Synthesis, Characterization, and Multifunctional Applications of Cu-Fe and Ni-Fe Nanomaterials

Kausar Shaheen, Zarbad Shah,* Ambreen Asad, Tofail Arshad, Sher Bahadar Khan, and Hongli Suo*

Cite This: ACS Omega 2020, 5, 15992−16002

ABSTRACT: Cu-Fe and Ni-Fe nanomaterials (NMs) were successfully obtained via a coprecipitation route. Phase analysis and the micro- and physiochemical structure studies for the as-synthesized NMs were carried out with advanced techniques such as TEM, SEM, XRD, XPS, BET, DRS, TGA, and FTIR. Particles with size ranging from 25 to 70 nm were displayed by all the characterization techniques. A surface area of ∼4.48 and 36.52 m²/g and band gap energies of ∼1.79 and 1.48 eV were calculated for Cu-Fe and Ni-Fe NMs, respectively. Saturation magnetization (Ms) ∼77.95 emu/g (for Cu-Fe) and 27.70 emu/g (for Ni-Fe) revealed superparamagnetism for both the NMs. The presence of ethanol and methanol as sacrificial agents contributed effectively toward electrocatalytic H-evolution as compared to pure NMs. Furthermore, under solar light irradiations, Cu-Fe and Ni-Fe NMs displayed 85 and 91% degradation during a time interval of 50 and 110 min, respectively, for toxic industrial methylene blue (MB) dye. Different operational variables such as the catalyst amount, pH values, various scavengers, reusability, and stability were thoroughly investigated. Moreover, in situ analysis was carried out in order to determine the mechanism for degradation reactions. A detailed study about various applications categorized the synthesized NMs as efficient candidates for toxic industrial waste cleanup and energy production at an industrial level.

1. INTRODUCTION

Metal oxide-based nanomaterials (NMs) have been utilized generously for the construction of photocells, transistors, diodes, sensors, catalysis, and various other applications regarding optoelectronics and photonics.1 Self-cleaning for glasses in polluted environments, environmental purification, pollutant remediation, and energy production could be effectively carried out with various NMs.2−8 Different morphologies including single crystals, spheres, rods, cubes, and thin films with controlled particle growths can be synthesized via the coprecipitation method by utilizing aqueous solutions at higher pressure.9,10 Factors such as temperature, pH, reaction rates, and capping agents can greatly influence the size, nucleation, and crystallization of particles inside metal oxide products and are extensively investigated by many researchers.11 Oxygen and hydrogen evolution reactions (i.e., OER and HER) during the water splitting technique can be utilized in the field of sustainable energy and storage applications.12−14 Multistep proton coupling and electron transference mechanism is seriously limiting the water splitting and hence making the process complex and kinetically sluggish.15,16 Therefore, it is very essential to enhance the reaction rate, to minimize the overpotential and to accelerate the energy conversion with utilization of some well-organized and proficient photoelectrocatalysts. Various NMs such as Pt/C, RuO₂, and IrO₂ have been intensively explored as an efficient HER and OER catalysts under an alkaline medium. However, due to the high cost of these materials, further investigations are still required to explore and develop some durable, active, and low-cost alternatives, such as transition metal oxides,17−19 perovskites,20−23 and molecular catalysts.24,25 ZnO- and TiO₂-based nanocomposites remained the most attractive, nonprecious, and best alternative for the last few decades due to their better morphology, larger surface area, and active site density. However, a large energy band gap, poor dispersion, and recycling difficulties have seriously lowered the photodegradation efficiency of these NMs.26−29 Thus, it is still a hot issue for researchers to fabricate a solar light-driven photocatalyst with exceptional characteristics to meet the requirements of utilizing solar energy.

Textile and dyeing industries pose serious ecological problems as they discard excessive dye effluent loads into...
water resources. About 20% of total dyes are washed off and added into the water bodies during the dyeing processes. Various remediation techniques such as filtration, adsorption, and coagulation have been employed to date. However, these methods are expensive, less efficient, and they only involve separation of dyes along with generation of secondary products, which need further processing. Semiconducting metal nanocomposites are of great importance as they can be used for catalytic degradation and energy production because they involve simple and low-cost synthesis steps with attractive optical properties and production of final biodegradable products. Previously many researchers attempted to improve the photocatalytic activity of various metals such as gold, silver, copper, iron, and nickel. Transition metals are considered to be very favorable for photodegradation of toxic dyes. Among the transition metals, copper nanoparticles (CuNPs) are very efficient and most widely used in catalysis, optics, magnetism, and heat transfer applications. The major issue in CuNPs is their extreme sensitivity to air and hence the aggregation. Various protecting agents, such as organic ligands and polymers are being utilized in order to develop air-stable CuNPs.

Cocatalyst loadings can promote the electron hole pairs and can improve the degradation efficiency of various NMs. Platinum and other noble metals can be used as favorable cocatalysts, but due to their high cost and less supply, it is crucially needed to investigate some noble metal-free cocatalysts. Transition metal such as Ni, Mo, and Co can be selected with improved and advanced properties. Ni-based nanocomposites are suggested to serve as an efficient candidate for degradation of organic dyes, production of H₂ from water splitting, and several magnetic applications.

Similarly, the materials involving iron have received remarkable importance due to their important utilization in various fields such as sensing, optics, magnetic recording, printing, MRI, and other biomedical applications. Iron nanoparticles can also be utilized in environment remediation techniques through conversion of solar energy into chemical energy. Various preparatory methods such as sol–gel, hydrothermal, electrochemical, and coprecipitation have been reported to fabricate the NMs. All these methods in common are complicated and expensive as compared to the coprecipitation route, which is relatively cost effective and convenient, involving an improved yield percent with reduced contamination.

This study is to develop bimetallic Cu-Fe and Ni-Fe NMs via the coprecipitation route. The simple and cost effectively synthesized samples exhibited superparamagnetism with Ms ∼77.95 emu/g for Cu-Fe and 27.70 emu/g for Ni-Fe NMs. A better H-production was revealed by the as-prepared NMs with the addition of sacrificial agents such as ethanol and methanol. The oxidation-resistant and stable NMs with better size and dispersion were also investigated catalytically against the commonly used toxic MB dye.

2. EXPERIMENTAL DETAILS

2.1. Materials Used. The compounds such as NaOH, Cu(NO₃)₂, Fe(NO₃)₃, Ni(NO₃)₂, C₂H₅OH, and CH₃OH were received from Sigma-Aldrich, while MB dye was obtained from BDH. These analytically pure chemicals were used without any further processing.

2.2. Synthesis of Cu-Fe and Ni-Fe NMs. Cu-Fe and Ni-Fe-NMs were prepared through the coprecipitation route. The chemicals such as [Ni(NO₃)₂ (18.27 g/mol), Fe(NO₃)₃(24.18 g/mol)] and [Cu(NO₃)₂(18.75 g/mol), Fe(NO₃)₃(24.18 g/mol)] were perfectly mixed with distilled water in equimolar ratios. Solutions were constantly stirred at room temperature. NaOH solution (0.1 M) was added in order to adjust the pH of the solutions. When the solutions were obtained with precipitates at a pH value of 9, they were further stirred at 60 °C for 2 h. Afterward, the solutions were filtered and finally washed with (H₂O:C₂H₅OH; 40:60%) in a centrifuge. The resultant precipitates were then dried in an oven for 24 h and were crushed into powder after cooling. The powders were annealed in a furnace at a temperature of 250 °C for 2 h in order to remove the unnecessary organic residues, nitrates, and hydroxide phases due to the utilization of precursor’s salt solutions. The annealed samples were stored in clean vials for further characterization and applications. The details about the preparation route for Cu-Fe and Ni-Fe NMs are schematically presented in Figure 1.

2.3. Instrumentation. Phase and crystallinity for the fabricated NMs were investigated through X-ray diffraction (XRD, CuKα-Analytical XPERT) with 2θ range ~10–90°. The chemical structure was investigated through Fourier transform infrared (FTIR) spectroscopy in a wavenumber range of 400-4000 cm⁻¹. Thermogravimetric analysis (TGA, PerkinElmer) was used to ensure the real content and thermal stability for the as-synthesized samples. A UV–vis solid spectrometer (2800-Wincom) was used to measure the band gap energies. Textural investigations were carried out with the help of Brunauer–Emmett–Teller (BET) isotherms using (BK300-JWGB) instrumentation. The chemical composition was studied by X-ray photoelectron spectroscopy (XPS) (250Xi-UK). The morphology and microstructure was studied with scanning electron microscopy (SEM, JEOL-6460) and transmission electron microscopy (TEM, JEOL-2100). M-H hysteresis curves were obtained with a magnetometer (PPMS-14LH) at 300 K and 15 T of magnetic field. Electrocatalytic H₂-evolution reactions were studied through a (Solar-IIAG) electrocatalytic system.

2.4. Electrocatalytic H₂-Evolution Reactions. Enclosed circulated instrumentation fitted with a gas analyzer, cooling arrangement, and vacuum system was utilized with 100 mL of aqueous suspension of 0.02 g fabricated NMs within a quartz cell. All the reactions were carried out with a UV-Xe lamp with the wavelength ranging from 200 to 400 nm in the presence of Ar gas. A gas chromatograph with a thermal conductivity detector (TCD) (9790-GC) was utilized for H-evolution
reactions. The chemical reduction method was used for Pt loadings with the synthesized electrocatalytic samples. Initially the samples were treated ultrasonically for 30 min and were cleaned with water and ethanol three times. Finally, the samples were kept in an oven for drying at 80 °C for 12 h.

2.5. Photocatalytic Reactions. A stock solution (0.03 mM) for the MB dye was prepared in a volumetric flask. Dye volume ∼100 mL with 1 g/L of Cu-Fe and Ni-Fe NMs was used for photocatalytic reactions with the exposure of solar light. A UV–visible spectrometer was utilized in order to monitor the concentration decrease for the MB dye by analyzing 5 mL of dye mixture solution at 10 min intervals. Initially, the reaction was placed in the dark for half an hour to investigate the adsorption/desorption phenomenon and afterward it was shifted under solar light for photocatalytic reactions. Concentration decrease and linear kinetics during photocatalysis were calculated by eqs 1–3.

\[
\frac{C_t}{C_0} = \ln \left( \frac{C_t}{C_0} \right) = -kt \tag{2}
\]

Figure 2. (a, b) XRD spectrum, (c) FTIR patterns, (d, e) TGA, (f) zeta potential, (g, h) band gap energies, and (i, j) textural parameters for Cu-Fe and Ni-Fe-NMs.
Where $C_o$ and $C_t$ shows concentration of the dye at initial and specific time during catalysis and $k$ is the constant of reaction.$^{43}$

3. EXPERIMENTAL METHODS AND RESULTS

3.1. Physiochemical Structural Investigation. XRD spectra were analyzed for the phase purity of the NMs as shown in Figure 2(a,b). The peaks at 36.5, 38.9, 48.7, 53.4, 58.3, 62.0, 66.2, 68.9, and 79.7° indicated formation of CuO nanocrystals (PDF-892531, 72-0629, and 65-6186) within Cu-Fe NMs. The Ni cubic phase was revealed by $2\theta = 38.1, 44.5, 51.8, 58.23,$ and 76.3°, and NiO was indicated at 42.12, 62.28, and 74.49, whereas Ni$_2$O$_3$ was displayed with peaks at 31.02 and 65.84 for Ni-containing samples (PDF-040850, 040835, and 47-1049). Strong diffraction peaks with $2\theta$ values of 28.26° (220) and 32.28° (222) denoted the crystalline phase for Fe$_3$O$_4$ nanoparticles (PDF-39-1346 and 89-4319). Scherter’s equation (eq 4) was utilized to calculate the particle size as 26.58 nm for Cu-Fe and 37.45 nm for Ni-Fe NMs.

Figure 3. (a) XPS analysis and (b–g) high resolution peaks for the constituent elements within Cu-Fe and Ni-Fe NMs.

\[
D(\%) = \left( C_o - C_t/C_o \right) \times 100 \quad (3)
\]

Where $C_o$ and $C_t$ shows concentration of the dye at initial and specific time during catalysis and $k$ is the constant of reaction.$^{43}$
In this equation, $d$ represents the average grain size, $k$ shows the shape factor, $\lambda$ is the X-ray wavelength, $\beta$ is FWHM, and $\theta$ is used for Bragg’s angle.

The FTIR spectrum confirmed the molecular structure transformation for the synthesized NMs. The bands at wavenumbers ranging from 3375 to 3475 cm$^{-1}$ were due to OH-stretching and the band at 1643 cm$^{-1}$ indicated OH-bending vibrations. $^{42-45}$ Bands within 1340-1400 cm$^{-1}$ were due to stretching vibration of NO$_3^-$. $^{42}$ C–O, M=O and M–O–M were confirmed due to wavenumbers ~1092 and 599 cm$^{-1}$, respectively, $^{42,43}$ as shown in Figure 2c.

Thermal stability of Cu-Fe and Ni-Fe NMs was investigated by TGA curves as displayed in Figure 2(d,e). Release of

$$d = \frac{k\lambda}{\beta \cos \theta}$$

Table 1. Detailed XPS Spectrum for Cu-Fe and Ni-Fe NMs

| peaks   | Cu-Fe            | Ni-Fe            | area (%)       |
|---------|------------------|------------------|----------------|
| Cu2p    | 944.22/939.10/935.05/931.93 | 879.50/862.96/860.31/856.16/855.03/847.61 | 35.59/124.25/54.94/42.78 |
| Ni2p    | 725.74/712.23    | 724.81/710.94    | 65.32/52.13    |
| Fe2p    | 532.19           | 536.09/533.09/530.52 | 64.52/55.09    |
| O1s     |                  | 100              | 18.99/59.53/21.48 |
| O1s     |                  |                  |                |

Figure 4. (a, b) SEM images, (c, d) TEM micrographs, and (e, f) TEM grain size distribution for Cu-Fe and Ni-Fe NMs.
moisture absorbed and methanol from precursors was calculated to be ∼19.16 and 13.46% for Cu-Fe and Ni-Fe NMs, respectively, at a temperature of 200 °C as the initial weight loss. During the second stage of decomposition, a loss of 1.26% (Cu-Fe) and 24.12% (Ni-Fe) was exhibited till the temperature was reached up to 600 °C. A total weight loss of ∼25.39 and 37.58% was measured for Cu-Fe and Ni-Fe NMs, attributed toward the disintegration of the metal composite framework. The highest loss of 37.58% was found for Ni-Fe NMs displaying a lower stability as compared to Cu-Fe NMs. Cu-Fe NMs have attained the final stability at a temperature of 650 °C, while Ni-Fe was stabilized a bit earlier (i.e., 600 °C).

Figure 2f shows the zeta potential for Cu-Fe and Ni-Fe NMs as a function of pH ranging from 3 to 12. As shown, the pH values for point of zero charge (pHpzc) were found as 7.10 and 3.69 for Cu-Fe and Ni-Fe NMs, respectively. A relatively high value of pHpzc for Cu-Fe-MNFs as compared to Ni-Fe-MNFs may be attributed toward the synthesis technique. The surface below the pHpzc value was revealed as positive, whereas above pHpzc it may be referred as negatively charged. Hence, the conclusion was that the surface of Cu-Fe-MNFs remained below the pHpzc value of Cu-Fe-MNFs as compared to Ni-Fe-MNFs. A single peak at 3.69 for Cu-Fe and Ni-Fe NMs, respectively, at a temperature of 200 °C for Cu-Fe and Ni-Fe NMs, respectively, indicating Fe existence as depicted in Figure 3(d,e). XPS for O is displayed in Figure 3(f,g) for Cu-Fe and Ni-Fe NMs. A single peak at 532.19 eV is shown within Cu-Fe NMs whereas, for Ni-Fe NMs, three peaks were found at 536.09, 533.09, and 530.52 eV. The peak at 530.52 eV may be due to surface O and the peak at 536.09 eV is assigned to adsorbed oxygen molecules.

3.3. SEM, TEM, and Grain Size Distribution. The micrographs for Cu-Fe and Ni-Fe NMs have displayed nonhomogeneous grains with different sizes. Aggregated particles with smooth surfaces were observed for Cu-Fe NMs (Figure 4a,c), whereas dense and short ranged cloudy layers were seen within the microstructure of Ni-Fe NMs (Figure 4b,d). SEM images revealed particles with size ranging from 49–55 nm for Cu-Fe to 46–70 nm for Ni-Fe NMs. Figure 4(e,f) represent TEM grain size distribution with average values ∼40.42 and 56.40 nm for Cu-Fe and Ni-Fe NMs, respectively. The microstructure and porosity for both the samples are well suited for enhanced photocatalytic performance.

3.4. Magnetization Analysis and Electrocatalytic H-Evolution Reactions. Magnetization curves at 25 °C for Cu-Fe and Ni-Fe NMs are shown in Figure 5a. Both the synthesized NMs exhibited super paramagnetic behavior. A saturation magnetization of ∼77.95 and 27.70 emu/g for Cu-Fe and Ni-Fe NMs was obtained at an external magnetic field ∼15,000 Oe. A decrease in magnetization ∼50.25 emu/g was noticed for Ni-Fe NMs as compared to Cu-Fe NMs. Increased surface disorder and particle size can be attributed toward the decreased magnetization. However, easy and quick isolation from reaction solutions can be carried out with a bar magnet due to low (i.e., 27.70 emu/g) magnetic potency for Ni-Fe NMs.

Furthermore, sacrificial reagents such as ethanol and methanol were used during electrocatalytic H-evolution
reactions. H$_2$ production rate $\sim 323.1$ and $621.7$ mmol/g/h was recorded for the Cu-Fe sample, whereas for Ni-Fe the rate was measured $\sim 143.2$ and $86.3$ mmol/g/h, much higher than the pure samples, as shown in Figure 5b. Usually, catalytic efficiency of materials can be greatly influenced due to low crystallinity and the presence of defects, which can activate the charge carrier recombination.$^{32,33}$ The enlarged surface area and smaller particle size can promote separation between photoinduced carriers and more reactive sites are provided, which in turn can enhance the H-evolution rate. Previously various mixed oxides of metals were utilized for the photodegradation of toxic organic dyes only,$^{37-41}$ but this work provides an important pathway for metal oxides to be utilized for water splitting into H$_2$ with easy and mild synthesis.

### 3.5. Photodegradation Performance of Cu-Fe and Ni-Fe NMs

Cu-Fe and Ni-Fe NMs were investigated for photodegradation of MB dye under solar light irradiations. At first, the reaction was carried out in the dark for half an hour and then was brought under solar light for catalytic reactions with continuous stirring. After regular intervals, 5 mL of solution mixture was observed through a UV–vis spectrometer. In the dark, low performance of catalysts was observed, as displayed in Figure 6(a,b). Absorption peaks were recorded at a wavelength of 665 and 664 nm for MB dye with Cu-Fe and Ni-Fe NMs, respectively. Decrease in absorption intensity for MB dye was notified with the passage of time. Concentration decrease, increase in the catalytic reaction, and linear kinetics (determined through eq 2) for MB dye are depicted in Figure 6(c–f). Cu-Fe and Ni-Fe NMs showed 85 and 91% efficiency for MB dye during 50 and 110 min, respectively. Increased irradiation time had a prominent effect upon the degradation percent. Table 2 summarizes the degradation parameters for Cu-Fe and Ni-Fe NMs against MB dye. This work is compared to the previous studies and is summarized in Table 3.

The factors, such as dose of the catalyst, pH, scavengers, reusability, and stability for Cu-Fe and Ni-Fe NMs were also investigated. Each sample (80 mg) was utilized against 0.03 mM MB dye for the said purpose.

### Table 2. Degradation Parameters for Cu-Fe and Ni-Fe NMs against MB Dye

| NMs | rate constant(min$^{-1}$) | correlation constant | catalytic efficiency (%) | time required (min) |
|-----|--------------------------|----------------------|-------------------------|---------------------|
| Cu-Fe | $1.76 \times 10^{-2}$ | 0.99 | 85 | 50 |
| Ni-Fe | $9.83 \times 10^{-3}$ | 0.96 | 91 | 110 |
3.5.1. Catalyst Loading Effect. Active sites can be enhanced by increased catalyst loadings for pollutant detoxification. However, beyond a certain limit, the catalytic yield is decreased even with an increase in the catalyst amount because a fixed number of $^\cdot$OH radicals are usually produced with a given amount of photocatalyst. Also, an increased concentration of MB dye can increase the opacity of the solution, thus preventing the penetration of light and reactivity of $^\cdot$OH radicals. As can be seen from Figure 7a, increased catalyst loading from 20 mg/80 mL to 80 mg/80 mL has increased the reaction rate.

3.5.2. pH Effect. Production of reactive $^\cdot$OH radicals, charge on the catalyst surface, and photodegradation ability is highly dependent on different pH values. Degradation of MB dye was systematically evaluated against both the NMs at pH values of 12, 9, 6, and 3 as shown in Figure 7b. Cu-Fe NMs indicated the highest catalytic activity at pH $\sim$12, whereas Ni-Fe NMs revealed maximum efficiency for MB dye at pH $\sim$9.

3.5.3. Scavenging Effect. The scavenging effect was studied using H$_2$O$_2$ and ethanol. The catalytic efficiency rate for MB dye with the ethanol scavenger was prominent for Cu-Fe NMs, whereas for Ni-Fe NMs, the H$_2$O$_2$ scavenger was found to be better as depicted in Figure 7c.

3.5.4. Reusability and Stability Check. Improved catalytic performance due to separation of photogenerated carriers and hence the reusability was investigated by analyzing the degradation activity for Cu-Fe and Ni-Fe NMs over several cycles. Fresh solution was used for degradation and then the catalyst was collected during each cycle. The collected catalyst was washed five times with distilled water, dried, and reused for a new degradation activity. The degradation efficiency for

### Table 3. Comparison between this Work and Previous Studies

| Sample | Pollutant | Time | Deg. (%) | Studies |
|--------|-----------|------|----------|---------|
| SF     | MB        | 80 min | 71%      | 44      |
| AF     | MB        | 80 min | 83%      | 44      |
| SC     | MB        | 180 min| 78%      | 45      |
| CC     | MB        | 180 min| 67%      | 45      |
| CS     | MO        | 180 min| 80%      | 45      |
| SC     | MO        | 180 min| 50%      | 45      |
| CC     | MO        | 180 min| 69%      | 45      |
| CF     | RhB       | 50 min | 77%      | 46      |
| AF     | MO        | 140 min| 45%      | 47      |
| FZF    | MO        | 60 min | 52%      | 48      |
| AN     | MO        | 120 min| 75%      | 49      |
| CF     | MB        | 50 min | 85%      | this work |
| NF     | MB        | 110 min| 91%      | this work |

![Figure 7](https://dx.doi.org/10.1021/acsomega.0c01259)  
Figure 7. Various factors such as (a) catalyst amount, (b) pH, (c) scavengers, (d) reusability, (e, f) XRD, and (g) FTIR for Cu-Fe and Ni-Fe NMs after photodegradation reactions.
Cu-Fe NMs was decreased up to 63% after the third cycle, whereas for Ni-Fe NMs, it was decreased up to 77%, as shown in Figure 7d. The loss of catalyst could be attributed toward the decreased percentage of degradation. However, NMs could be used effectively almost up to three cycles, with satisfactory results.

Additionally, stability for the synthesized NMs was also confirmed through FTIR and XRD spectra after degradation reactions. As displayed in Figure 7(e–g), no change in the XRD and FTIR patterns revealed the greater stability and suitability of the as-synthesized NMs as an efficient photocatalyst for many cycles. Table 4 summarizes the recyclability results for Cu-Fe and Ni-Fe NMs.

Table 4. Reusability Results for Cu-Fe and Ni-Fe NMs

| no of cycles | Cu-Fe NMs | Ni-Fe NMs |
|--------------|-----------|-----------|
| R1           | 78.95     | 90.75     |
| R2           | 69.68     | 88.96     |
| R3           | 63.48     | 77.23     |

3.6. In Situ Analysis for Active Species Determination and Degradation Mechanism of MB Dye. An in situ technique was followed for the determination of the active species, which were considered as holes (h⁺), electrons (e⁻), hydroxyl radicals (OH⁻), and superoxide radicals (O₂⁻). Ethanol and hydrogen peroxide solutions of 0.1 mL were mixed as scavengers. The superoxide radicals and holes were captured through the change in concentration of dyes. Usually, photogenerated holes and the powerful oxidant OH radicals have an important role during the photocatalytic activity. The toxic dyes are oxidized by these holes directly, or by the combination of holes and OH⁻ ions. Schematic and mathematical details for MB dye degradation with Cu-Fe and Ni-Fe NMs are shown in Figure 8 and eqs 5–17, respectively.

\[
h^+(\text{energy}) + \text{NMs} \rightarrow \text{NMs}(e^- + h^+) \quad (5)
\]

\[
dye + h^+ \rightarrow \text{intermediate products} \rightarrow \text{H}_2\text{O} + \text{CO}_2 \quad (6)
\]

\[
h^+ + \text{H}_2\text{O} \rightarrow \text{H}^+ + \text{OH} \quad (7)
\]

\[
h^+ + \text{OH}^- \rightarrow \text{OH} \quad (8)
\]

\[
dye + \text{OH} \rightarrow \text{intermediate products} \rightarrow \text{H}_2\text{O} + \text{CO}_2 \quad (9)
\]

The dissolved oxygen is combined with electrons in the following way.

\[
e^- + \text{O}_2(\text{aq}) \rightarrow \text{O}_2^- \quad (10)
\]

dye + O₂⁻ → intermediate products → H₂O + CO₂

\[
\text{H}_2\text{O} + \text{O}_2^- \rightarrow \text{OOH} + \text{OH}^- \quad (12)
\]

\[
\text{H}^+ + \text{OOH} \rightarrow \text{OH}^- + \text{OH} \quad (13)
\]

\[
h^+ + \text{OH}^- \rightarrow \text{OH} \quad (14)
\]

Reactive species of oxygen are generated due to electrons. Anion oxide radicals are usually obtained during the catalytic process. Sometimes H₂O₂ production can also decompose OH⁺ in several ways:

\[
e^- + \text{O}_2(\text{aq}) \rightarrow \text{O}_2^- \quad (15)
\]

\[
\text{H}^+ + \text{O}_2^- \rightarrow \text{HO}_2^- \quad (16)
\]

\[
\text{H}^+ + e^- + \text{HO}_2^- \rightarrow \text{H}_2\text{O}_2 \quad (17)
\]

4. CONCLUSIONS

This research work involved the fabrication and multifunctional applications of Cu-Fe and Ni-Fe NMs. Particles with a size range below 70 nm were displayed by XRD, SEM, and TEM. The presence of constituent elements within fabricated samples was indicated by XPS analysis. Low band gaps ~1.79 and 1.48 eV and surface areas ~4.48 and 36.52 m²/g were calculated for Cu-Fe and Ni-Fe NMs, respectively. Hysteresis loops confirmed superparamagnetism with Ms values ~77.95 and 27.70 emu/g for Cu-Fe and Ni-Fe NMs, respectively. The NMs were revealed as much better for H-evolution in the presence of sacrificial agents such as methanol and ethanol as compared to pure samples. MB dye was degraded up to 85% with Cu-Fe and 91% with Ni-Fe NMs. The in-depth details about the properties and degradation ability indicated that Ni-Fe NPs are better as compared to Cu-Fe NPs. Furthermore, in situ analysis of the degradation mechanism and various applications can lead toward cost-effective and improved applications for the as-synthesized NMs at an industrial level.

AUTHOR INFORMATION

Corresponding Authors

Zarbhad Shah – Department of Chemistry, Bacha Khan University Charsadda, Charsadda 24420, Khyber Pakhtunkhwa, Pakistan; orcid.org/0000-0003-4805-3091; Email: zshej@bkuc.edu.pk, zshej@hotmail.com

Hongli Suo – The Key Laboratory of Advanced Functional Materials, Ministry of Education, Beijing University of Technology, Beijing 100124, China; Email: honglisuo@bjut.edu.cn

Authors

Kausar Shaheen – The Key Laboratory of Advanced Functional Materials, Ministry of Education, Beijing University of Technology, Beijing 100124, China; Department of Physics, Jinnah College for Women, University of Peshawar, Peshawar 25120, Khyber Pakhtunkhwa, Pakistan

Ambreen Asad – Department of Chemistry, Bacha Khan University Charsadda, Charsadda 24420, Khyber Pakhtunkhwa, Pakistan

Tofail Arshad – Department of Chemistry, Bacha Khan University Charsadda, Charsadda 24420, Khyber Pakhtunkhwa, Pakistan

Sher Bahadar Khan – Center of Excellence for Advanced Materials Research and Chemistry Department, Faculty of Materials Research and Chemistry Department, Faculty of Chemistry, Bacha Khan University Charsadda, Charsadda 24420, Khyber Pakhtunkhwa, Pakistan; Email: zshej@bkuc.edu.pk, zshej@hotmail.com

https://dx.doi.org/10.1021/acsomega.0c01259
ACS Omega 2020, 5, 15992–16002

Figure 8. Schematic representation of catalytic reactions with Cu-Fe and Ni-Fe NMs.
Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.0c01259

Notes
The authors declare no competing financial interest.

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