



Fe/Cu Bimetallic Oxide as a Multifunctional Adsorbent and Fenton Assisted Catalyst for Efficient Removal of Oil and Methylene Blue

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ABSTRACT

Current water treatment technologies often face limitations such as the generation of secondary chemical by-products and high energy demands, highlighting the need for more efficient and sustainable alternatives. In this context, our study presents a multifunctional water purification system based on Fe/Cu bimetallic oxide nanoparticles designed for the effective treatment of oily wastewater and the removal of persistent dyes such as methylene blue (MB). The Fe/Cu bimetallic oxide catalyst was synthesized via a hydrothermal route, and its performance was evaluated through both adsorption and Fenton-based degradation mechanisms. Structural and chemical analyses were carried out using X-ray Diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, and UV–Vis spectroscopy. The synthesized catalyst exhibited high removal efficiencies for both pollutants, with the 5:2 Fe: Cu composition outperforming the 5:1 and 5:3 ratios. This optimal ratio achieved 95.4% removal of methylene blue at pH 10 using 3 g/L catalyst dosage within 5 min reaction time. Additionally, the catalyst demonstrated nearly 75% reduction in oil droplet size for 500 ppm oil emulsion treatment. The study demonstrates that Fe/Cu bimetallic oxide overcomes the narrow pH limitation of the conventional Fenton system through Cu mediated redox synergy. These results highlight the strong catalytic activity and multifunctional capability of the Fe/Cu bimetallic oxide. Owing to its efficiency, rapid action, and adaptability, this approach holds significant promise for wastewater applications across industries such as oil refining, petroleum processing, textiles, and paper manufacturing.

Keywords: Adsorption; Bimetallic Oxide; Fenton Reaction; Oil-water emulsion.

1. INTRODUCTION

Oily wastewater contains a complex mixture of dissolved and suspended organic and inorganic pollutants and is recognized as one of the most hazardous industrial effluents. Oil-in-water and water-in-oil emulsions—commonly generated in the petroleum and metalworking industries—lead to increased transportation costs, corrosion of equipment, and environmental pollution. Oil droplets are typically categorized based on size: free oil (>150 μm), dispersed oil (20–150 μm), emulsified oil (<20 μm), and dissolved hydrocarbons (<5 μm). While larger droplets can be removed by conventional physical processes such as gravity separation and dissolved air flotation, emulsified and dissolved oils require advanced treatment technologies (Adetunji *et al.* 2021).

Textile wastewater poses another major environmental threat due to the discharge of large quantities of synthetic dyes. Methylene blue or its abbreviation MB, a widely used cationic dye, is known for its toxicity and carcinogenicity, and even trace amounts can inhibit photosynthesis in aquatic systems. Although several treatment methods exist—including

coagulation, filtration, biodegradation, and biosorption, many fail to fully mineralize dyes due to their stability and solubility (Khan *et al.* 2023; Roy and Saha, 2021; Al-Tohamy *et al.* 2022).

Metal oxide nanoparticles have recently gained considerable attention due to their large surface area, strong adsorption capacity, chemical tunability, and catalytic properties (Farahani *et al.* 2022; Khan *et al.* 2016). Iron oxide nanoparticles (e.g., magnetite, maghemite, hematite) are particularly notable for their high surface area, porosity, and superparamagnetic behaviour. However, nanosized Fe oxides tend to agglomerate, reducing active surface area; this is typically mitigated by surface modification using surfactants or polymers (Zhou *et al.* 2020).

Fenton reactions, which rely on the catalytic decomposition of H_2O_2 to produce hydroxyl radicals, are widely used for degrading organic pollutants. Conventional Fe-based Fenton processes, however, function efficiently only under acidic conditions. Copper-based catalysts can operate over a broader pH range, but the rapid oxidation of Cu^+ to Cu^{2+} limits their standalone

use. Combining Fe and Cu in a bimetallic oxide system overcomes these limitations by exploiting synergistic effects that enhance electron transfer, radical production, and catalytic stability (Qin *et al.* 2011; Ghasemi *et al.* 2020; Zin *et al.* 2013; Elgarahy *et al.* 2021; Karpuz *et al.* 2009; Kumar, 2019; Legaria *et al.* 2015; Shahwan *et al.* 2021).

Although several studies have investigated Fe-based and Cu-based catalysts for wastewater treatment, limited studies have focused on multifunctional Fe/Cu bimetallic oxide systems capable of simultaneously treating oily wastewater and dye pollutants through combined adsorption and Fenton-like degradation mechanisms. Furthermore, optimization of Fe/Cu ratios for rapid degradation efficiency under broader pH conditions remains insufficiently explored. Therefore, the development of an efficient Fe/Cu hybrid oxide catalyst with enhanced catalytic activity, rapid degradation kinetics, and multifunctional pollutant removal capability is of significant research interest.

Thus, Fe–Cu hybrid oxides present a promising platform for dual-purpose treatment of dye-containing and oily wastewater. This work aims to synthesize Fe–Cu bimetallic oxide nanoparticles, optimize their composition, and evaluate their efficacy in MB degradation and oil emulsion breakdown through adsorption and Fenton-like mechanisms.

2. MATERIALS AND METHODS

2.1 Materials

The materials used for the experiments include MB powder (dye content > 85%), which was purchased from Sigma-Aldrich, India. 1 mM H₂O₂ (30%), N-N-Dimethylformamide (DMF, 99.8% purity), ferric chloride hexahydrate (FeCl₃·6H₂O), copper nitrate trihydrate (Cu (NO₃)₂ · 3H₂O), methanol was purchased from Nice Chemicals (P), Ltd, India, and sunflower oil.

2.2 Preparation of Synthetic Wastewater Containing MB and Oil Emulsion

Preparation of oil emulsions with varying concentrations, specifically 250, 500, and 750 parts per million (ppm), was done by subjecting sunflower oil, as a representative oil, to magnetic stirring in a 250 mL volume of distilled water for a duration of 1 hour. Subsequently, the mixture underwent ultrasonication for an additional 30 mins, ensuring well-dispersed oil emulsions of size 30µm. Characterization studies for oil emulsions using Chemical oxygen demand and turbidity analysis for calibration curves were drawn. Methylene blue solution was bulk prepared at 1000 ppm, whereupon dilution to different concentrations (ppm) was made, and calibration curves were drawn using a U-Vis spectrophotometer (Nguyen *et al.* 2020).

2.3 Synthesis of Fe-Cu Bimetallic Oxide Catalyst

The method involves hydrothermal synthesis of a bimetallic oxide catalyst using 1.461g ferric chloride hexahydrate, 0.831 g terephthalic acid, and different concentrations of copper nitrate trihydrate (0.302, 0.604, and 0.906 g) in 30 mL of DMF, thus synthesizing bimetallic oxide catalysts of ratios such as 5:1, 5:2, and 5:3 g wt of ferric chloride hexahydrate and copper nitrate trihydrate, respectively. The mixture was homogenized by magnetic stirring for 1 hour at room temperature and then immediately transferred into a 100 mL autoclave lined with a Teflon liner for 120°C for 24 h. The resulting product was solvent-washed using methanol 3 times and dried in a hot air oven at 60°C (Khan *et al.* 2016; Zin *et al.* 2013).

Terephthalic acid was used as an organic coordinating ligand during hydrothermal synthesis to regulate nucleation and growth of the Fe/Cu oxide framework. The presence of terephthalic acid improves particle dispersion, prevents excessive agglomeration, and enhances surface functionalization of the synthesized catalyst.

The overall recovery yield of the synthesized catalyst after multiple syntheses, washing, and drying was approximately 60 -75% and repeated synthesis showed consistent catalyst formation under the selected conditions, depending on the Fe/Cu composition ratio.

2.4 Characterization Studies of Fe-Cu Bimetallic Oxide

The characterization of the bimetallic oxides was done using X-ray diffraction to check for their crystal structure and orientation. The chemical composition was analyzed using the Fourier transform infrared spectrometer (PerkinElmer Spectrum IR) in the range 4000- 600 cm⁻¹. A Shimadzu UV Spectrophotometer (UV1800) was used to analyze the UV spectrum at a wavelength of 664 nm for MB after calibration. A trinocular microscope at 400x magnification was used to analyze oil emulsions and the changes to the oil emulsion after treatment.

2.5 Removal Efficiency/Degradation Studies Experiments

2.5.1 Methylene Blue Removal Study

For MB degradation studies, the 50-ppm stock solution of MB was taken in an Erlenmeyer flask and kept in the orbital shaker at 80 rpm for 1 h at room temperature, into which the bimetal catalyst was introduced. Two mechanisms, namely adsorption and the Fenton reaction, were tested for removal efficiency, where for the Fenton reaction, an additional 1 mL of H₂O₂ was added. The absorbance of the solution was

subsequently measured using a UV-Vis Spectrophotometer after filtering out the catalyst, and the percentage of degradation was computed using Equation (1). The following formula was used to determine the degradation percentage (Mohamed *et al.* 2021):

$$D\% = \frac{C_0 - C_f}{C_0} \quad \dots (1)$$

where, C_0 is the initial concentration in mg/L, C_f is the final concentration in mg/L of the solution.

During the study, the optimum bimetallic concentration of Fe-Cu oxide was determined by varying ratios of 5:1, 5:2, 5:3 (w/w). The catalyst dosage was determined by varying dosages from 1- 3 g/L. The optimum pH was studied by testing the same with altered sample pH of 3, 7, and 10 (Chen *et al.* 2022; Luong *et al.* 2022; Kobayashi *et al.* 2014).

2.5.2 Oil Emulsion Removal Studies

For oil emulsion removal studies 250,500 and 750 ppm stock solution of oil emulsion were taken in an Erlenmeyer flask and kept in the orbital shaker at 100 rpm for 1 h at room temperature. Mechanisms-adsorption and Fenton reaction were performed at different Fe-Cu oxide ratios and the efficiency were analyzed by the degrees of size reduction in the oil emulsion sample after treatment using a trinocular microscope at 400x magnification (Nieto *et al.* 2011; Raju *et al.* 2024).

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

Figure 1 displays the XRD of synthesized Fe oxide-Cu bimetallic oxide at different ratios of 5:1,5:2, 5:3. Odd peaks at $2\theta=24.8^\circ$, 30° at plane (200), (220) indicated the presence of iron oxide (Fe_2O_3) while a peak at $2\theta=32.7^\circ$ at plane (111) indicated the presence of copper oxide (Cu_2O), rather than the presence of Fe-Cu bimetals (Ulucan-Altuntas *et al.* 2019; Wu *et al.* 2021). This observation was attributed to the increased tendency of oxidation resulting from the drying process in a hot air oven. Furthermore, the consistent decrease in the peak intensity of iron oxide with an increased copper ratio is due to the addition of copper. The high sharp peaks for iron, as opposed to copper, can be attributed to the rapid crystallization of iron at high procedure temperatures (Qin *et al.* 2011). The absence of Fe-Cu alloy peaks further confirms that the two metals exist as discrete oxide phases (Fe_2O_3 and Cu_2O) rather than forming a solid solution or intermetallic compound (Karthikeyan *et al.* 2016). Despite the absence, the catalytic synergy therefore arises from interfacial contact between Fe_2O_3 and Cu_2O crystallites rather than from a mixed-lattice electronic effect where electron transfer between the $\text{Fe}^{3+}/\text{Fe}^{2+}$ and $\text{Cu}^{2+}/\text{Cu}^+$ redox couples enhance catalytic

activity. Similar phase-separated Fe-Cu oxide systems exhibiting interfacial redox synergy have been reported in previous studies (Nguyen *et al.* 2020; Hussain *et al.* 2021; Manickam *et al.* 2020).

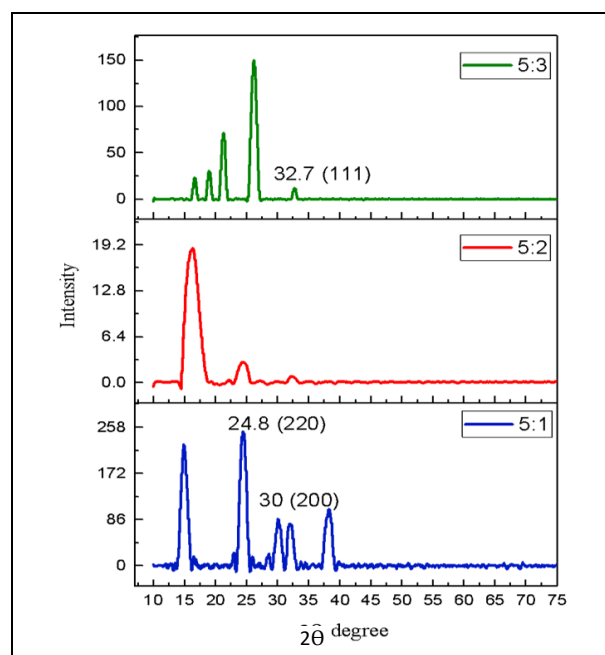


Fig 1: XRD peak analysis of synthesized hybrid iron -copper oxides of ratios 5:1,5:2,5:3

3.2 FT-IR Analysis

As shown in Figure 2 the FT-IR spectrum of the Fe-Cu oxides material reveals distinct absorption peaks indicative of its molecular composition and functional groups. Notably, characteristic peaks as seen in the figure were observed for both Fe_2O_3 and Cu_2O within the ranges of $430\text{-}537\text{ cm}^{-1}$ and $744\text{-}600\text{ cm}^{-1}$, respectively, suggesting the presence of these metallic constituents in the filler. Additionally, the spectrum exhibited peaks corresponding to specific functional groups, where a peak in the range $1330\text{-}1430\text{ cm}^{-1}$ was indicative of OH bending vibrations, typically associated with carboxylic acids, and a broad peak in the range $3300\text{-}3400\text{ cm}^{-1}$ representative of OH stretching vibrations, commonly found in alcohols. These findings provide valuable insights into the molecular structure and composition of the hybrid oxide, facilitating its further characterization (Shahwan *et al.* 2021; Nguyen *et al.* 2020; Chen *et al.* 2022).

3.3 Removal Efficiency Studies of MB

3.3.1 Optimum Fe Oxide-Cu Ratio

Figure 3 illustrates the comparative efficacy of Fenton-driven and adsorption-based MB removal using a 1g/wt loading of catalyst with varying bimetallic oxide ratios. It was observed that the 5:1 ratio exhibited

superior MB removal via Fenton, achieving a 94.5% removal rate attributed to a higher Fe ratio, thus leading to more Reactive Oxygen species (ROS) generation (Raju *et al.* 2024; Nguyen *et al.* 2020). Conversely, the 5:3 ratio catalyst demonstrated enhanced adsorption, with a 96% removal rate linked to an increase in surface functional groups such as hydroxyl (–OH) and carboxyl (–COOH) groups and masking of Fe oxide due to a higher Cu ratio. Notably, catalysts with a 5:2 ratio demonstrated exceptional removal rates for both mechanisms, achieving 97.8% and 96.5% removal rates, respectively, thereby establishing the 5:2 bimetal ratio as optimal (Nguyen *et al.* 2020).

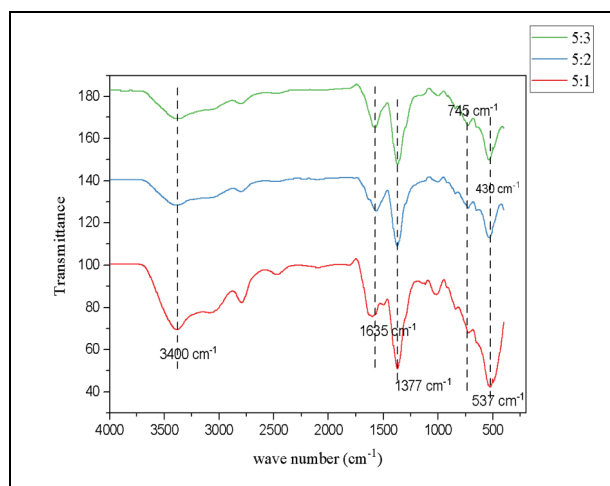


Fig 2: FT-IR peak analysis of synthesized hybrid iron -copper oxides of ratios 5:1,5:2,5:3

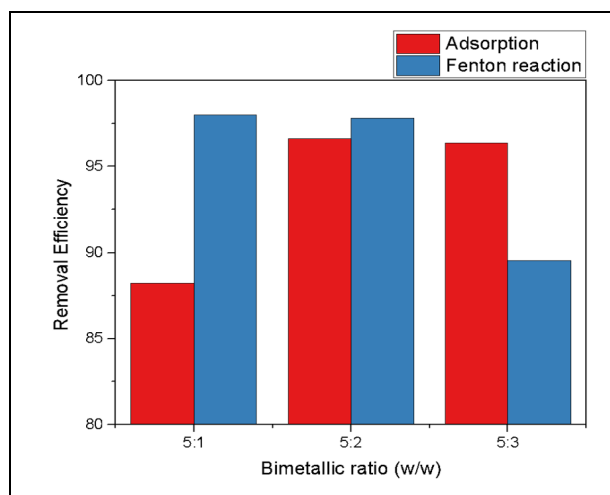


Fig 3: Removal efficiency of Fe-Cu bimetallic oxide at various ratios (5:1,5:2,5:3)

3.3.2 Time Study Performed at 3g/L Catalyst Dosage at 50 ppm

Figure 4 illustrates a degradation curve that shows the reaction time for adsorption and the Fenton reaction for 3g/L catalyst dosage of a 5:2 ratio catalyst. It

is observed that a sharp curve indicating more than 90% removal of MB occurs for both mechanisms during the initial 5 mins, followed by a gradual increase, then attaining a steady state. This clearly shows that the optimal time for degradation of MB occurs at 5 mins of the 60 mins study. Though removal by both mechanisms was comparable, Fenton showed higher removal for the same reaction time. This was due to the reduction potential difference of 0.777 volts between Cu and Fe, which enhances electron emission from the Fe surface, which helps in the generation of accelerated electron emission, facilitates electron transfer to reactants, such as H_2O_2 , promoting the generation of hydroxyl radicals ($\bullet OH$) crucial for oxidative degradation processes (Ulucan-Altuntas *et al.* 2019; Wu *et al.* 2021).

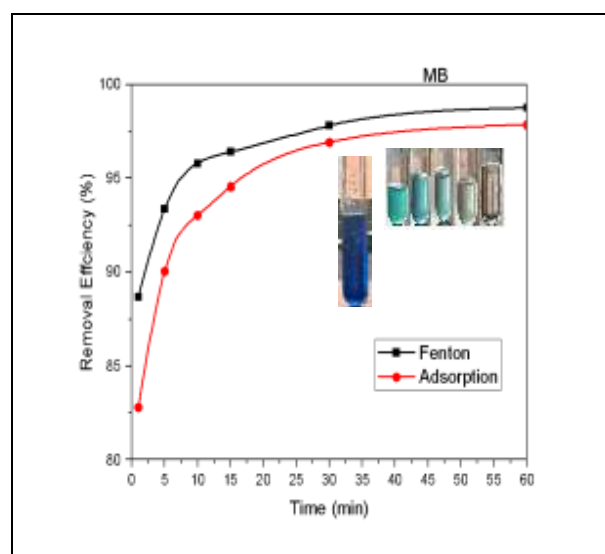


Fig 4: Comparative kinetics of MB removal through Fenton-assisted degradation and adsorption mechanisms

3.3.3 Effect of Catalyst Dosage

For this study, as shown in Figure 5, the removal efficiency (%) of a 50 mg L^{-1} MB solution was evaluated at different catalyst dosages (1, 2, and 3 g L^{-1}) using the optimized Fe/Cu ratio of 5:2 (w/w). Adsorption experiments were conducted using the catalyst alone, while Fenton degradation experiments were performed in the presence of $1 \text{ mL H}_2\text{O}_2$. All experiments were carried out at room temperature with orbital shaking (80 rpm) for 60 min. The bars represent the percentage removal of MB calculated from the change in concentration before and after treatment. The results demonstrate that increasing catalyst dosage enhanced MB removal for both mechanisms due to the increased availability of active surface sites and catalytic centers. The highest removal efficiencies were achieved at a catalyst dosage of 3 g L^{-1} , reaching 98.7% for the Fenton process and 96.5% for adsorption. Fenton denotes the heterogeneous Fenton-like oxidation process involving Fe/Cu catalyst and H_2O_2 , while Adsorption denotes pollutant removal by surface

adsorption in the absence of H_2O_2 . (Pavithran *et al.* 2024).

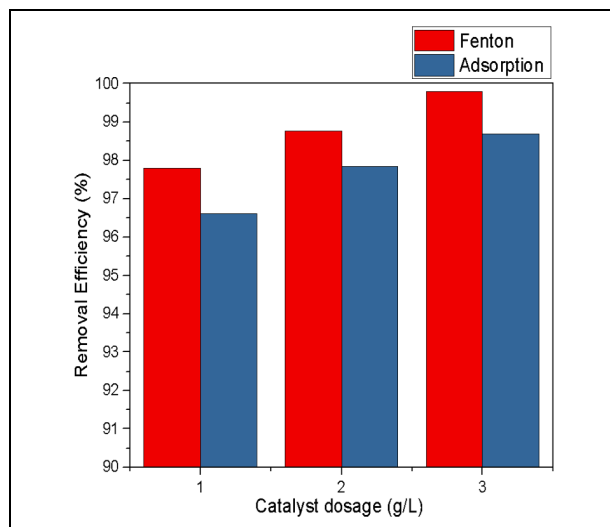


Fig 5. Effect of catalyst dosage on MB removal efficiency using Fe/Cu bimetallic oxide catalyst through adsorption and Fenton-assisted degradation mechanisms

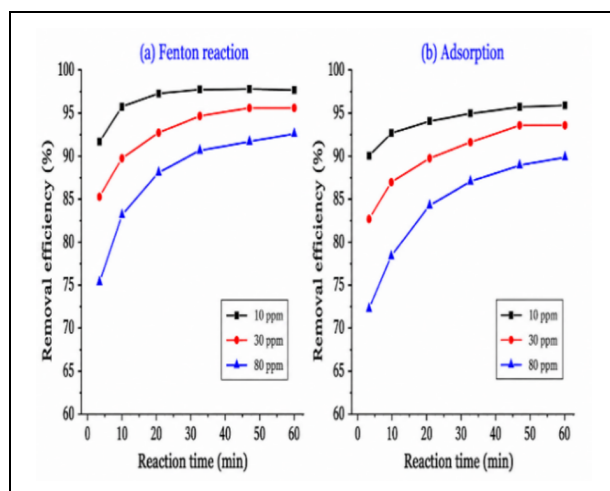


Fig. 6: Effect of initial concentration of the pollutant MB on removal of MB using Fe/Cu bimetallic oxide catalyst through (a) Fenton reaction and (b) Adsorption

3.3.4 Effect of Initial Concentration of Pollutant

The effect of initial concentration on the removal of MB concerning time is tabulated in Figure 6. It is evident that as the concentration of the pollutants increases, the effectiveness of pollutant destruction diminishes. The efficiency of MB degradation in 5 min of the reaction fell from 97 to 93% as the MB concentration increased from 10 to 80 ppm for Fenton and likewise, for adsorption, it fell from 96 to 90 % from 10 to 80 ppm. This is because raising the concentration of pollutants leads to a relatively lower concentration of OH than for Fenton (Nguyen *et al.* 2020).

3.3.5 Effect of pH

pH is one important parameter with respect to the degradation of pollutants. Here, the study was conducted in a pH range of 3 to 10, and the results are depicted in Figure 7. The degradation of MB by Fenton was seen to attain its maximum efficiency at a pH of 10. At alkaline pH, hydroxide ions (OH^-) can serve as efficient scavengers for Reactive Oxygen species ROS, such as $\cdot\text{OH}$ radicals, promoting their sustained activity in degrading MB molecules (Nguyen *et al.* 2020) due to copper extending the pH range of the reaction. However, for adsorption, the removal shows a slight decrease at neutral pH due to deactivation of Fe active sites, which function in acidic pH (Pavithran *et al.* 2024).

The optimal performance at pH 10 deviates from classical Fenton chemistry (optimal pH 2.8-3.5) but this is consistent with Cu-containing Fenton-like systems (Zhou *et al.* 2020; Hussain *et al.* 2021). At alkaline pH, Cu^+ species remain more stable against rapid oxidation to Cu^{2+} than Fe^{2+} does, sustaining the $\text{Cu}^+/\text{Cu}^{2+}$ redox cycle that drives H_2O_2 activation (Nguyen *et al.* 2020; Qin *et al.* 2018). Additionally, at high pH, Fe^{3+} precipitates as $\text{Fe}(\text{OH})_3$, which can itself catalyze heterogeneous Fenton-like reactions at the oxide-water interface (Nidheesh, 2015). The Cu_2O phase (identified by XRD) provides active Cu^+ sites that resist hydroxide passivation, and the OH^- ions contribute to the formation of surface-bound hydroxyl radicals ($\equiv\text{Fe/Cu-OH}\cdot$) that retain high oxidative activity at the catalyst surface (Zhou *et al.* 2020).

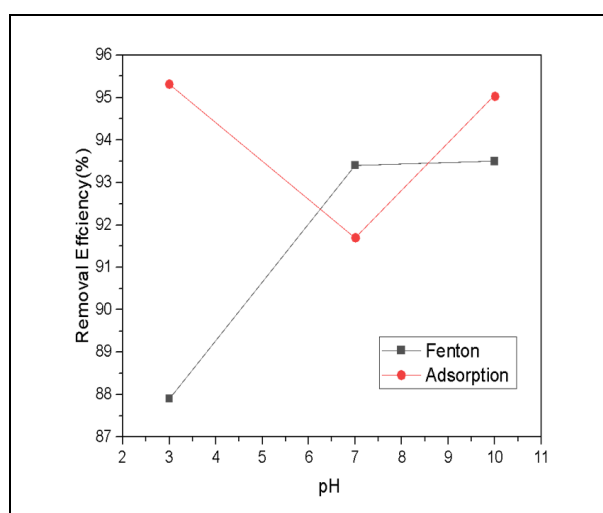


Fig 7: Effects of pH on removal efficiency of MB using Fe/Cu bimetallic oxide catalyst through fenton reaction and adsorption

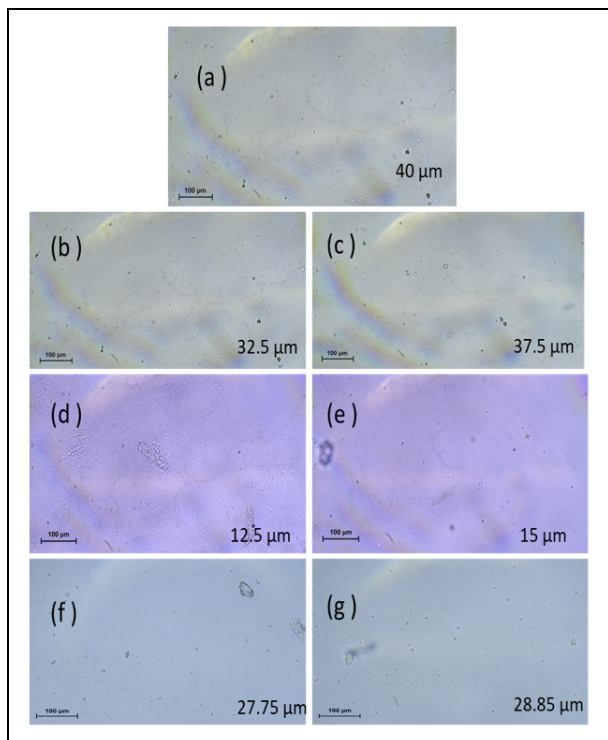


Fig 8: Removal efficiency of Fe-Cu oxides on 500 ppm oil-water emulsion (a) before (40 μm) after of (b) 5:1 ratio catalyst after Fenton degradation (c) 5:1 ratio catalyst after adsorption (d) 5:2 ratio catalyst after fenton degradation (e) 5:2 ratio catalyst after adsorption (f) 5:3 ratio catalyst after Fenton degradation and (g) 5:3 ratio catalyst after adsorption

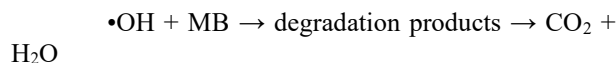
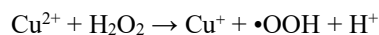
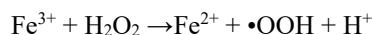
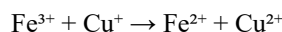
3.4 Oil Emulsion Studies

Through our study on both Fenton and adsorption mechanisms, as shown in Figure 8, significant reduction of oil emulsion size was seen with a notable reduction of over 75% from the original 30 μm of 500 ppm. Particularly, the utilization of a 5:2 bimetallic oxide composition demonstrated enhanced size reduction, attributable to synergistic effects between the metals involved. The superior performance of the Fenton mechanism over adsorption was observed, with Fenton exhibiting greater effectiveness in reducing emulsion size. This outcome can be credited to the oxidative nature of the Fenton reaction, which facilitates more efficient breakdown of oil droplets (Raju *et al.* 2024). Notably, the application of the 5:2 bimetallic oxide composition resulted in emulsion sizes of 12.5 and 15 μm .

3.5 Proposed Mechanism of Degradation

The degradation mechanism of methylene blue MB by the Fe/Cu bimetallic oxide catalyst in the presence of H_2O_2 is proposed based on the synergistic redox cycling between Fe and Cu species. In the heterogeneous Fenton-like system, Fe^{2+} and Cu^+ active sites catalytically decompose H_2O_2 to generate highly reactive hydroxyl radicals ($\bullet\text{OH}$), which are responsible for oxidative degradation of organic pollutants.

The primary reactions involved are as follows:



The spontaneous electron transfer between Cu^+ and Fe^{3+} depicted in equation 3 promotes continuous regeneration of Fe^{2+} active sites, thereby sustaining the catalytic cycle and enhancing hydroxyl radical production. Simultaneously, adsorption of methylene blue molecules onto the catalyst surface increases pollutant proximity to reactive oxygen species, further improving degradation efficiency. The combined adsorption and catalytic oxidation mechanisms contribute to the excellent multifunctional performance of the Fe/Cu bimetallic oxide catalyst for wastewater treatment applications also sustaining the catalytic cycle and explaining the superior performance of the bimetallic system over single-metal oxides. (Luong *et al.* 2022; Pavithran *et al.* 2024).

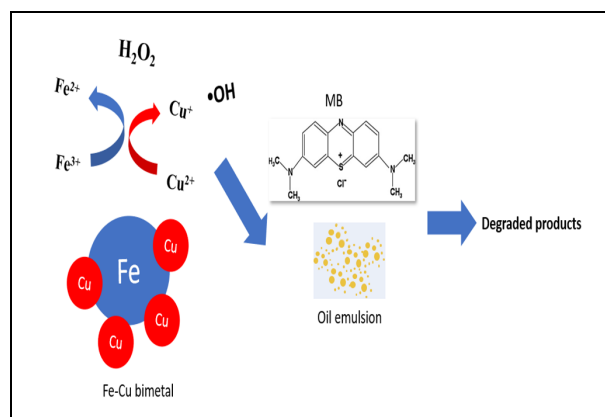


Fig. 9: Proposed mechanism of pollutant removal

4. CONCLUSION

Fe/Cu bimetallic oxide synthesized through the hydrothermal method demonstrated excellent multifunctional performance for simultaneous removal of methylene blue dye and oily wastewater. Among the investigated compositions, the 5:2 Fe/Cu ratio exhibited the highest catalytic and adsorption efficiency due to enhanced synergistic interaction between Fe and Cu active sites. Under optimized conditions of 3 g/L catalyst dosage and 5 min reaction time, the catalyst achieved 95.4% methylene blue degradation and nearly 75% reduction in oil droplet size.

The catalyst exhibited rapid degradation kinetics and maintained strong catalytic activity over a broader pH range, particularly under alkaline conditions, highlighting the beneficial role of Cu species in extending Fenton-like activity. The enhanced degradation efficiency was attributed to continuous redox cycling between $\text{Fe}^{2+}/\text{Fe}^{3+}$ and $\text{Cu}^+/\text{Cu}^{2+}$ species, which promoted efficient hydroxyl radical generation.

Overall, the developed Fe/Cu bimetallic oxide catalyst demonstrates strong potential as an environmentally friendly and highly efficient material for industrial wastewater remediation. Future work will focus on catalyst reusability, real industrial wastewater treatment, long-term stability studies, and large-scale process implementation.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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