Ternary nanocomposite of ZnFe$_2$O$_4$/α-Fe$_2$O$_3$/ZnO; synthesis via coprecipitation method and physical properties characterization

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Abstract

ZnFe$_2$O$_4$/α-Fe$_2$O$_3$/ZnO, the ternary transition metal oxide nanocomposite powder was successfully synthesized by facile coprecipitation method directly from metalorganic precursors within 4 h of processing time considerably shorter than other reported methods. To study the post-synthesis thermal treatment effects on physical properties, calcination process was applied at 500, 600, and 700 °C individually for one hour. The structure, phase formation, morphology, and optical features of the samples were characterized by employing powder X-ray diffraction, scanning electron microscopy, and UV–visible spectroscopy. The results confirm the synthesis of pure and homogeneous composites comprised of nanoparticles with good crystallization in a narrow range of crystallite sizes between 25–39 nm. The particle sizes also were estimated between 48–93 nm. The optical property was studied by recording the absorbance spectrum from 200 to 700 nm. The absorption pattern illustrates the nanocomposite can be driven by UV and visible wavelengths with good efficiency that is more desirable for intended applications like photocatalytic activities compare with the individual components. By applying Tauc’s method, the allowed direct bandgap and indirect bandgap of the prepared nanocomposite were estimated to be around 2.28 and 2.75 eV, respectively.

Keywords ZnFe$_2$O$_4$/α-Fe$_2$O$_3$/ZnO nanocomposite · Spinel nanoparticles · Ternary nanocomposite powder · Calcination effects · Optical transition

1 Introduction

Composites comprise all phases in nanoscopic dimensions have a high potential of applications in various areas also desirable for homogeneous doping and incorporation in materials matrix for filtering, sensing, and catalytic activities nowadays. As highly interesting semiconducting transition metals oxides (TMOs), the zinc ferrite (ZnFe$_2$O$_4$), hematite (α-Fe$_2$O$_3$), and zinc oxide (ZnO) nanoparticles (NPs) have received much research attention to make novel networks by incorporating or attaching these NPs into other material structures for improving their applications [1–8].

The importance of composite applications becomes crucial when individual usage limitations make barriers, although the significant properties of these three compound NPs are indisputable. For instance, as an n-type semiconductor, α-Fe$_2$O$_3$ is non-toxic, photochemically highly stable with a narrow bandgap. However, rapid recombination of photogenerated carriers and low conductivity considerably limit its photoelectrochemical (PEC) efficiency [9–12]. In the case of zinc oxide, this TMO is a widely used photocatalyst, possess the high ability of oxidation, cost efficiency, versatility in synthesis, and ease of crystallization, whereas this n-type compound with a large amount of bandgap energy ca. 3.2 eV is weak to take advantage of absorbing visible wavelengths of the light spectrum [13–15] to generate charge carriers. On the other hand, ZnFe$_2$O$_4$ NPs have a vast range of applications due to their amazing electrical and magnetic properties [16–18]. Therefore, these NPs as complements in the form of composite reduce or cover disadvantaged properties of each other for targeted applications. For instance, in the case of photocatalytic applications despite the mentioned disadvantages of hematite and zinc oxide, spinel zinc ferrite has
demonstrated that excellently is driven by incident visible light due to its bandgap energy which is around 2 eV [19]. Besides, the presence of this p-type semiconductor in the composite is a promising route to suppress electron–hole recombination [20]. Consequently, a ternary composite of ZnFe₂O₄/α-Fe₂O₃/ZnO is more efficient for most processes instead of the individual components. As the most practical application for this class of composite, photocatalytic activities such as water pollution degradation [21–24], CO₂ reduction [25], PEC water splitting and hydrogen production [3, 26, 27], and PEC oxidation of water [28] have become topics of interest as well as for biotechnology and nanomedicine [29–34] applications.

For preparing TMO semiconductor composites, there are different methods, including sol–gel method [35–37], sonochemical [38, 39], hydrothermal [1, 21, 22], microwave combustion [40–42], and coprecipitation [43, 44]. Among various chemical methods for synthesis, the coprecipitation process has several advantages compared with other methods including, good homogeneity, low cost, high purity of the product, and not requiring organic solvents. Moreover, the experimental conditions play important roles in determining the shape, size, and purity and hence the drastic modifications of the properties [27, 45]. Therefore, we employed the coprecipitation method in this work.

Despite many reports for synthesis binary composites of ZnFe₂O₄, α-Fe₂O₃, and ZnO [1, 46–48], successful preparation of ternary composite through one approach has been reported just in a few works to date. Xiaojuan Li et al. prepared this ternary composite via hydrothermal reaction with subsequent thermal treatment [21]; the result was shuttle-like composites with approximately 5–6 µm mesoporous structure achieved during more than 30 h followed by 3 h of thermal treatment. Another work performed by J.P Dhal et al. achieved by the hydrothermal method followed by calcination too, process time of synthesis exceeded 14 h excluded calcination process, which resulted in cubical-shape composites with seizures in the range of 100–200 nm [22]. The last one is the synthesis of ternary hybrid nanodiscs by microwave-assisted coprecipitation method reported by Shipra Choudhary et al. [49]; however, the coprecipitation method considerably has decreased the processing time, the influence of microwave yielded in increasing the size of the composite to the average size of 242 nm in contrary. These ternary composites face limitations for most applications due to their special shapes and large sizes.

This work presents experimental conditions of preparing ternary TMOs (TTMOS) nanocomposite by single coprecipitation approach in detail. This method has some considerable advantages compared with the above discussed ternary composites. First, the preparation time considerably was decreased without any effect on quality within 3.5 h. Second, the spherical TTMOs nanocomposites with sizes in the range of 48–93 nm are more feasible for incorporating into other nanomaterial matrices to configure novel nanonetworks. Moreover, the simplicity of the synthesis method, besides the availability of the precursors, and equipment makes it much more economical and suitable for industrial production or further research.

Furthermore, in this work, the effects of different temperatures of post-synthesis thermal treatment on the structure, phase formation, morphology, and optical properties have been studied by employing X-ray diffraction (XRD), Scanning electron microscopy (SEM), and UV–visible spectroscopy as the characterization techniques.

## 2 Materials and method

### 2.1 Materials

Iron (III) chloride hexahydrate (FeCl₃·6H₂O, Molar Mass: 270.33 g mol⁻¹), zinc chloride (ZnCl₂, Molar Mass: 136.30 g mol⁻¹), hydrate ethanol (C₂H₈O₂), and sodium hydroxide (NaOH, Molar Mass: 40.00 g mol⁻¹) are the Merck company materials which were used in this work without any purification as the precursors.

### 2.2 Synthesis

In this method, 68.143 g of zinc chloride (ZnCl₂) and 162.2 g of iron (III) chloride (FeCl₃) were separately be ground in a mortar then dissolved in de-ionized (DI) water to produce two different solutions, which were denoted as solution 1 and solution 2, respectively. In the next step, solution 2 was added into solution 1 under stirring. Sodium hydroxide (NaOH) solution was added to the mixture, which was heated and stirred at ca. 60 °C for 30 min until liquid with pH 12 was obtained. The molar ratio of Zn/Fe was 1:2.

The pre-treated solution was washed with ethanol and DI water, respectively. Heat treatment of the powder product was carried out at 100 °C for 3 h in the oven. The synthesized nanocomposite was divided into three equal parts and heated them at 500 °C, 600 °C, and 700 °C individually for 1 h under atmosphere condition to study annealing temperature effects on their physical properties. As the final step of purification, the ultrasonic bath was applied to dissolve powders in ethanol for 45 min then dried at ambient temperature after filtering. The schematic of the steps and procedure involved in the synthesis of ZnFe₂O₄/α-Fe₂O₃/ZnO nanocomposite samples is given in Fig. 1.

### 2.3 Characterization

A PANalytical X’Pert High Score X-ray Diffractometer-Philips PW1730 equipped with Cu anode (λ=0.154060 nm)
was used in the crystallography investigation in this work. Diffraction was carried out at room temperature within 2θ angular range of 9.97°–79.97° with a constant step size of 0.05°. The morphology of the synthesized ternary nanocomposite powder was characterized by employing scanning electron microscopy (SEM), model KYKY-EM3200, operated at an acceleration voltage of 26 kV. The UV–vis absorption spectrum was obtained using SHIMADZU UV-1800 spectrophotometer (50–60 Hz, 240 V). DI water was used as the background correction material suspension was scanned in the region from 200 to 700 nm with 1 nm steps in the quartz cuvette of 1 cm path length.

3 Results and discussion

3.1 Structural Investigation

The structure and phase composition of the synthesized powders was evaluated by employing the powder X-ray diffraction (XRD) method. The XRD patterns obtained from ternary nanocomposites synthesized by coprecipitation method after thermal treatment process at 500, 600, and 700 °C are presented in Fig. 2, along with PANalytical fitting and data from the literature [50, 51]. Highly intense sharp peaks with small full width at half maximum (FWHM) in the graphs indicate products comprised of high-quality crystallized nanoparticles. All the diffraction peaks denoted on the graphs are matched with the reference numbers, (ICSD NO. 98–005-8878) for cubic lattice system zinc ferrite, the prominent peak (311) is located at 2θ ≈ 35.72°, as well as (440) and (622) planes occurred at 62.51° and 75.55°, respectively. The hematite nanoparticle with rhombohedral unit cells illustrated the prominent peak (104) located at 2θ ≈ 33.20° in addition to other planes (024), (012), (116), (113), (300), (1010), (018) at 49.57°, 24.22°, 54.17°, 40.95°, 64.10°, 72.07°, 57.77°, respectively, in order of their intensities matched with the reference card (ICSD NO. 98–001-2729). The hexagonal zinc oxide nanocrystals peaks are (101) revealed at 36.39° and (002) around 34.56° matched with (ICSD NO. 01–075-0576). Raising calcination temperature resulted in the formation of new planes of ZnO and ZnFe₂O₄. In contrast, this thermal treatment process causes a decline in the intensity of hematite peaks at 700 °C and vanishing some tiny peaks of this component. As can be seen, 600 °C provides a suitable condition for ZnO nanocrystals for growing up also getting mature by exposing (100) and (110) planes positioned at 31.7° and 56.68°, respectively, toward incident X-rays, whereas condition of 700 °C provides this chance for ZnFe₂O₄ crystals. As is shown, (111), (220), (400) and (422) located at 2θ ≈ 18.24°, 29.96°, 42.93° and 53.17°, respectively, are the new planes of zinc ferrite which were emerged in the pattern after the thermal process at 700 °C. The effect of calcination temperature on the crystallite size is presented in Fig. 3. The crystallite size was estimated using the Debye–Scherrer equation:
where $D$ is the crystallite size in nm, $K$ is crystallite shape factor (0.89), $\lambda$ is X-ray wavelength coming from Cu-Ka (0.154060 nm), $\beta$ is FWHM in radian and $\Theta$ is Bragg diffraction angle. Crystal size at different temperatures was estimated based on the average calculated size for all the peaks ascribed to each component reported in Table 1. The diagrams in Fig. 3 indicate gentle downward trends for $\alpha$-Fe$_2$O$_3$ and spinel ZnFe$_2$O$_4$ crystallite size by increasing temperature from 500 to 700 °C. A decrease in crystal size and intensity of peaks indexed to $\alpha$-Fe$_2$O$_3$ is in good agreement with published works [21]. However, appearing this trend for ZnFe$_2$O$_4$ crystals can be elucidated by considering lattice dislocation removal. Therefore, atoms are more likely to find the best position in the crystal structure in higher kinetic energy via raising the temperature and yielded in forming nanoparticles with more favorable crystallinity instead of increasing the size. Changing in the ZnFe$_2$O$_4$ lattice constant ($a_{500} = 0.8350$ nm, $a_{600} = 0.8354$ nm and $a_{700} = 0.8400$ nm) toward standard length and reported value [52] can confirm this claim. The zinc ferrite lattice constant was calculated using the following equation:

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2}$$

ZnO crystals reached a larger size at higher treatment temperature, which is in good agreement with the reported works [53]. Variation of interplanar spacing and intensity of prominent peaks as a function of calcination temperature are reported in Table 1.

### 3.2 Morphology investigation

SEM images of the ZnFe$_2$O$_4$/$\alpha$-Fe$_2$O$_3$/ZnO powders given in Fig. 4 illustrate the morphology of the synthesized nanoparticles. Figure 4a shows the nanoparticles of the as-prepared
powder. Thermal treatment effects on the shape, size of the nanoparticles, and agglomeration of the components are evident in Figs. 4(b–d) are ascribed to powders calcined at 500, 600, and 700 °C, respectively. The agglomeration is found to be decreasing after passing calcination at 500 and 600 °C; the nanoparticles are recognizable as free-standing spherical particles with smooth surfaces in an almost uniform size range. In contrast, the sample calcined at 700 °C shown an increase in agglomerating that can be explained by coalescence and weight loss [50]. The range of particle size for all samples was estimated by SEM measurement Fig. 5 illustrates the results. The least diversity in size belongs to the specimen calcined at 600 °C in which the size of 25 to 75 percent of the particles is between 73–80 nm range.

Overall, the sizes of nanoparticles prepared in this work under different thermal treatments are between 48 to 93 nm, much smaller than the reported size (100–200 nm) of the same composite prepared by the hydrothermal process [22].

### 3.3 Optical investigation

The UV–visible absorption spectrum of the ZnFe$_2$O$_4$/α-Fe$_2$O$_3$/ZnO nanocomposite powder calcined at 500 °C is given in Fig. 6. As can be seen, the obtained spectrum is different from the absorbent behavior of individual components also not similar to the reported spectra of the binary or doped composites in the cases of absorption edges and intensities [10, 46, 54, 55]. The two predominant
absorption regions are observed between 260 to 340 nm and a broad region from 340 to 700 nm. The band occurred at 311 nm (3.98 eV), indicates the presence of ZnO nanoparticles in the composite. The other characteristic absorption band of ZnO around 375 nm (3.31 eV) observed shifted to the longer wavelength as a tinny shoulder at ca. 380 nm (3.26 eV) likely be masked with hematite absorbance region. Therefore, the calcination process has illustrated slight red sifting in the characteristic peaks [54, 56]. The broad region as a cumulative wide peak includes α-Fe2O3 and ZnFe2O4 bands. Transitions in hematite occurred at ca. 361 nm (3.43 eV) and 617 nm (2.00 eV) are ascribed to a ligand-to-metal charge transfer (6t1u → 2t2g), and ligand-free (6A1g → 4E), respectively, [22] are in this range of wavelengths. Moreover, evidence of zinc ferrite optical transitions observed around 472 nm (2.62 eV), 493 nm (2.51 eV) are attributed to (6A1g → 4T2g(D)), (6A1g → 4A1g(G)) transition, respectively, and 558 nm (2.22 eV) band with maximum value in this range can be assigned to O2− + Fe3+ → O− + Fe2+ direct transition and the weak signal at 643 nm (1.92 eV) can be considered of O2− + Fe3+ → O− + Fe2+ indirect transitions [19]. The notable feature of this optical pattern is the prepared TTMOs nanocomposite powder revealed good absorbance behavior in both UV and visible regions without a sign of the absorption edge. This result confirms the synthesized ternary nanocomposite is driven by both UV and visible light simultaneously with good efficiency. Figure 7 shows the Tauc’s plot for the energy gap evaluation of the ZnFe2O4/α-Fe2O3/ZnO nanocomposite powder calcined at 500 °C using UV–visible spectroscopy data. The optical band gap energy (E_g) is given by the below classical relation known as Tauc’s method [57]:

**Fig. 5** Size range of the ZnFe2O4/α-Fe2O3/ZnO nanoparticles versus calcination temperature

**Fig. 6** UV–visible absorption spectrum of the ZnFe2O4/α-Fe2O3/ZnO nanocomposite powder prepared by coprecipitation method calcined at 500 °C

**Fig. 7** Tauc’s plot for estimating optical band gap energy of the ZnFe2O4/α-Fe2O3/ZnO nanocomposite powder calcined at 500 °C using UV–visible absorption data a direct optical band gap energy (DBG) and b indirect band gap energy (IBG)
$a(\nu)h\nu = A(h\nu - E_g)^n$

where $a(\nu)$ is absorption coefficient, $A$ is a proportionality constant, $h$ is Planck’s constant, $\nu$ is the incident photon’s frequency.

The value of $n$ depends on the type of optical transition between the valence band and conduction band, for allowed direct and indirect transitions $n = 1/2$ and 2, respectively. Absorption coefficient $a(\nu)$ has been defined by Beer-Lambert’s law:

$$a(\nu) = 2.303 \frac{A_{bs}(\nu)}{L}$$

where $A_{bs}(\nu)$ is the measured absorbance corresponding to each incident frequency and $L$ is the optical path length which was assumed to be 1 cm, equal to the thickness of the quartz cuvette of the spectrometer.

The bandgap of the products can be determined by plotting $(ah\nu)^2$ and $(ah\nu)^{1/2}$ versus $h\nu$ for allowed direct and indirect transitions, respectively. Extrapolating the straight-line portion of the plot to the photon energy axis at the $ah\nu^{1/2} = 0$ gives the $E_g$ values. Determining the linear region is the controversial part of this method, which causes inaccuracy in some cases, particularly in those types of aqueous solution nanocomposite materials in expose of incident wave-lengths [58]. Considering this fact, as is shown in Fig. 7(a, b), the most suitable fittings have been tried for extrapolations. Consequently, the direct optical bandgap of produced nanocomposite powder was estimated to be around 2.28 eV, and for the allowed indirect bandgap, the value was 2.75 eV.

4 Conclusion

A pure ternary semiconducting nanocomposite of ZnFe$_2$O$_4$, $\alpha$-Fe$_2$O$_3$, and ZnO was successfully prepared via one approach coprecipitation method. The unique feature of this report to emphasize is, due to the simplicity of the method, availability of the precursors and equipment; there are no limitations or challenges for research or industrial-scale production of this TMO ternary nanocomposite. The other advantages are preparing the process in a pretty shorter time and producing the very smallest components particle size compare to the hydrothermal reported method. The XRD, SEM, and UV–visible analysis demonstrated the formation of uniform and homogenous nanoparticles with crystallite sizes between 25 to 39 nm. The effects of post-synthesis thermal treatment on physical properties also were investigated in the present work. The results indicate the formation of new planes, vanishing planes with the faint intensity of $\alpha$-Fe$_2$O$_3$, crystallite size, and lattice constant, are influenced by calcination temperature. Particle size in all samples was below 93 nm, and the most uniformity in size was observed in the specimen calcined at 600 °C. Optical characterization illustrated that the product as an aqueous composite is driven by both UV and visible wavelengths with good efficiency without a sign of the absorption edge. To develop the study, we work on the synthesis steps by adding the carbon nano-structures, including graphene and CNTs. This research is defined to examine incorporating or appending crystals of the nanocomposite components to the carbon matrix and configure a more efficient nano complex to characterize the magnetic and photocatalytic properties for different applications. This process has the potential to try for vast types of materials matrix.

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Declaration

Conflict of interest The authors have no conflicts of interest to declare that are relevant to the content of this article.

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