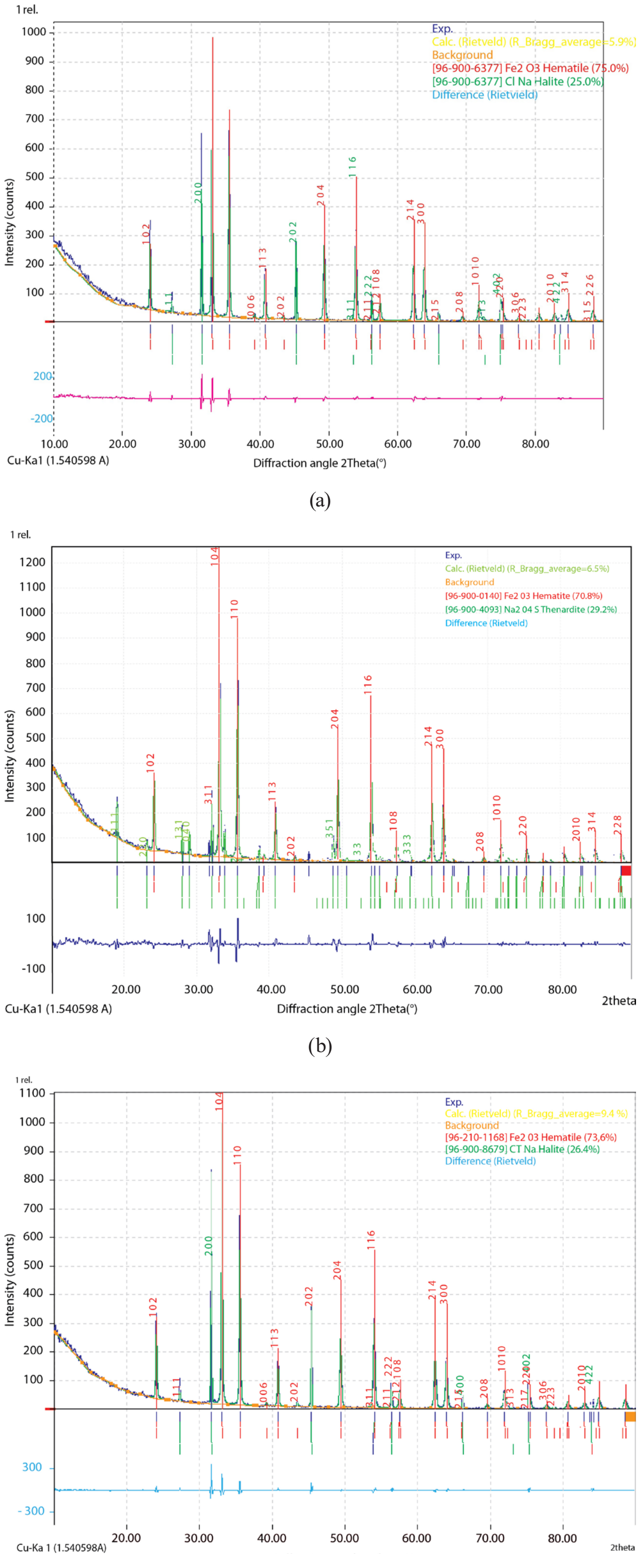
Fig. 2 EDS spectrum of α-Fe_2O_3 nanocatalyst with CTAB added surfactant

the Na^+ ion of the NaOH precipitant and the Cl^- ion of the $FeCl_3$ precursor used. Meanwhile, Na_2SO_4 appeared in the Fe_2O_3-SDS catalyst which could originate from the process where Na^+ and SO_4^{2-} ions were involved. This aligns with the study conducted by Géhin et al. [19] where Na_2SO_4 was formed when the remaining water evaporated after filtration.

3.3 Surface area of the nanocatalyst

Based on the measurement of nitrogen gas adsorption isotherms, the BET model was used to quantify the specific surface area of the catalyst. Based on the adsorption isotherm obtained from the measurement, the specific surface area of the material was determined. The surface areas of the synthesized catalysts are shown in Table 2.



(a)

(b)

(c)

Fig. 3 XRD pattern of the α-Fe_2O_3 catalysts modified with (a) CTAB; (b) SDS; (c) PEG

Table 1 Comparison of crystallite size of the synthesized α-Fe_2O_3 nanocatalyst with various surfactant

Catalysts	Average crystallite size (nm)
Fe_2O_3-CTAB	35
Fe_2O_3-SDS	38
Fe_2O_3-PEG	42

Table 2 Comparison of surface area of the synthesized α-Fe_2O_3 nanocatalyst with various surfactant

Catalysts	Surface area (m^2/g)
Fe_2O_3-CTAB	4
Fe_2O_3-SDS	17
Fe_2O_3-PEG	2

Based on Table 2, it was found that the α-Fe_2O_3 catalyst with the addition of SDS surfactant has the greatest surface area of 17 m^2/g. The observed differences in surface area related to the surfactant's influence during the synthesis and not due to residual carbon because the surfactants were effectively removed during the calcination process as indicated by the EDS result. SDS is an anionic surfactant that tends to form smaller, more compact micelles due to the hydrophilic sulfonate group and the long hydrophobic tail [20]. This allows for a more controlled formation of nanostructures, leading to a smaller particle size and a higher surface area. In contrast, CTAB (cationic surfactant) and PEG (nonionic) tend to form larger aggregates. According to Kushwaha and Chauhan [10], this can lead to less control over the particle size and structure, which may result in a lower surface area compared to SDS. Catalysts with smaller nanoparticle sizes have larger surface areas [21]. Catalyst activity may be increased by the catalyst's surface area, which is advantageous for the contact with the reactants [15].

3.4 Optimization of pH on phenol degradation

The degradation of phenol is carried out using the photo-Fenton process, namely by using a combination of hydrogen peroxide and UV light. Hydrogen peroxide is a strong oxidizer that can oxidize a mixture of salts and non-salts (like phenol) in solution, while UV light can trigger the breaking of bonds in organic compounds [3]. This combination method can produce a more effective and faster process for degrading phenol. In this study, the effectiveness of degradation is followed by the %Degradation. The phenol concentration was measured by a UV-VIS spectrophotometer. One of the factors that can have an impact on the ability of the Fenton process to degrade organic molecules is pH. Therefore, in this study, the degradation process was carried out using the α-Fe_2O_3 catalyst with pH variations of 3, 6 and 8 which aims to obtain the optimum pH in the phenol degradation process. The results of the phenol degradation test

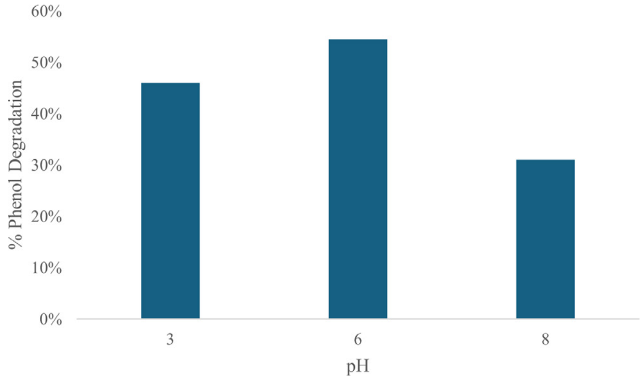


Fig. 4 Effect of pH on the performance of α-Fe_2O_3 nanocatalyst in phenol degradation

with pH variations are shown in Fig. 4.

Based on Fig. 4, the degradation of phenol with the photo-Fenton process using the nano α-Fe_2O_3 catalyst is optimal at pH 6, i.e., slightly acidic conditions with a %Degradation of 54%. This result is different from the research of Permata et al. [7] who used ZnO as a photocatalyst where phenol was more degraded in basic conditions with the largest degradation percentage of 41% at pH 8. This can occur due to differences in the catalysts used. The α-Fe_2O_3 catalyst contains Fe^{3+} ions which can react with OH^- in basic conditions, thereby reducing its effectiveness in forming hydroxyl radicals in the photo-Fenton reaction mechanism. In addition, based on Wardhani et al. [22], Fe_2O_3 as a photocatalyst has a charge that matches the pH of its environment. The surface of the Fe_2O_3 photocatalyst will be positively charged at a pH below its point of zero charge (PZC), which is pH 8. The phenol compound which has an aromatic ring, so it is rich in negatively charged electrons. Therefore, phenol is more easily adsorbed on the positively charged Fe_2O_3 surface. The results of this study also have a good agreement with the research of Setyaingtyas et al. [3] which also used the Fenton reagent, namely $FeSO_4$, where the photo-Fenton process is effective in slightly acidic conditions.

3.5 Comparison of nanocatalysts with various surfactant on phenol degradation

In this study, the phenol degradation test with the photo-Fenton process was carried out using α-Fe_2O_3 nano-catalysts synthesized with various types of surfactants. The operating condition of pH 6 was chosen based on the results of previous experiments showing that the photo-Fenton process was effective at that pH. The Fenton process without UV irradiation was also carried out in this study as a comparison. The percentage of phenol degradation of various α-Fe_2O_3 nano catalysts is shown in Fig. 5.

Based on Fig. 5, the photo-Fenton process with UV irradiation is proven to be more effective than the Fenton process because the •OH radicals formed in the photo-Fenton process are of higher concentration. This occurs because the photons produced by UV light can drive the photocatalysis reaction by the α-Fe_2O_3 semiconductor which produces •OH radicals. This is consistent with studies of Setyaningtyas et al. [3] which used the $FeSO_4$ catalyst where the photo-Fenton process was more effective in degrading phenol compared to the Fenton process because of the formation of more hydroxyl radicals. The results of this study also show that phenol photodegradation using the Fe_2O_3-SDS catalyst is most effective compared to the α-Fe_2O_3 catalyst with CTAB and PEG surfactants. This can be caused by the surface area of the Fe_2O_3-SDS catalyst which is the largest compared to other catalysts so that the phenol adsorption capacity of the reactant molecules is higher so that the reaction rate is also higher. The high activity of the Fe_2O_3-SDS catalyst can also be caused by the presence of the thenardite phase which may catalyze phenol degradation.

We conducted a comparative analysis of the catalytic performance of our α-Fe_2O_3 catalysts against other catalysts documented in the literature concerning the degradation of phenol (Table 3) [4, 23–27]. Our α-Fe_2O_3 clearly exhibits sufficient photo-Fenton activity under relatively mild conditions regarding temperature, pH, lamp intensity, and H_2O_2 concentration, highlighting its potential efficacy in light of contemporary research findings.

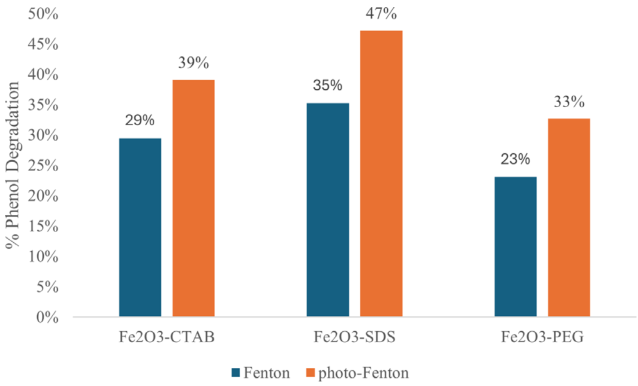


Fig. 5 Comparison of phenol degradation percentage of the synthesized α-Fe_2O_3 nanocatalyst with various surfactant

Table 3 Catalytic results of degradation of phenol over different catalysts

Entry	Catalyst	Reaction conditions	Phenol degradation	Ref.
1	$FeSO_4·7H_2O$	Room Temperature (RT), 2 h, pH 6, UV lamp, 1000 mg/L catalyst, H_2O_2/catalyst = 0.05	64.9%	[4]
2	Fe_2O_3 QDs/g-C_3N_4	RT, 120 min, pH 5, 500 W halogen lamp, 500 mg/L catalyst, 30 mg/L phenol, 15 mmol/L H_2O_2 (30 wt%)	56.7%	[23]
3	Fe(III)/Bi_2MoO_6	RT, 180 min, 400 W halogen lamp, 1000 mg/L catalyst, 10 mg/L phenol	42%	[24]
4	$CuFe_2O_4$	RT, 120 min, UV lamp, 300 mg/L catalyst, 200 mg/L phenol, 0.5 M H_2O_2 (30 wt%)	49%	[25]
5	ZnO	RT, 120 min, pH 5, sunlight, 4000 mg/L catalyst, 200 mg/L phenol, 2 mmol/L H_2O_2	45%	[26]
6	AC-ZnO	RT, 120 min, pH 5, sunlight, 4000 mg/L catalyst, 200 mg/L phenol, 2 mmol/L H_2O_2	68%	[26]
7	TiO_2	RT, 120 min, pH 4, 300 W xenon lamp, 1000 mg/L catalyst, 10 mg/L phenol, 22.9 mmol/L H_2O_2 (30 wt%)	42%	[27]
8	α-Fe_2O_3	RT, 120 min, pH 6, 10 W UV lamp, 1000 mg/L catalyst, 50 mg/L phenol, H_2O_2/catalyst = 0.05, 1.47 mmol/L H_2O_2	47%	This work

4 Conclusion

The present study concluded that the addition of surfactants for synthesizing α-Fe_2O_3 nanocatalysts through coprecipitation plays an important role in preventing particle agglomeration. The α-Fe_2O_3-SDS nanocatalyst has the largest surface area compared to the α-Fe_2O_3-CTAB and α-Fe_2O_3-PEG nanocatalysts. The results of the experiment showed that Fe_2O_3-SDS nanocatalyst has the highest performance compared to Fe_2O_3-CTAB and Fe_2O_3-PEG nanocatalysts. The highest percentage of phenol degradation of 47% was obtained with the photo-Fenton process. This result was obtained under benign conditions with a 50 mg/L starting phenol concentration and a 120-min reaction time. This study also concluded that the photo-Fenton process is more effective than the Fenton process in degrading phenol compounds because UV irradiation can form more hydroxyl radicals. The results of this study provide an overview of the potential of α-Fe_2O_3 nanocatalysts made with various surfactants as effective photo-Fenton catalyst to degrade organic materials.

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