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# Simbang Darah (*Iresine herbstii*) extract mediated hydrothermal method in the synthesis of zinc ferrite spinel nanoparticles used for photocatalysis and antibacterial applications

Rahmayeni [a,*], Rike Febrialita [a], Yeni Stiadi [a], Yulia Eka Putri [a], Nofrijon Sofyan [b], Zulhadjri [a]

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## ARTICLE INFO

## ABSTRACT

This paper reports the use of Simbang Darah (*Iresine herbstii*) leaf extract to synthesize $ZnFe_2O_4$ spinel ferrite nanoparticles via hydrothermal method. Biochemical compounds in Simbang Darah leaf extract act as a capping agent and a stabilizer to form nanostructures. XRD patterns and FTIR spectrums confirm the formation of the cubic spinel crystal structure of $ZnFe_2O_4$ nanoparticles. SEM and TEM characterization revealed that $ZnFe_2O_4$ spinel ferrite morphology has a uniform spherical shape in nanoscale. EDX results showed that the compositional mass ratios were relevant as expected from the synthesis. $ZnFe_2O_4$ nanoparticles have an absorption edge in the visible region with Eg values within the range 2.05–2.11 eV in the DRS UV–Vis analysis. The as-prepared samples exhibit paramagnetic behavior according to the magnetic hysteresis curve. $ZnFe_2O_4$ nanoparticles were proven effective in direct red 81 dye photodegradation (99.66%) after exposure to solar light irradiation for two hours. On top of that, $ZnFe_2O_4$ nanoparticle exhibited antibacterial activity against *S. aureus* and *E. coli*.

## 1. Introduction

Zinc ferrite ($ZnFe_2O_4$) is an important magnetic material that has attracted significant interest due to its unique optical, magnetic, and electrical properties. It has a normal spinel phase where $Zn^{2+}$ ions occupy the tetrahedral site, and $Fe^{3+}$ ions occupy the octahedral site. In nanoscale, the changes of $ZnFe_2O_4$ structure into mixed spinel can increase its magnetic properties to become superparamagnetic [1,2]. Zinc ferrite has been widely used in many fields such as biomedicine [3], the production of battery anodes [4], gas sensors [5,6], planar drug delivery [7], besides acting as antibacterial agent [8], photocatalysts, and absorbent material [9,10]. It can be manufactured via various techniques such as co-precipitation route [11], one step and multistep hydrothermal method [12,13], solvothermal method [10], combustion route [14], solution combustion reaction [6], thermal method using reducing and capping agents [15], and soft chemical route [16]. Each method has its advantages and disadvantages such as simplicity, convenience, temperature, and hazardous chemicals usage.

Many researchers are currently focused on using natural substances derived from plants or microorganism to synthesize functional materials. This approach can replace hazardous chemicals and offer an environmentally friendly synthesis [17]. In addition, the utilization of several plant extracts as stabilizers in the biosynthesis of ferrite material with nanostructures has been proven to be environmentally friendly, easy to obtain, low-cost, and non-toxic [18].

In recent years, there have been many reports of the green synthesis of $ZnFe_2O_4$ compound by different methods. For instance, Kombaiah et al. [19] synthesized $ZnFe_2O_4$ using *Hibiscus rosa-sinensis* extract as a reducing and stabilizer agent via conventional and microwave combustion methods. Meanwhile, Patil et al. [14] used sugarcane juice as fuel in $ZnFe_2O_4$ synthesis via combustion method. Additionally, Matinise et al. [20] utilized *Moringa oleifera* extract as chelating and reducing agents in the synthesis of $ZnFe_2O_4$. Furthermore, *Limonia aciddissima* juice was used as fuel in the synthesis of $ZnFe_2O_4$ using microwave by Madhukara Naik et al. [21]. Moreover, Sriramulu et al. [17] used *Aegle marmelos* leaves extract in the synthesis of $ZnFe_2O_4$. The biosynthesis of zinc ferrite produced small particles with a homogeneous, smooth, and stable structure [18].

Simbang Darah (*Iresine herbstii*) is a family of *Amaranthaceae* with various leaf colors ranging from purple or dark purple to green (Fig. 1). Its leaves have been used as a traditional remedy for eczema and acne scars for humans. Besides, Simbang Darah leaves are boiled in water and

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**Fig. 1.** Simbang Darah (*Iresine herbstii*) leaves.

used as an antipyretic and tonic to treat anemia. In the biomedical field, Simbang Darah has been used as an anti-cancer and antimicrobial agent [22,23]. Simbang Darah contains several compounds including 3′, 4′, 7-trihydroxy-6-methoxy flavone, N-feruloyltyramine, glycinebetaine and trigonelline, betacyanin acylate-amaranthine, isoamaranthine, iresin I, isoiresin I (= iresin II), iresinin III and iresinin IV [24]. In the latest research, this plant was found to contain isoflavone compounds 2′, 2, 5-trimethoxy-6,7-methylenedioxysiisoflavone and tlatlancuayin isoflavones (2′, 5-dimethoxy-6,7-methylenedioxyisoflavone) [25]. Several advantages of using Simbang Darah leaves include easy to obtain, easy to extract, and economical. As of today, there have been no reports of $ZnFe_2O_4$ hydrothermal synthesis using Simbang Darah leaf extract.

In this study, a green synthesis approach was used to synthesize $ZnFe_2O_4$ magnetic material via the hydrothermal method using Simbang Darah extract as a capping and a stabilizer agent. The obtained $ZnFe_2O_4$ nanoparticles were characterized using several instruments to analyze the crystal structure and crystallite size, morphology, magnetic properties, optical properties, binding energy, and interatomic interactions in their spinel structures. The photocatalytic activity of $ZnFe_2O_4$ was evaluated on the degradation of direct red 81 dyes under natural sunlight. Direct red 81 with molecular formula $C_{29}H_{19}N_5Na_2O_8S_2$ is one of the synthetic dyes in the textile industry and has been known for its carcinogenic nature and toxicity towards animals and humans [26,27]. Furthermore, the antibacterial activity of $ZnFe_2O_4$ was also tested against pathogenic bacteria found in wastewater, i.e., *S. aureus* and *E. coli*.

## 2. Materials and methods

### 2.1. Materials

$Zn(NO_3)_2.4H_2O$ (Sigma-Aldrich) and $Fe(NO_3).9H_2O$ (Sigma-Aldrich) were used as precursors without further treatment. NaOH (Merck) was used as a mineralizer, with Nutrient agar, and distilled water. Direct red 81 dye was obtained from the local textile factory. Meanwhile, Simbang Darah (*Iresine herbstii*) leaves were obtained from Alahan Panjang, West Sumatra, Indonesia. The pathogenic bacteria were obtained from the Microbiology Laboratory, Faculty of Agriculture, Universitas Andalas. All solutions were prepared using deionized water.

### 2.2. Extract preparation

Simbang Darah leaves (Fig. 1.) were separated from the stems, washed with distilled water, and dried in a closed room (25–28 °C) for ten days. Then, the dried leaves were blended into fine powder form. A total of 5 g of Simbang Darah leaf powder was added to 100 mL of distilled water and heated for 2 h at 58 °C under agitation at 300 rpm. After that, the extract was filtered and stored in a refrigerator momentarily before synthesizing zinc ferrite nanoparticles.

### 2.3. Synthesis of $ZnFe_2O_4$ nanoparticles

The $ZnFe_2O_4$ nanoparticles were synthesized via the hydrothermal method [28] using Simbang Darah leaf extract as a capping agent. A total of 1.49 g $Zn(NO_3)_2.4H_2O$ and 4.04 g $Fe(NO_3)_3.9H_2O$ were mixed in 50 mL Simbang Darah extract solution. The concentration of Simbang Darah solution was varied by adding 1, 3, and 5 mL of concentrated extract in 50 mL of solution. The mixture was stirred at 500 rpm for 1 h before adding 4 M sodium hydroxide (NaOH) to the mixture until pH of 12 was achieved. The stirring was continued for another 2 h. Later, the suspension was poured into an autoclave tube and heated at 180 °C for 3 h. The resulted precipitation was filtered, washed several times using distilled water until the pH 7 was achieved, and then heated in an oven at 110 °C for 4 h. The obtained $ZnFe_2O_4$ powders were labeled as $ZnE_1N$, $ZnE_3N$, and $ZnE_5N$, respectively. In order to investigate the effect of temperature on the structure formation of ferrite, the samples were calcined at 500 °C for 2 h. After that, the calcined ferrite powders were labeled as $ZnE_1NK$, $ZnE_3NK$, and $ZnE_5NK$ for 1, 3, and 5 mL extracts, respectively.

### 2.4. Material characterization

The phase composition and crystalline structure of zinc ferrite nanocrystals were studied using X-ray diffraction (PANalytical X′pert Pro Multipurpose Diffractometer) with Cu-Kα radiation ($\lambda = 1.5406$ Å) at 2θ ranging from 10° to 90°. The structural parameters were refined using the Le Bail method available in RIETICA software [29]. Then, the average crystallite size was calculated using the standard Scherer formula [18]. Surface morphology and chemical composition of ferrite were examined using a scanning electron microscope (SEM, JEOL JSM-6360LA) embedded with energy-dispersive X-ray spectroscopy (EDS). Detailed morphology of the samples was investigated using a transmission electron microscope (TEM, JEM-1400). High-resolution transmission electron microscopy (FEI, Technai G2, F30) was employed to probe the microstructure, particle size distribution, and high-resolution imaging of zinc ferrite. The binding energy of zinc ferrite was determined by X-ray photoelectron spectroscopy (XPS ULVAC-PHI Quantera SXM). The UV–Vis spectrophotometer analyzed the photoabsorption measurement of ferrites (Shimadzu UV–Vis 2450). The samples' magnetic properties were investigated using a vibrating sample magnetometer (VSM OXFORD 1.2H). FTIR spectra were recorded using a Nicolet IR200FT-IR spectrometer using KBr pellet technique. The adsorption-desorption isotherms were conducted using a surface area and pore size analyzer (BET, Quantachrome Nova 2000 E).

### 2.5. Determination of zinc ferrite photocatalytic activity

The photocatalytic activity of the synthesized $ZnFe_2O_4$ nanoparticles was determined under solar light irradiation for direct red 81 dye adapted from [28]. $ZnFe_2O_4$ catalyst in powder form was added to a petri dish containing 20 mL of 35 mg $L^{-1}$ direct red 81 solutions (pH = 6) and stored in a dark box for 30 min to investigate the adsorption process. The mixtures were then exposed for 2 h under natural solar light irradiation. Furthermore, the liquid was separated from the catalyst, and supernatant absorption was measured at a wavelength of 530 nm using a UV–visible spectrophotometer. The degradation percentage was calculated with $\% \ Deg. = (A_0 - A_t)/A_0 \times 100\%$ using the absorbance of direct red 81, where $A_0$ and $A_t$ are the initial absorbance and the absorbance at time $t$, respectively. Moreover, the same experiments were performed with the $ZnE_1N$, $ZnE_3N$, $ZnE_5N$, $ZnE_1NK$, $ZnE_3NK$, and $ZnE_5NK$ samples. Two samples with the best activity were further tested for several parameters related to photocatalytic activity, i. e., radiation duration (0.5, 1, 1.5, and 2 h) and dye concentration (25, 30, 35, 40, and 45 mg $L^{-1}$). The samples were retested in the same manner to investigate their durability and stability against repeated use. Prior to retesting, the used samples were rinsed with distilled water and
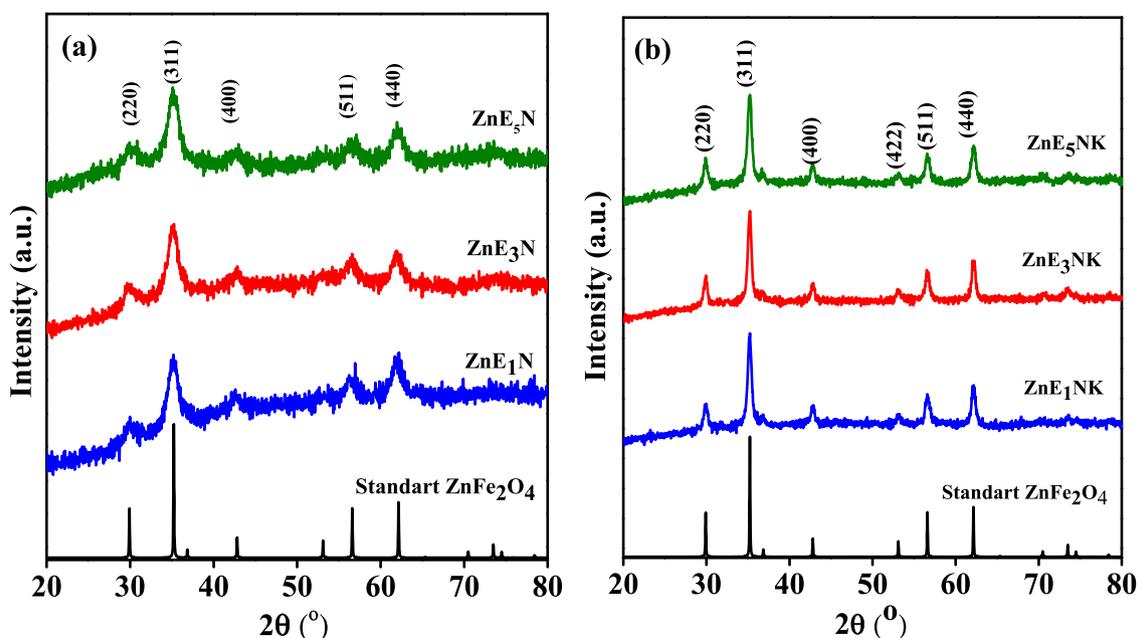
**Fig. 2.** XRD patterns of $ZnFe_2O_4$ before (a) and after calcination (b) at 500 °C prepared using Simbang Darah leaf extracts at various concentrations.

later heated in an oven at 110 °C for 2 h for activation.

## 2.6. Determination of zinc ferrite antibacterial activity

The antibacterial activities of the samples were screened using two pathogenic bacteria, *S. aureus* and *E. coli,* according to [30,31]. Nutrient agar (NA) was used as a growth media to observe nanoparticles' bactericidal activity in-vitro. Firstly, NA was autoclaved at 120 °C for 20 min and then cooled to 45 °C. Petri dishes for the antibacterial assay were also sterilized in an autoclave. Meanwhile, the bacteria were subcultured in 5 mL of sterile distilled water overnight. A total of 0.1 mL of the bacterial suspension was added to 20 mL of NA media and homogenized. Then, the NA media containing the bacteria was poured into a sterile petri dish and allowed to solidify. The $ZnFe_2O_4$ samples were inserted into the wells that had been prepared in the NA media. Amoxicillin was used as a positive control and water as a negative control in this experiment. The inhibition zones of the samples were measured after incubation at 37 °C for 24 h.

## 3. Results and discussion

### 3.1. XRD powder analysis

The XRD patterns of the $ZnFe_2O_4$ samples synthesized via the hydrothermal method in the presence of Simbang Darah leaf extract before and after calcination are shown in Fig. 2. XRD patterns of $ZnE_1N$, $ZnE_3N$, and $ZnE_5N$ are shown in Fig. 2a. All XRD peaks have been identified and confirmed to be single-phase $ZnFe_2O_4$ indexed to F*d3m* cubic crystal structure (ICSD-158837). The crystallite sizes calculated using the Debye–Scherer's formula are 7.2, 6.8, and 6.0 nm for $ZnE_1N$, $ZnE_3N$, and $ZnE_5N$, respectively. The decrease in crystallite size can also be observed visually through the broadening of the diffraction peaks.

Similarly, XRD analysis was also carried out on calcined $ZnE_1NK$, $ZnE_3NK$, and $ZnE_5NK$ samples, as shown in Fig. 2b. The XRD patterns were similar to that of Fig. 2a, but with sharper and well-resolved peaks. The crystallite sizes of the $ZnE_1NK$, $ZnE_3NK$, and $ZnE_5NK$ samples increase with the calcination process recording 15.8, 16.9, and 14.4 nm, respectively. These results indicated that the calcination accelerates the crystal's grain growth due to the coalescing of many crystallites of the same orientation, forming a large grain with a particular orientation
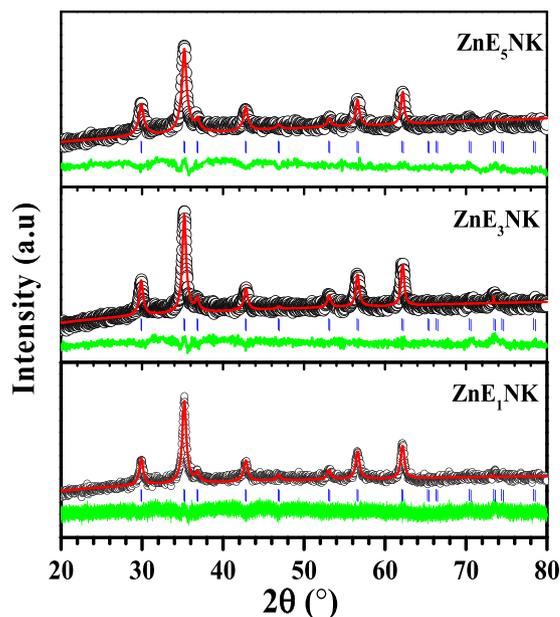


**Fig. 3.** Rietveld refinement patterns of $ZnFe_2O_4$ after calcination at 500 °C.

[32]. Both XRD pattern and crystallite size of the samples suggested that the Simbang Darah leaf extracts at varying concentrations influenced the formation of $ZnFe_2O_4$ ferrite material with spinel structure. The

**Table 1**
Refined structural parameters of single-phase $ZnFe_2O_4$ samples after calcination.

| Parameter | $ZnE_1NK$ | $ZnE_3NK$ | $ZnE_5NK$ |
|---|---|---|---|
| Space group | *Fd3ms* | *Fd3ms* | *Fd3ms* |
| Crystal class | Cubic | Cubic | Cubic |
| $a = b = c$ (Å) | 8.4499 | 8.4487 | 8.4458 |
| $V$(Å$^3$) | 603.33 | 603.07 | 602.45 |
| $Z$ | 8 | 8 | 8 |
| $R_p$(%) | 2.12 | 2.65 | 2.16 |
| $R_{wp}$(%) | 2.68 | 3.15 | 2.78 |
| $\chi^2$ | 1.41 | 3.91 | 1.59 |
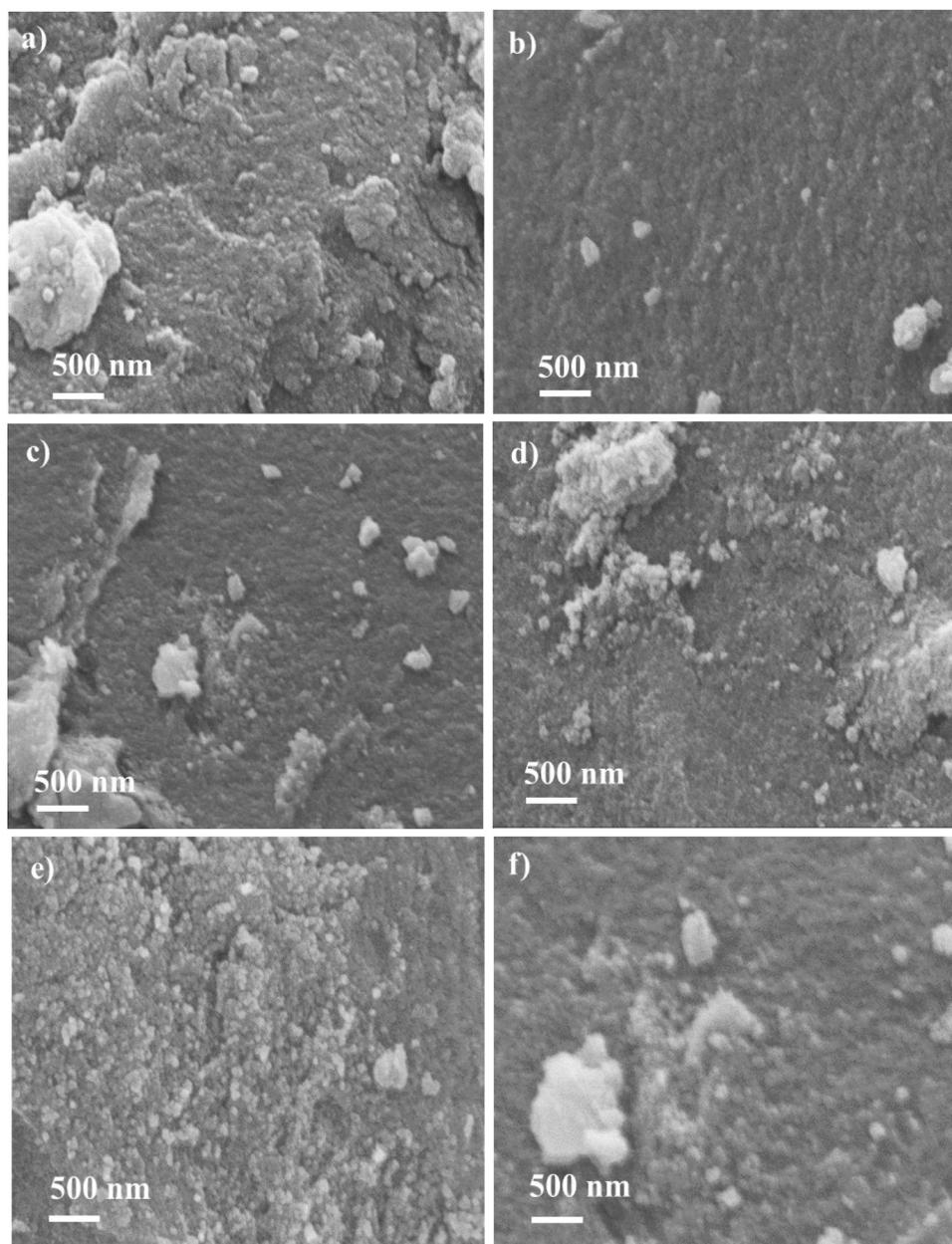
**Fig. 4.** Secondary electron images of (a) $ZnE_1N$, (b) $ZnE_3N$, (c) $ZnE_5N$, (d) $ZnE_1NK$, (e). $ZnE_3NK$, and (f) $ZnE_5NK$.

decreased in crystallite size with the increase of the extract concentration revealed that the secondary metabolites of the Simbang Darah could control the crystal growth.

The structural parameters of the nanoparticles were investigated using the Le Bail refinement method available in the RIETICA program. Initial parameters for the refinement were used from the cubic F$d3m$ $ZnFe_2O_4$ (ICSD-158837). Fig. 3 shows Le Bail profile to the XRD patterns of $ZnFe_2O_4$ samples after calcination. All XRD peaks were well-fitted with the Bragg reflections in the F$d3m$ space group, confirming that all samples were single-phase [33]. The refined structural parameters are given in Table 1. Besides, the lattices parameters and cell volume were relatively constant for all samples, demonstrating that the concentration of extract did not change the structure of $ZnFe_2O_4$.

### 3.2. SEM analysis

The morphology of synthesized $ZnFe_2O_4$ nanoparticles was analyzed using SEM instrument. Fig. 4 shows that all secondary electron images of

the samples have spherical shapes morphology with some clusters and aggregated nanoparticles. Fig. 4(a-c) demonstrates the morphology of $ZnE_1N$, $ZnE_3N$, and $ZnE_5N$ particles. The samples have more distinct homogeneity spherical morphologies with smaller particle size and less aggregation. Meanwhile, the $ZnE_1NK$, $ZnE_3NK$, and $ZnE_5NK$ particles also exhibited spherical morphologies with slightly larger particle size and agglomerations.

Generally, the agglomerating tendency is found in ferrite nanoparticles due to its high surface energy, magnetic nanomaterials [34]. The estimated particle size distribution ranged from 10 to 40 nm for $ZnE_1N$, $ZnE_3N$, and $ZnE_5N$, whereas for $ZnE_1NK$ $ZnE_3NK$, and $ZnE_5NK$ is in the range of 20–80 nm. These results are in agreement with [35] which have nanoparticle diameters ranging from 10 nm to 50 nm but smaller and homogeneous than that obtained by [34]. The homogeneous shape and the small size of ferrite particles are very beneficial for their photocatalytic activity applications. Smaller grain size has a larger surface area, leading to better interaction with the dye molecules in the liquid medium. Thus, it can be concluded that the morphology of the
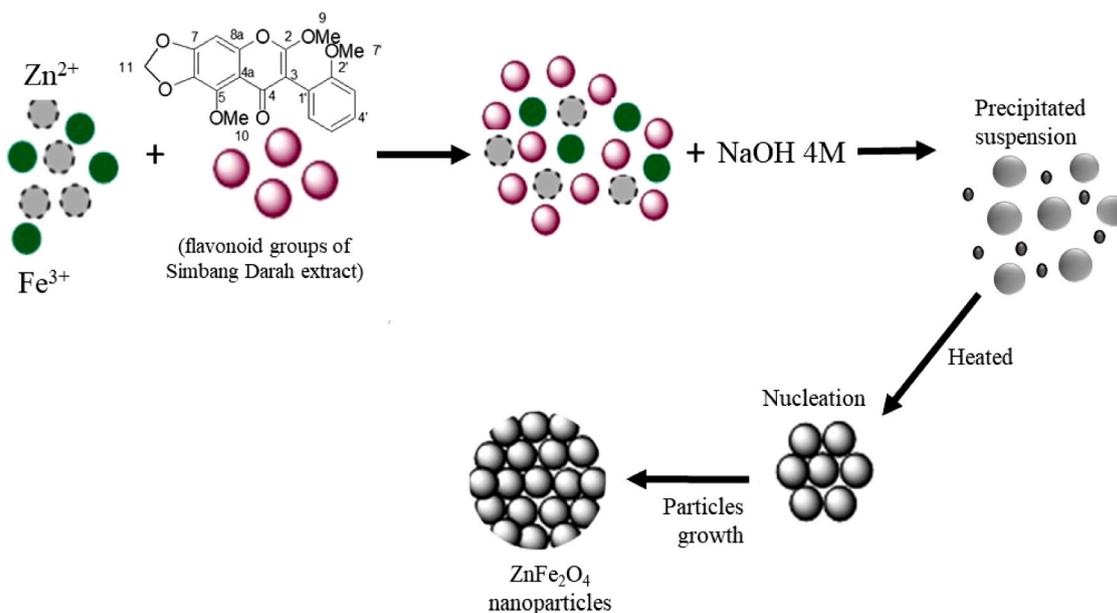
**Fig. 5.** Schematic diagram of the proposed formation mechanism of $ZnFe_2O_4$ nanoparticles in the presence of Simbang Darah extracts as a capping agent.



| Element | Wt% | At% |
|---------|-----|-----|
| OK | 27.06 | 57.89 |
| FeK | 43.87 | 26.89 |
| ZnK | 29.07 | 15.22 |
| Matrix | Correction | ZAF |

| Element | Wt% | At% |
|---------|-----|-----|
| OK | 27.30 | 57.97 |
| FeK | 47.99 | 29.19 |
| ZnK | 24.70 | 12.84 |
| Matrix | Correction | ZAF |
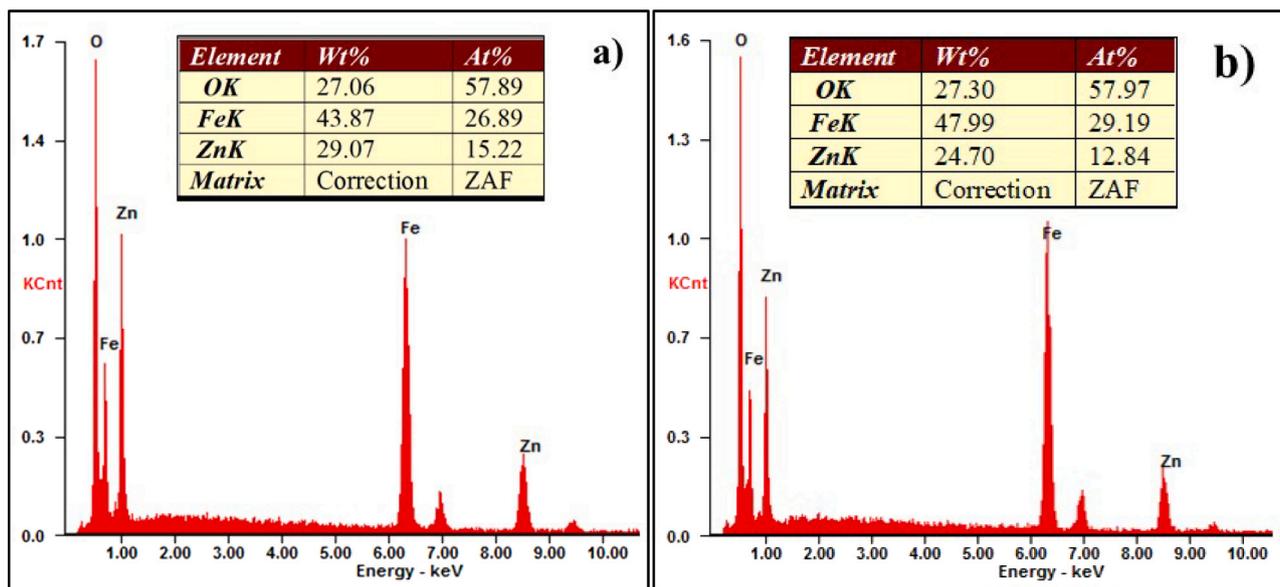
**Fig. 6.** EDX spectrum of $ZnE_1N$ (a) and $ZnE_1NK$ (b).

materials is affected by Simbang Darah leaf extract concentration and the calcination process. The proposed mechanism towards the formation of $ZnFe_2O_4$ nanoparticles in the presence of Simbang Darah extracts as the capping agent is presented in Fig. 5.

### 3.3. EDX analysis

The composition of the elements in the ferrites obtained from EDX is shown in Fig. 6. The EDX spectrum shows the presence of Zn, Fe, and O elements only in both samples. The weight percentages of Zn, Fe, and O elements in $ZnE_1N$ particles were 27.06%, 43.87%, and 29.07%, respectively, whereas $ZnE_1NK$ were 24.70%, 47.99%, and 27.3%, respectively. Fe and Zn's percentage in the samples suggested that the ratio of Fe: Zn atoms was close to 2:1, confirming the formation of $ZnFe_2O_4$ ferrite spinel structure. As shown in the spectra, the peaks are ascribed to the photocatalyst elements, and no other elements are found.

It indicated the high purity of $ZnFe_2O_4$ samples.

### 3.4. TEM analysis

Detailed morphology and structure of $ZnFe_2O_4$ were investigated using TEM (Fig. 7). Fig. 7(a-b) show the TEM photographs of $ZnE_1N$ that revealed particle-like shape with a uniform size distribution in the range of 6–8 nm. This finding correlates to the data obtained using XRD. Fig. 7b inset shows the particles size distribution and particle count histogram, resulting in more particles having a grain of 6 nm. The selected area electron diffraction (SAED) pattern (Fig. 7d) shows the spot type pattern as an indication of good crystallinity. The distinct lattice fringes were evident in the high-resolution TEM (HRTEM) image (Fig. 7c) suggesting that $ZnFe_2O_4$ is good crystalline in nature. The crystal planes were also clearly visible from the images, and the lattice plane fringes were used to calculate the d-spacing values. The d-spacing
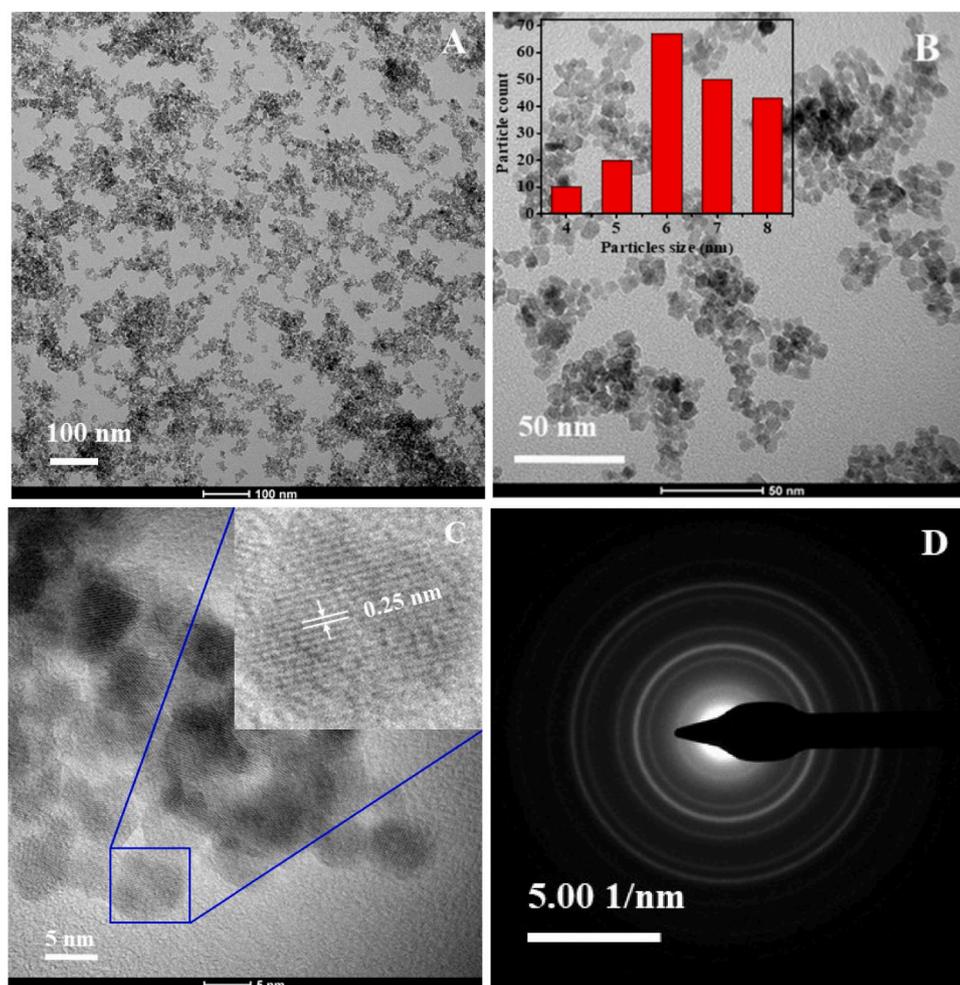
**Fig. 7.** TEM micrograph (a-b), HRTM micrograph (c), and SAED pattern of ZnE$_1$N (d). Insert in b) is the histogram of particles distribution.

of 0.25 nm corresponded to the (311) crystal plane of ZnFe$_2$O$_4$. This value aligned with the value of 2.5 Å, calculated using the Bragg's equation using the XRD result of a representative ZnFe$_2$O$_4$ [36]. This finding is further supported by the presence of intense rings that were well resolved in the SAED pattern.

### 3.5. XPS analysis

The X-ray photoelectron spectroscopy (XPS) measurement was used to analyze the chemical state of the elements in the ZnE$_1$N spinel ferrite. The results are displayed in Fig. 8(a and b). From the full spectrum of ZnFe$_2$O$_4$ in Fig. 8a, it can be observed that the sample consisted of Zn, Fe, O, and C binding energies with no other impurities. In Fig. 8b, the binding energies of Zn 2p$_{1/2}$ and Zn 2p$_{3/2}$ are 1045 eV and 1022 eV, respectively. Compared with the standard spectrum, these energies are suitable for Zn$^{2+}$ [37]. The spectrum of Fe shows two peaks located at 708, and 720 eV for 2p$_{3/2}$ and 2p$_{1/2}$ corresponding to Fe$^{3+}$ ion in the spinel ferrite. The presence peak of C 1s at 284.8 eV was introduced during the reaction process or exogenously substances during testing.

### 3.6. UV–Vis DRS analysis

The optical properties of ZnFe$_2$O$_4$ nanoparticles synthesized via the hydrothermal method and further calcined at 500 °C were investigated using UV–Visible reflectance spectroscopy. The samples' UV-DRS spectrum shows a strong absorption band at the visible light area (Fig. 8c). The band gap energy of the ZnFe$_2$O$_4$ nanoparticles was evaluated from the spectrum using the Kubelka-Munk model [38]. For the band gap

determination, a graph (a Taucplot) is plotted between $[F(R)h\nu]^2$ and energy (eV), and the intercept is the band gap energy as shown in Fig. 8d. The band gap energy for all samples is shown in Table 1. From Table 1, it can be seen that the band gap energies are in the range 2.05–2.11 eV, which are in good agreement with the previously reported values [14] using sugarcane juice extract and another report [39] using *Hibiscus rosa sinensis* extract. The band gaps of the synthesized ZnFe$_2$O$_4$ are in the range of 2.05–2.11 eV, which are expected to reduce the possibility of recombination of electrons from the conduction band to valence band and thus improve the catalytic activity. The band gap variation depends on various factors such as crystallinity, crystallite size, structural parameters, lattice strain, and the presence of defects or impurities [8,38]. In our case, the variation of band gap correlates with the crystallinity and crystallite size [21]. The observed blue shift in Fig. 9a is mainly attributed to the smaller crystallite size of the samples.

### 3.7. VSM analysis

The magnetization curves of ZnFe$_2$O$_4$ and the value of magnetic saturation (*Ms*), magnetic remanent (*Mr*) and coercive field (*Hc*) before and after calcination are shown in Fig. 9 and Table 2. The hysteresis curve shows that all ZnFe$_2$O$_4$ un-calcined samples have paramagnetic behavior.

The *Ms* value for the un-calcined samples increased (1.69, 1.74, and 2.08 emu/g) as the crystallite size decrease. Similarly, *Ms* increased when the sample was calcined at 500 °C because of the arrangement of atoms and dipoles in the crystal lattice as the temperature is raised. It was observed that all *Ms* values of the synthesized samples are smaller
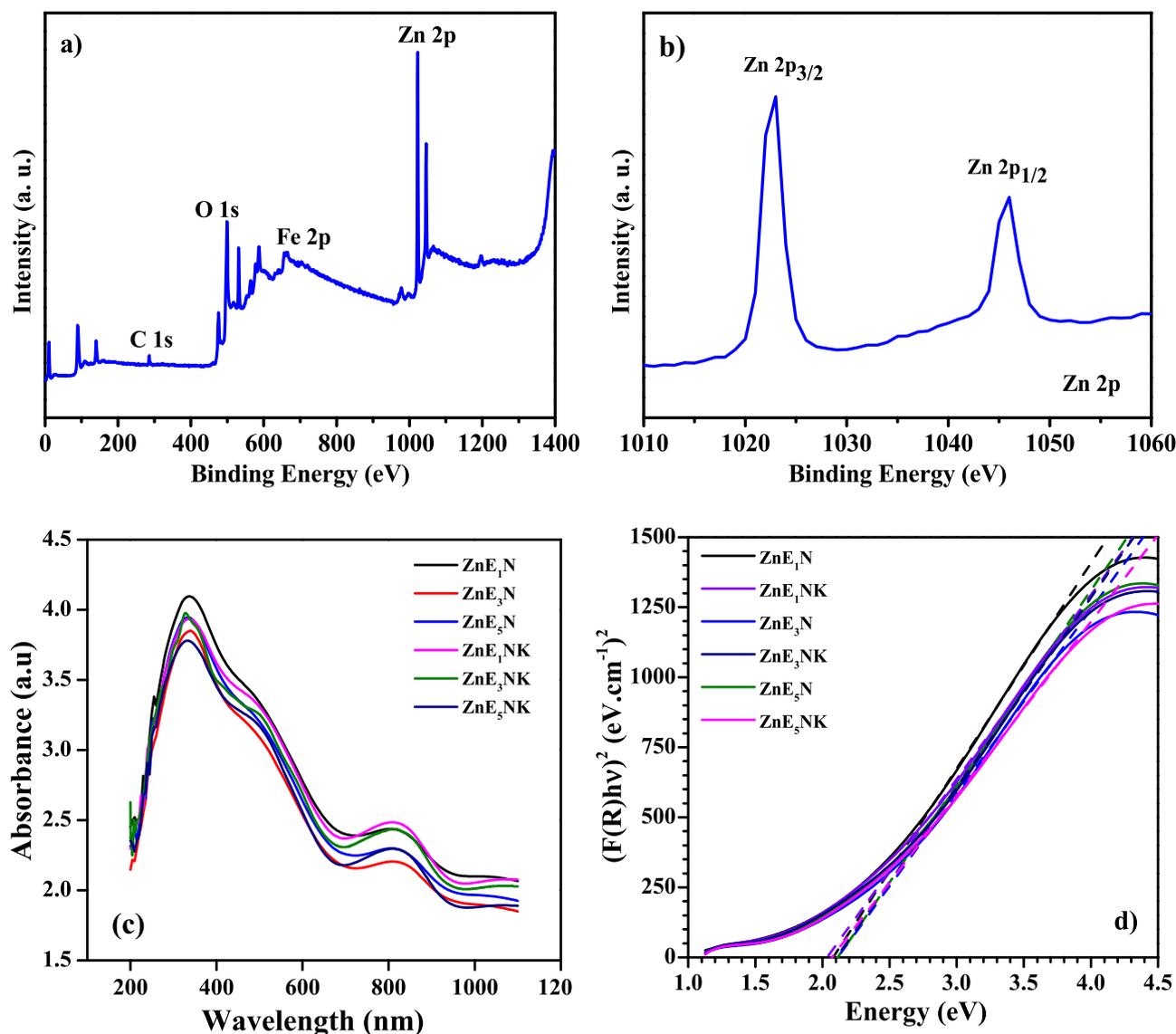
**Fig. 8.** XPS spectrum of ZnE$_1$N (a and b), DRS UV–Vis spectrum (c) and band gap energy (d) of samples.

than those obtained by [38]. Moreover, *Ms*, *Mr*, and *Hc* values obtained in this experiment indicated that the Simbang Darah leaf extract concentrations affect the crystallinity and magnetic properties of ZnFe$_2$O$_4$. Furthermore, the calcined samples of ZnE$_1$NK, ZnE$_3$NK, were paramagnetic, while ZnE$_5$NK exhibited superparamagnetic behavior. The calcination process of ZnFe$_2$O$_4$ will change ferrite behavior from paramagnetic to superparamagnetic. The presence of magnetic properties confirmed the efficacy of zinc ferrite as a photocatalyst because it can be separated from the liquid using an external magnetic field. Thus, these materials can be used for subsequent photocatalytic processes.

### 3.8. FTIR analysis

The structure of ZnFe2O4 ferrite nanoparticles before (a) and after calcination (b) was further studied by characterizing the samples using FTIR as shown in Fig. 10. Two prominent absorption bands of the spinel ferrite were observed in all spectra of the samples ranging from 300 to 600 cm$^{-1}$ indicating the spinel ferrite of ZnFe$_2$O$_4$. Apart from that, strong absorbance bands around 540–580 cm$^{-1}$ corresponded to Zn–O interaction at the tetrahedral site, whereas the bands around 300–450 cm$^{-1}$ were attributed to the stretching of Fe–O in the octahedral site [14].

The spectrums of ZnE$_1$N, ZnE$_3$N, ZnE$_5$N, ZnE$_1$NK, ZnE$_3$NK, and ZnE$_5$NK at 414, 424, 416, 430, 430, and 434 cm$^{-1}$ respectively, correspond to the intrinsic stretching vibration of Fe-O at the octahedral position, whereas the other absorption bands at 546, 558, 546, 548, 550, and 560 cm$^{-1}$ correspond to the stretching of Zn–O at the tetrahedral position. The adsorption band shifted with the decrease of particle size and with the increase of temperature. The difference in the absorption region was probably due to the varying Zn–O bond distance in the ZnFe$_2$O$_4$ spinel structure. Besides, the spectrum also showed that the intensity of the calcined sample (Fig. 10b) was sharper than the uncalcined sample (Fig. 10a). This observation is probably due to the calcination process that formed a regular ferrite spinel structure. From the obtained spectrum bands, it can be concluded that the resulting sample is spinel ferrite. The peaks around 1400–1600 cm$^{-1}$ for the uncalcined sample indicated the presence of bending and stretching of C=O, C–O, OH, and COO$^-$ derivatives of the secondary metabolites and other organic compounds contained in the Simbang Darah leaf extract [7].
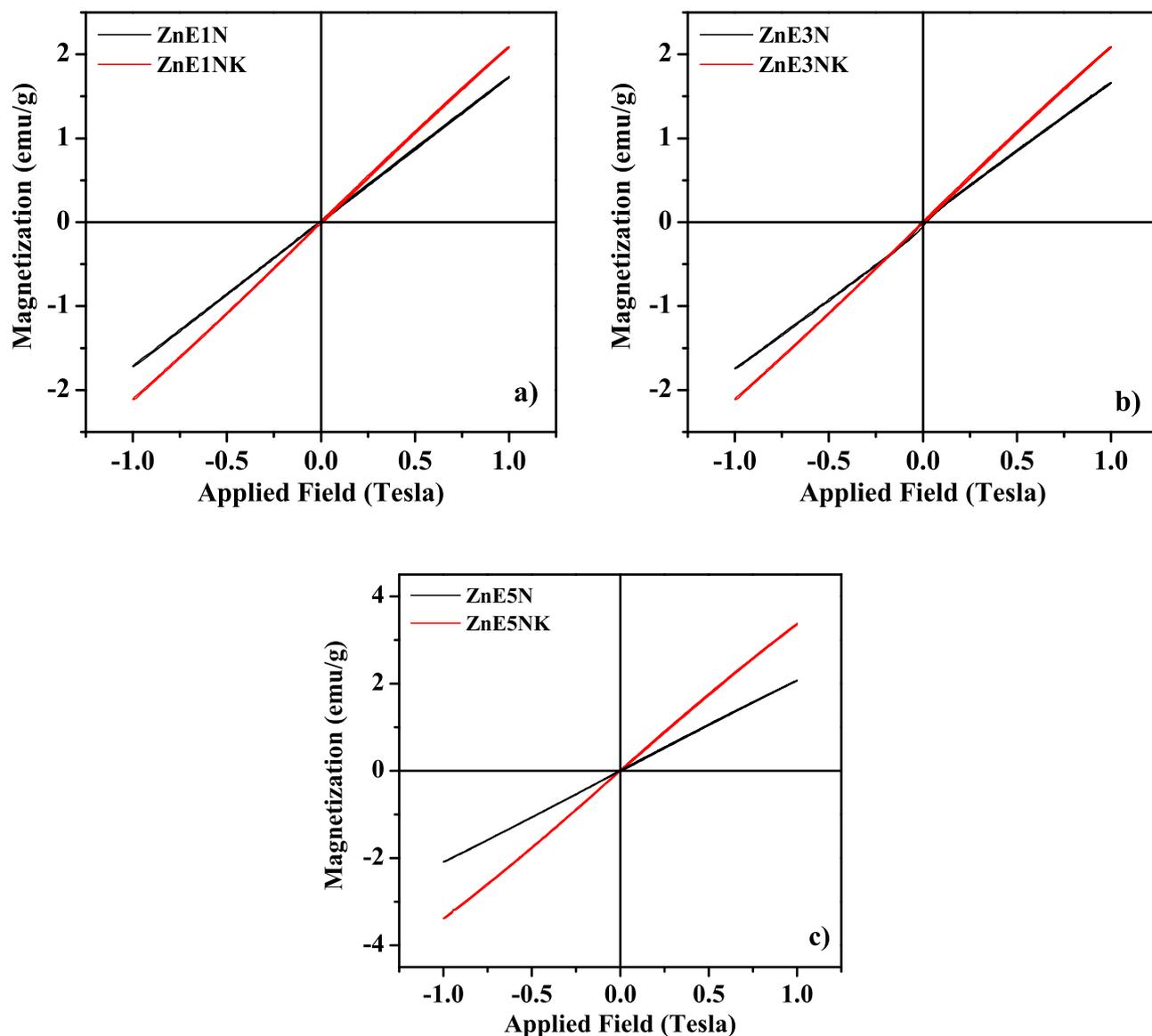
**Fig. 9.** Magnetic properties of samples (a) ZnE$_1$N and ZnE$_1$NK (b) ZnE$_3$N and ZnE$_3$NK, and (c) ZnE$_5$N and ZnE$_5$NK.

**Table 2**
Crystallite size, band gap, and magnetic behavior of Zn-ferrites before and after calcination.

| Catalysts | Crystallite Size | Band gap | Magnetic saturation ($Ms$) | Magnetic remanent ($Mr$) | Coercivity field ($Hc$) |
|---|---|---|---|---|---|
| | nm | eV | (emu/g) | | Tesla |
| ZnE$_1$N | 7.2 | 2.08 | 1.69 | 0.006 | 0.006 |
| ZnE$_3$N | 6.8 | 2.11 | 1.74 | 0.026 | 0.011 |
| ZnE$_5$N | 6.2 | 2.12 | 2.08 | 0.014 | 0.004 |
| ZnE$_1$NK | 15.8 | 2.05 | 2.07 | 0.013 | 0.007 |
| ZnE$_3$NK | 16.9 | 2.11 | 2.11 | 0.011 | 0.006 |
| ZnE$_5$NK | 14.4 | 2.10 | 3.36 | 0.007 | 0.006 |

*3.9. BET analysis*

The nitrogen adsorption-desorption isotherms of the ZnE$_1$N sample was measured using the Brunauer-Emmett-Teller (BET) technique to investigate the specific surface area and porous structure of the ZnE$_1$N and ZnE$_1$NK (Fig. 11). The findings revealed the specific relation between the adsorbate's concentration and its adsorption degree with the

adsorbent surface [16]. According to IUPAC classification, the ZnE$_1$N and ZnE$_1$NK are type IV isotherms, characterized by their mesoporous structure [40].

The branch preceding the hysteresis loop corresponded to monolayer adsorption whereas the hysteresis loop is associated with capillary condensation in relatively coarse pores or the interstices between the particles of the zinc ferrite spinel. The increase in the adsorption branch with the sharp decline in the desorption branch observed at P/Po was greater than 0.6 due to the capillary condensation of N$_2$ into the mesoporous structure, indicating good homogeneity of the nanocomposite [41]. The specific surface area of samples obtained from the nitrogen adsorption-desorption measurement was 134.3 and 125.5 m$^2$/g for ZnE$_1$N, and 114.28 and 110.27 m$^2$/g for ZnE$_1$NK, respectively. The decreased surface area of ZnE$_1$NK after calcination may be attributed to the recrystallization of the zinc ferrite and subsequent increase in average crystallite size [16]. The total pore volume of ZnE$_1$N is 0.265 cc/g and 0.250 cc/g for ZnE$_1$NK. The samples' average pore diameter calculated using the Barrett-Joyner-Halenda equation was 1.69 and 1.71 nm for ZnE$_1$N and ZnE$_1$NK, respectively. The increased pore size after calcination may be due to the particles' rearrangement that led to the loss of the actual pores. Thus, the large surface area would
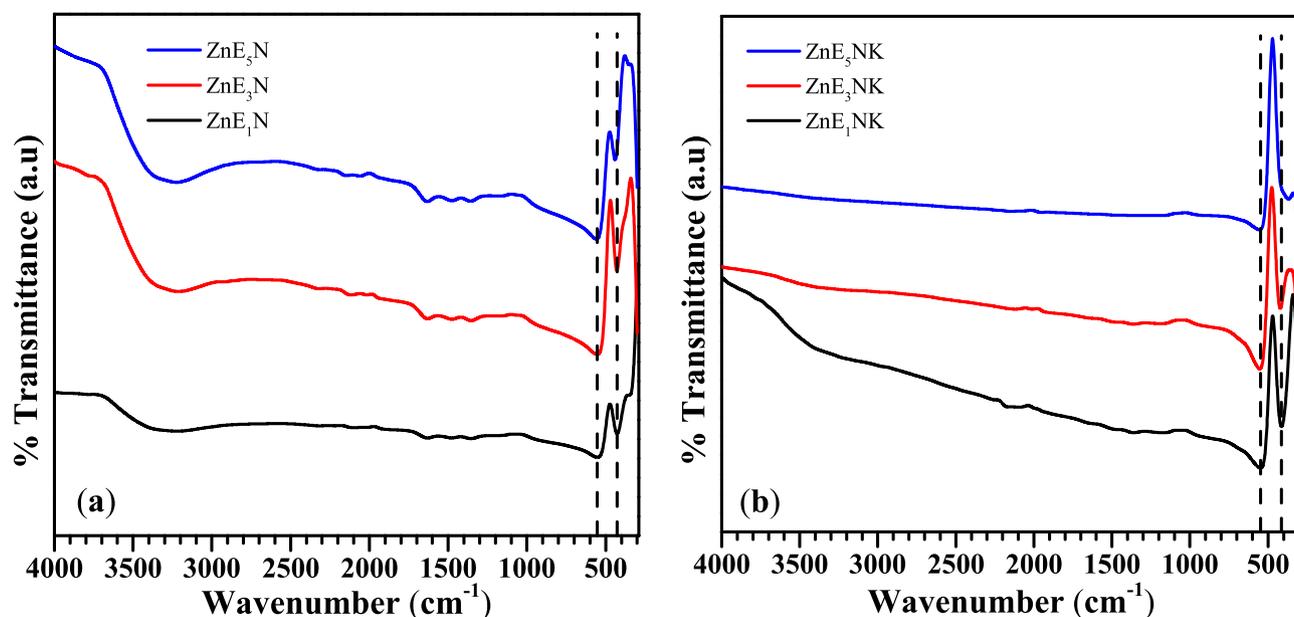
**Fig. 10.** FTIR spectrums of $ZnFe_2O_4$ before (a) and after calcination (b).
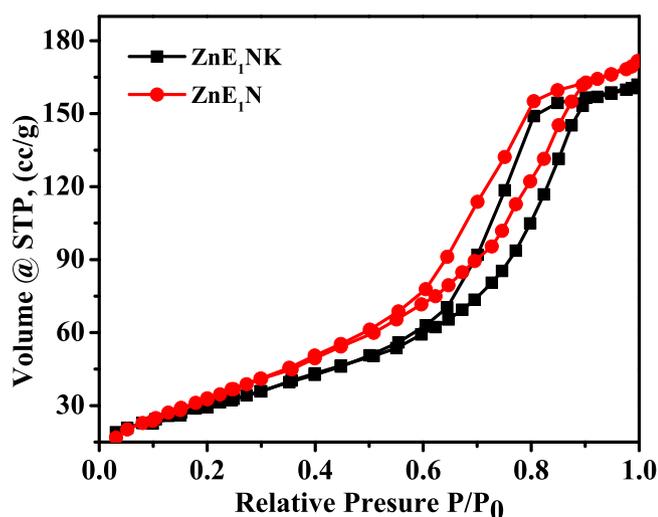


**Fig. 11.** Nitrogen adsorption–desorption isotherms of the $ZnE_1N$ and $ZnE_1NK$.

make this material useful in photocatalytic processes.

### 3.10. Photocatalytic activity

Photocatalytic activity of the zinc ferrite prepared samples was determined based on the degradation of direct red 81 dyes using direct sunlight as a light source (Fig. 12). The experiment was carried out in triplicates. Sunlight is used for the photocatalytic process because it is an abundant, low-cost, and environmentally friendly, renewable source of clean energy. Fig. 12a shows the photocatalytic activity of all prepared samples on the degradation of direct red 81 dye under direct sunlight. The results showed that all catalysts have good activity against the degradation of direct red 81 dyes. The highest percentage of degradation was obtained using $ZnE_1N$ as expected because of its smaller particle size and homogeneous morphology compared to other samples that contain more active sites to interact with the light to produce hydroxide radicals as illustrated in the electron images (SEM and TEM images). Hydroxyl radicals are known to be responsible for the photodegradation process of organic pollutants in the presence of natural sunlight.

The effect of dye concentration on the rate of degradation was determined by varying the concentration of direct red 81, as shown in Fig. 12b using $ZnE_1N$ and $ZnE_1NK$ as catalysts. The optimal concentration was obtained at 30 mg $L^{-1}$ with degradation percentage of 99.8% and 95.8% for 2 h using $ZnE_1N$ and $ZnE_1NK$, respectively. Meanwhile, Fig. 12c shows the effect of irradiation time on the degradation percentage of direct red 81 dyes in the presence of $ZnE_1N$ and $ZnE_1NK$. It was found that the degradation of dyes increased with irradiation time since the amount of OH· radicals formed increased with time. The OH· radicals were active groups that played a role in breaking down the dyes molecules into simpler compounds (ex: $H_2O$ and $CO_2$) [42]. Furthermore, the maximum degradation percentage (99.66%) was obtained using $ZnE_1N$ for 2 h under solar irradiation. Since $ZnE_1N$ catalyst has higher dye decomposition efficiency than its calcined counterpart, all further experiments were performed using $ZnE_1N$.

An important property of photocatalysts is their recovery and repeated usage in catalytic processes. To test this characteristic, the $ZnE_1N$ catalyst was repeatedly used in the photodegradation process of direct red 81 dyes under natural sunlight. In between each cycle, the catalysts were recovered via filtration, washed with distilled water, and dried at 105 °C. Fig. 12d shows the degradation percentage of direct red 81 dyes using $ZnE_1N$ for four cycles which demonstrated that the catalyst remained fairly effective after being used for 4 cycles, indicating its stability and ability to be used repeatedly. Photodegradation of dyes/organic pollutants by different catalysts can be seen in Table 3.

The kinetics for photocatalysts are described in the Langmuir-Hinshelwood model, which can be expressed by $ln(C_0/C_t) = kt$ when the dye concentration is relatively low (where $C_0$ is the initial concentration of dye, $C_t$ is the concentration at time $t$ and $k$ is the apparent first-order rate constant). By plotting $ln(C_0/C_t)$ vs irradiation time, $t$, $k$ can be determined, which can be used as an assessment index for evaluating the photocatalytic efficiency of synthesized materials. Based on Fig. 12e, it was observed that the kinetic plot for catalysts was somewhat linear. Hence, the photodegradation reaction of direct red 81 dyes with $ZnE_1N$ catalyst is of the first order with an $r$ value of 0.985 and $k = 0.01204$ s$^{-1}$.

The primary mechanism of photocatalytic activity (Fig. 13) in the presence of $ZnFe_2O_4$ as a catalyst was initiated through the electrons transfer ($e^-$) from the valence band (VB) to the conduction band (CB) if a certain amount of photon energy was absorbed by the electrons, leaving a hole ($h^+$) in the VB. Then, CB's electrons reacted with $O_2$ in the water to form superoxide radical anions through a reduction reaction.
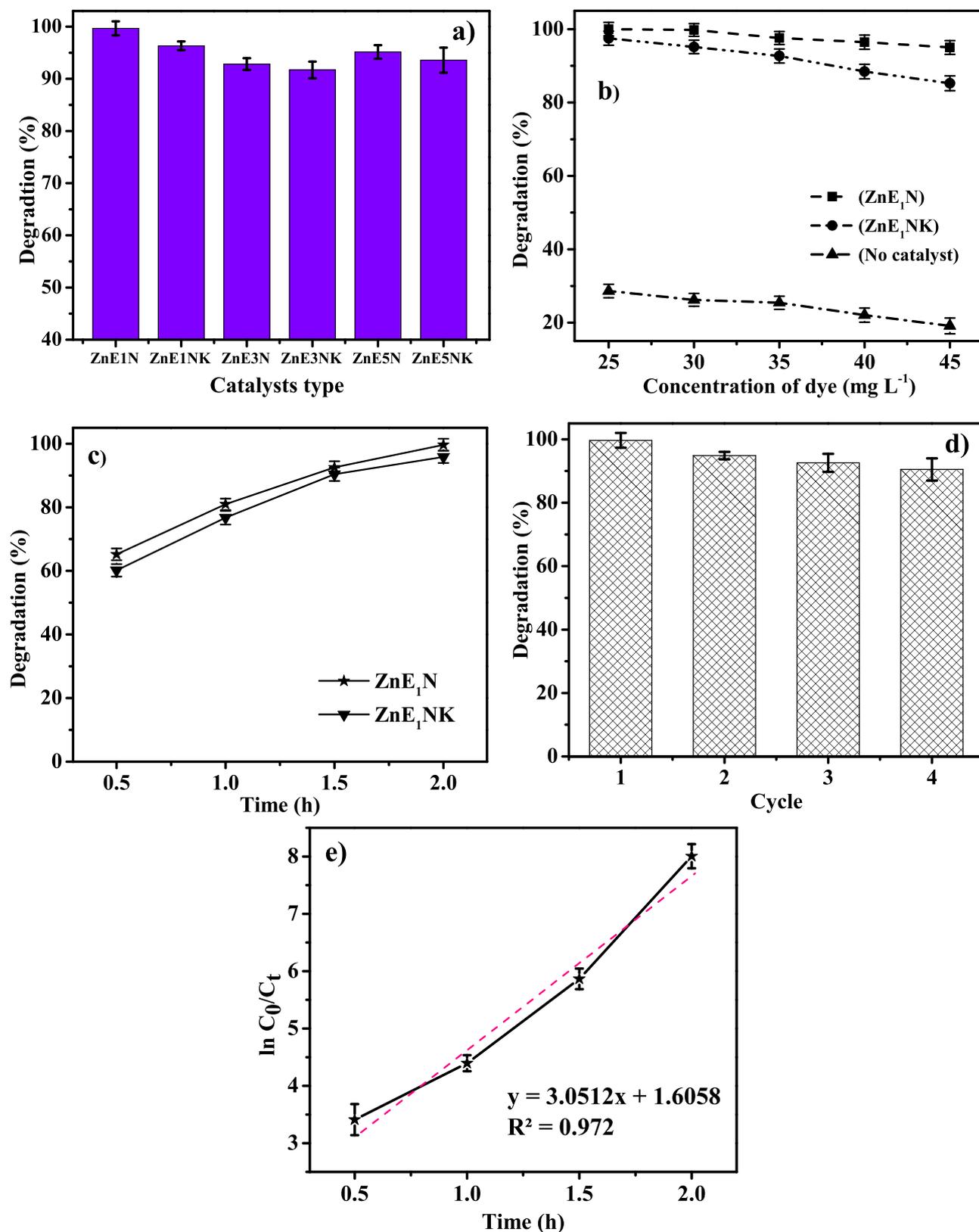
**Fig. 12.** Degradation percentage of the dye with various catalyst, dye concentrations, and irradiation time (a-c), photocatalytic stability determination (d), and reaction kinetics of the photocatalytic process (e).

Superoxide radicals then underwent protonation to form $H_2O_2$, which then dissociated to form OH· radicals. This radical avoided the recombination of $e^-/h^+$ pairs and participated in the oxidation process. On the other hand, $OH^-$ derived from the decomposition of water molecules reacted with the hole ($h^+$) in VB through oxidation reactions, forming OH· radicals. Furthermore, OH· radicals attacked the organic pollutants or dyes to form unstable intermediates and broke them down into simple molecules, i.e., water, and carbon dioxide gas [46].

**Table 3**

Comparing photodegradation of dyes/organic pollutants by different catalysts.

| No | Catalyst | Synthesis method | Name of dyes/ organic pollutant | % of degradation | Ref. |
|---|---|---|---|---|---|
| 1. | $ZnFe_2O_4$ | Green synthesis | Direct Red 81 | 99.66 | This work |
| 2. | $ZnFe_2O_4$ | Co-precipitation | Methylene Blue | 99 | [11] |
| 3. | $ZnFe_2O_4$ | Combustion route | Rhodamine B, Methylene Blue | 98 | [14] |
| | | | | 95 | |
| 4. | $ZnFe_2O_4$ | Co-precipitation | Acid Blue 113 | 92 | [16] |
| 5. | $ZnFe_2O_4$ | Green method | Methylene Blue | 99.6 | [21] |
| | | | Evan Blue | 89 | |
| 6. | $ZnFe_2O_4$ | Green synthesis | Tetracycline | 80 | [34] |
| 7. | $ZnFe_2O_4$ | Combustion | Congo Red | 95 | [9] |
| | | | Rhodamine B | 93.6 | |
| 8. | $ZnFe_2O_4$ | Hydrothermal | Methylene blue | 90 | [43] |
| 9. | $NiFe_2O_4$/natural mineral | Microwave hydrothermal | Sodium dodecyl benzene sulfonate | 100 | [13] |
| 10. | $ZnFe_2O_4$/ Polyaniline | Co-precipitation | Rhodamine B | 85 | [10] |
| 11. | $ZnO\text{-}CoFe_2O_4$ | Green method | Direct Yellow | 95 | [28] |
| 12. | $MnFe_2O_4$ | Green method | Direct Red 81 | 56.5 | [30] |
| 13. | $SnFe_2O_4$/Polypyrrole | Sonication | Tartrazine | 100 | [44] |
| 14. | $NiFe_2O_4$ | Co-precipitation | Methyl Orange | 72.66 | [45] |
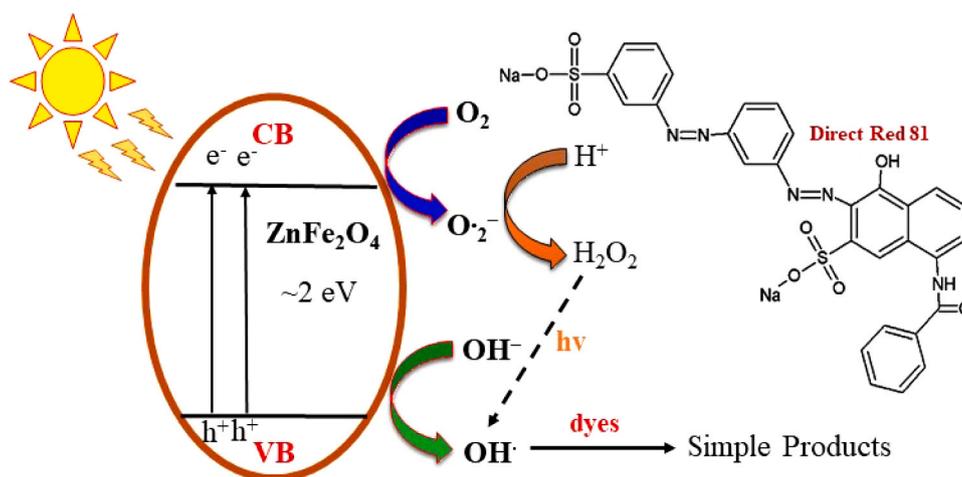


**Fig. 13.** The proposed photocatalytic mechanism of the direct red 81 using $ZnFe_2O_4$ as a catalyst.
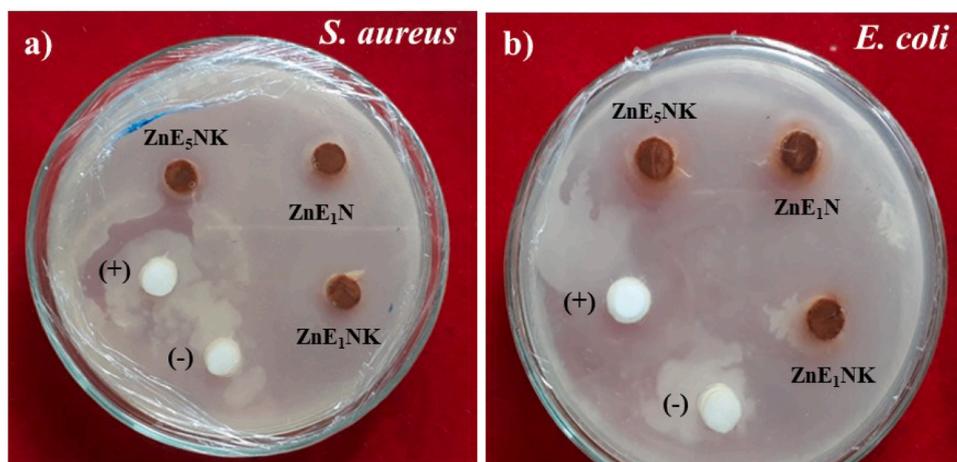


**Fig. 14.** Antibacterial activity of $ZnFe_2O_4$ on *S. aureus* (a) and *E. coli* (b).

## 3.11. Antibacterial activity

The antibacterial assay for $ZnE_1N$, $ZnE_1NK$, and $ZnE_5NK$ samples was conducted using Gram-positive (*S. aureus*) and Gram-negative (*E. coli*) as pathogens. The results are shown in Fig. 14. $ZnE_1N$, $ZnE_1NK$, and $ZnE_5NK$ ferrite samples showed positive antibacterial activity against *S. aureus* with an inhibition zone of 10.9, 10, and 9.4 mm in respectively, whereas for *E. coli* were 9.6, 4.9, and 7.5 mm, respectively. Amoxicillin and water were used as a positive and negative control in this experiment. The bacterial activity of zinc ferrite depends on the size, morphology, chemical molecule diffusion ability, and the discharge of metal ions. In addition, the surface area is crucial in the elevated and promising activity of $ZnFe_2O_4$ nanoparticles against all the tested pathogenic microbes. There are many reasonable mechanisms for the antimicrobial action of $ZnFe_2O_4$ nanoparticles, including radical groups generated through the redox reactions during the photocatalytic process attack the bacteria's cell membrane components and inhibit the growth of these microorganisms [47].

## 4. Conclusion

The ferrite spinel of $ZnFe_2O_4$ nanostructures has been successfully synthesized using Simbang Darah extract as a capping agent via the hydrothermal method. Characterization using several instruments showed that the samples were in the form of homogeneous spherical granules. The synthesized zinc ferrite nanoparticles exhibited paramagnetic and absorbent behavior under the visible light with a band gap energy of ~ 2 eV. The $ZnFe_2O_4$ nanoparticles showed effective photodegradation for direct red 81 at 99.66% for 2 h under solar light irradiation. Moreover, these nanoparticles demonstrated antibacterial activities against *S. aureus* and *E. coli*. Therefore, it can be concluded that Simbang Darah extract provided a good yield of ferrite nanoparticles with desirable properties. Thus, it can be used to synthesize other materials in nanoscale.

## CRediT authorship contribution statement

**Rahmayeni:** Conceptualization, Methodology, Validation, Writing - Original Draft, **Rike Febrialita:** Investigation, Visualization, **Yeni Stiadi:** Writing - Original Draft, **Yulia Eka Putri:** Visualization, Validation, **Nofrijon Sofyan:** Writing - Review & Editing**, Zulhadjri:** Writing - Review & Editing.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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