Malaysian Journal of Microscopy 2026 22(1) 150-160

MALAYSIAN JOURNAL OF MICROSCOPY

Journal homepage: https://malaysianjournalofmicroscopy.org

RESEARCH ARTICLE

COPPER LOADED MAGNETIC HYDROCHAR DERIVED FROM COCONUT HUSK FOR CATALYTIC REDUCTION OF 4-NITROPHENOL

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Abstract. 4-nitrophenol (4-NP) is a hazardous pollutant that poses significant risks to human health and the environment. One promising approach to address this contaminant is the use of hydrochar derived from biomass sources. In this study, hydrochar was produced through hydrothermal carbonisation of coconut husks. The synthesised hydrochar was then integrated with magnetite and further loaded with Cu to form Cu magnetic hydrochar (Cu-MHC). The properties and catalytic performance of the Cu-MHC were examined by synthesising with varying copper loadings (1 %, 5 %, and 10 %). Various factors influencing the degradation of 4-NP into 4-aminophenol (4-AP) using sodium borohydride ($NaBH_4$) as the reducing agent were investigated, including the effects of copper loading, catalyst dosage, and the initial concentrations of 4-NP. The catalytic performance was compared across different copper loadings against that without copper (MHC) and showed that MHC achieved 13.25 %, while 1 % Cu-MHC reached 15.38 % in 3 minutes. A significant improvement was observed at 5 % Cu-MHC with 95.92 %, followed by 10 % Cu-MHC at 92.62 % in 3 minutes. From the results, 5 % Cu-MHC is the most effective catalyst for the reduction of 4-NP and was further characterized using x-ray diffraction (XRD), field emission scanning electron microscope with energy dispersive x-ray spectroscopy (FESEM-EDX) and transmission electron microscope (TEM). The results showed that 30 mg of 5 % Cu-MHC could effectively degrade 0.12 mM 4-NP (98 %) within 3 minutes. TEM analysis revealed predominantly spherical nanoparticles with diameters between 15 and 20 nm, highly distributed across the hydrochar. FESEM-EDX mapping confirmed the presence of copper and describe well distribution of copper on the surface of magnetic hydrochar nanocomposite. This is further supported by the absence of Cu peaks in the XRD spectra, which may indicate a high dispersion of copper species on the magnetite hydrochar, which contributes to the efficient catalytic reduction of 4-NP.

Keywords: Magnetic hydrochar, catalytic reduction, 4-nitrophenol, hydrothermal carbonisation.

Article Info

Received 6 January 2026
Accepted 7 May 2026
Published 8 June 2026

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ISSN: 1823-7010, eISSN: 2600-7444

1. INTRODUCTION

4-nitrophenol (4-NP) is a toxic pollutant commonly found in industrial waste from the production of dyes, pharmaceuticals, paper, and pesticides. It is harmful to aquatic organisms and potentially carcinogenic to humans, making its removal from wastewater essential [1]. Various treatment methods, such as adsorption, microbial degradation, photocatalysis, and electrochemical processes, have been studied. However, these methods are often limited by high operational costs, secondary pollution risks, incomplete degradation, and low selectivity [2]. Consequently, catalytic reduction is preferred for its efficiency, cost-effectiveness, and ability to convert toxic 4-NP into non-toxic 4-aminophenol [3]. Beyond environmental remediation, this transformation is significantly important because 4-aminophenol is widely recognised as a precursor for the pharmaceutical industry in the production of analgesic and antipyretic drugs. Furthermore, its utility extends to more versatile industrial chemistry, such as the production of corrosion inhibitors, hair dyes, and photographic developers [1].

Metal nanoparticles are widely used as catalysts because their high surface area provides numerous active sites. Specifically, nanostructures composed of copper, silver, gold, and platinum are highly regarded for their exceptional catalytic, optical, and electronic properties. These properties make them essential materials in fields like energy, healthcare, and environmental remediation. Copper nanoparticles are valued for their stability, redox ability, and economic value and are readily available in nature [4]. However, their small size and high surface energy make them prone to agglomeration, which diminishes their catalytic performance. To overcome this, nanoparticles are impregnated onto porous supports, such as carbon-based materials like hydrochar, which improves their dispersion, prevents agglomeration, and enhances their activity [5]. Agricultural residues, such as coconut husks, serve as excellent, sustainable feedstocks for hydrochar production due to their high carbon content and strong adsorption capacity. According to Hamad et al. [2], supporting copper nanoparticles on oxygen-rich hydrochar creates a highly efficient catalyst that combines adsorption with high redox activity. The hydroxyl (–OH) and carboxyl (–COOH) groups on the hydrochar surface enhance pollutant adsorption, and copper nanoparticles facilitate rapid electron transfer during catalytic reduction.

Furthermore, traditional recovery methods, such as filtration and centrifugation, are often time-consuming and lead to significant catalyst loss during the washing cycles. Incorporating magnetite enhances sustainability by enabling efficient magnetic separation and catalyst recovery. This simplifies the recycling process while ensuring the catalyst can be reused in successive cycles without losing structural integrity or catalytic performance. Chen et al. [6] reported that magnetite supported on carbonaceous materials enhances catalyst stability and reusability for wastewater treatment, while also enabling the high-value utilisation of biomass waste. Despite extensive studies on various metal-supported catalysts, limited research has focused on the synergistic integration of copper nanoparticles with magnetic hydrochar specifically derived from coconut husk to create high-performance, cost-effective, and easily recoverable catalytic systems.

The combination of Cu active sites, hydrochar support, and magnetic separation capability is expected to enhance catalytic efficiency while enabling easy recovery and reuse of the catalyst. Addressing this gap is crucial for developing sustainable remediation technologies that utilise agricultural waste. Therefore, this study synthesised a Cu-MHC catalyst via hydrothermal carbonisation to investigate the catalytic reduction activity of 4-NP. The synthesised Cu-MHC catalyst was further characterized by XRD, FESEM-EDX and TEM for elemental and structural studies.

2. MATERIALS AND METHODS

2.1 Materials

Coconut husks were sourced from Lee Agro Trading in Jitra, Kedah, Malaysia, as the main raw material for synthesising the Cu-MHC catalyst and were further tested for catalytic reduction activity. The chemicals used in this study include Iron (III) chloride ($FeCl_3$) (R&M Marketing), iron (II) sulfate ($FeSO_4$) (Acros Organics), ammonium hydroxide (NH_4OH) solution (R&M Marketing), copper (II) nitrate trihydrate ($Cu(NO_3)_2{\cdot}3H_2O$) (Acros Organics), sodium hydroxide (NaOH) (Thermo Fisher Scientific), 4-NP (Acros Organics) and sodium borohydride ($NaBH_4$) (R&M Marketing).

2.2 Preparation of hydrochar from coconut husk

The coconut husk was dried in an oven at 150 °C for 6 hours. The dried material was ground using a blender for 3 to 5 minutes to reduce the material into smaller, uniform particles. Hydrothermal carbonisation was then applied to convert the coconut husk to hydrochar. Briefly, 3 g of ground coconut husk was mixed with 75 mL and placed in a Teflon-lined stainless-steel reactor at 200 °C for 6 hours. After cooling to room temperature, the obtained hydrochar was collected and oven-dried at 110 °C for 7 hours.

2.3 Synthesis of Cu Magnetic Hydrochar Nanocomposites (Cu-MHC NC)

At room temperature, 1.6 g of iron (III) chloride and 0.6 g of iron (II) sulfate in a 2:1 molar ratio was stirred with 0.5 g of hydrochar in 30 mL of distilled water at room temperature, maintaining a weight-to-weight, 1:1 mass ratio of iron oxide to hydrochar. Subsequently, 6 mL of ammonium hydroxide (28 %) was added dropwise at a rate of 1 mL per minute. The mixture was transferred into a Teflon-lined stainless-steel reactor and heated at 150 °C for 3 hours. Then, the resulting magnetic hydrochar was washed with distilled water to remove any impurities and dried at 80 °C.

To produce the Cu magnetic hydrochar, copper precursors were first synthesised using the co-precipitation method. A 0.1 M solution of copper nitrate (II) trihydrate was dissolved in 100 mL of deionised water. Then, 1 M NaOH solution was gradually added under continuous stirring with a magnetic stirrer until the pH reached 10, indicated by the formation of a blue precipitate. The blue precipitate was filtered, rinsed with deionised water and calcined in an oven at 200 ℃ for 1 hour to decompose the nitrate precursor and stabilise the copper species. After the drying process, copper nanoparticles will then be dispersed in distilled water and stirred for 15 minutes. Magnetic hydrochar was added to the mixture and stirred for an additional 60 minutes for proper impregnation. The resulting nanocomposite was dried at 90 °C for 4 hours.

2.4 Catalytic reduction of 4-nitrophenol

The catalytic reduction of 4-NP was carried out according to the method described by Nasrollahzadeh et al. [7], with slight modifications. The catalyst was used to accelerate the reduction of this toxic organic pollutant into less harmful substances. Specifically, a 0.12 mM aqueous solution of 4-NP was prepared. A 10 mM $NaBH_4$ solution was freshly prepared by dissolving 3.78 mg in a 100 mL volumetric flask. Then, 25 mL of 10 mM $NaBH_4$ solution was mixed with 25 mL of 0.12 mM 4-NP in a 100 mL beaker under constant stirring for 1 minute. Then, 30 mg of Cu-MHC catalyst was added to the mixture and stirred at room temperature. About 3.5 mL of the sample mixture was withdrawn and filtered through a 0.45µm syringe filter to remove the catalyst particles. The reaction progress was monitored using a UV-Vis spectrophotometer (Varian Cary 50) by recording the absorption spectra every 35 seconds until the bright yellow solution became colourless. The reduction of 4-NP was measured at an absorbance wavelength of 400 nm. The percentage reduction of 4-NP was calculated using Equation (1).

$$\text{Percentage Reduction (\%)} = \left(\frac{C_0 - C_t}{C_0}\right) x\ 100 \tag{1}$$

C_0 is the initial concentration of 4-NP, and C_t is the concentration at time t.

2.5 Materials Characterisation

The as-synthesised Cu-MHC and MHC alone were characterised using several analytical techniques. Crystalline phase analysis was performed using X-ray diffraction (XRD) (Rigaku Miniflex 600), recording diffraction angles (2θ) from 20 to 80 degrees at a wavelength of λ = 0.154 nm. The resulting diffraction profiles were matched with standard Joint Committee on Powder Diffraction Standards (JCPDS) files. Surface morphology analysis was conducted by Field Emission Scanning Electron Microscopy (FESEM) (JEOL IT800) equipped with on-system Energy-Dispersive X-ray Spectroscopy (EDX) for elemental analysis. While the Transmission Electron Microscope (TEM) (Talos L120C) provide the morphology, particle size and crystalline information of the synthesised catalyst.

3. RESULTS AND DISCUSSION

3.1 Catalytic Reduction of 4-nitrophenol by Cu-MHC Catalyst

The catalytic performance of the Cu-MHC catalyst prepared using coconut husk was evaluated using the catalytic reduction process of 4-NP to 4-aminophenol (4-AP) in the presence of $NaBH_4$. The introduction of $NaBH_4$, 4-NP solution initially a bright yellow, following the addition of Cu-MHC, the colour gradually faded, confirming the conversion of 4-NP to 4-AP. The schematic representation of the reduction of 4-NP to 4-AP over the Cu-MHC catalyst in the presence of $NaBH_4$ is shown in Figure 1. The catalyst enhanced the reaction efficiency, accelerating the conversion of 4-NP to 4-AP. The effects of Cu loading, catalyst amount, and 4-NP concentration on the degradation efficiency were subsequently examined

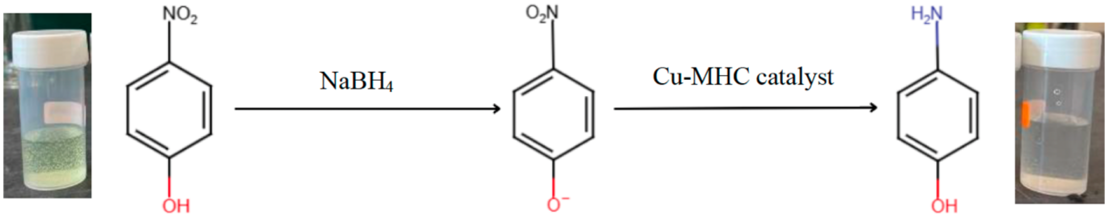

Figure 1: Schematic illustration of the catalytic reduction of 4-NP to 4-AP over Cu-MHC in the presence of $NaBH_4$

For the effect of different percentages of Cu loading on MHC, various percentages of Cu (1 %, 5 % and 10 %) were impregnated onto MHC. The findings, as illustrated in Figure 2(a), show that the 5 % Cu-MHC catalyst exhibited superior performance, with the highest percentage reduction of 95.92 % within 175. This was followed by the 10 % Cu-MHC (92.62 %), 1 % Cu-MHC (15.38 %) and MHC alone (13.25 %). These results suggest that at lower Cu loading (1 %), the number of Cu active catalytic sites is insufficient to promote significant reduction. Meanwhile, at higher Cu loading (10 %), excess copper can lead to nanoparticle agglomeration and pore blockage, as supported by EDX elemental mapping results. This reduces the effective catalytic surface area and limits copper utilisation efficiency, a phenomenon also mentioned by Ali et al. [8].

Subsequently, four different dosages of 5 % Cu-MHC (10, 20, 30, and 40 mg) were evaluated to determine the optimal catalyst dosage for 4-NP reduction. Figure 2 (b) illustrates the 4-NP reduction profile over time while maintaining 4-NP concentration. The results showed that all tested dosages exhibit a rapid initial increase but level off after reaching an optimum point. This behaviour may be attributed to the saturation of active sites relative to the available substrate. Once the 4-NP molecules have fully occupied the accessible copper sites, adding excess catalyst does not further enhance the reaction rate, as the substrate has become saturated.

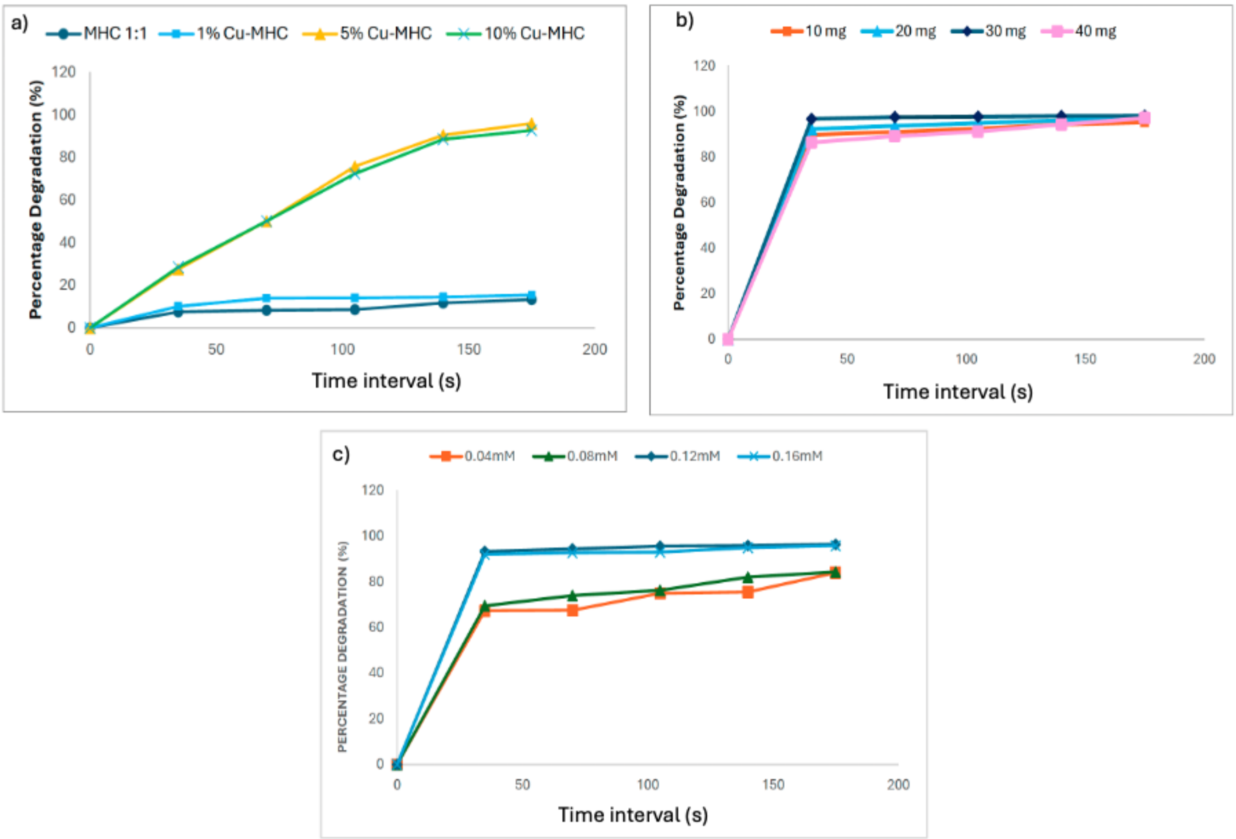

Figure 2: Reduction of 4-NP by Cu-MHC as a function of (a) Cu loading, (b) catalyst dosage and (c) 4-NP concentration

Among the tested dosages, 30 mg of catalyst achieves a slightly higher reduction efficiency of 98 % within 175 seconds. The reduction efficiencies for 10, 20, 30, and 40 mg of catalyst were nearly identical, with all dosages reaching near-complete reduction within a similar timeframe. This suggests that increasing the catalyst dosage beyond a certain level does not significantly enhance performance. The results indicate that even a minimal amount of catalyst (10 mg) is sufficient to achieve near-complete reduction of 4-NP, underscoring the cost-effectiveness and efficiency of the process by minimising the required catalyst dosage.

Furthermore, the influence of varying initial 4-NP concentrations was examined. Experiments were conducted using 0.04, 0.08, 0.12, and 0.16 mM of 4-NP to identify the optimal concentration for the reaction with 30 mg of 5 % Cu-MHC. From Figure 2 (c), the profile demonstrates an initial rapid rise in reduction percentage for all concentrations, which subsequently slows down as the reaction proceeds and eventually reaches a plateau as 4-NP is depleted. Higher concentrations of 4-NP (0.12 mM and 0.16 mM) exhibited excellent reduction properties, with 0.12 mM concentration exhibiting the highest percentage reduction (96.43 %). The increased concentration gradient between the 4-NP solution and the catalyst surface may enhance the diffusion of 4-NP onto the active sites, thereby optimising the reduction percentage. Conversely, at lower concentrations (0.04 mM and 0.08 mM), the reduction percentage was slightly decreased, suggesting that under very dilute conditions, the collision frequency between the 4-NP molecules and the Cu-MHC surface decreases, potentially making the reaction mass-transfer-limited. The optimal concentration of 4-NP for efficient reduction was found to be 0.12 mM, achieving 96.43 % reduction, followed closely by 0.16 mM with 92.05 % reduction within 175 seconds.

3.2 XRD Analysis

The elemental and crystallinity of 5 % Cu/MHC catalyst can be obtained using the X-ray Diffraction (XRD) pattern illustrated in Figure 3. The diffraction peaks observed at $2\theta = 30.16^\circ$, 35.53°, 43.18°, 53.57°, 57.11° and 62.71°correspond to the (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) planes of Fe_3O_4, respectively, consistent with the face-centered cubic (FCC) structure of magnetite (JCPDS 19-0629). Furthermore, the phase analysis of MHC alone confirmed a diffraction pattern corresponding to the characteristic planes of Fe_3O_4, aligning with results reported by Patiño et al. [9]. Similar diffraction patterns observed for the 5 % Cu-MHC catalyst indicate that the magnetic material was successfully incorporated into the hydrochar, forming a composite. Moreover, the absence of copper peaks in the XRD pattern of 5 % Cu-MHC could be attributed to either a relatively low copper loading or to copper particles with crystallite sizes below the XRD instrument's detection limit. However, the elemental distribution observed in the EDX mapping (Figure 5(e)), suggests that the copper species are highly dispersed across the magnetite hydrochar matrix. In addition, the slight shifts to a lower 2-theta in most of 5 % Cu-MHC peaks strongly indicate that copper ions have successfully integrated on the magnetite crystal lattice, confirming the effective incorporation of copper into the structure as reported by Zhang et al. [10].

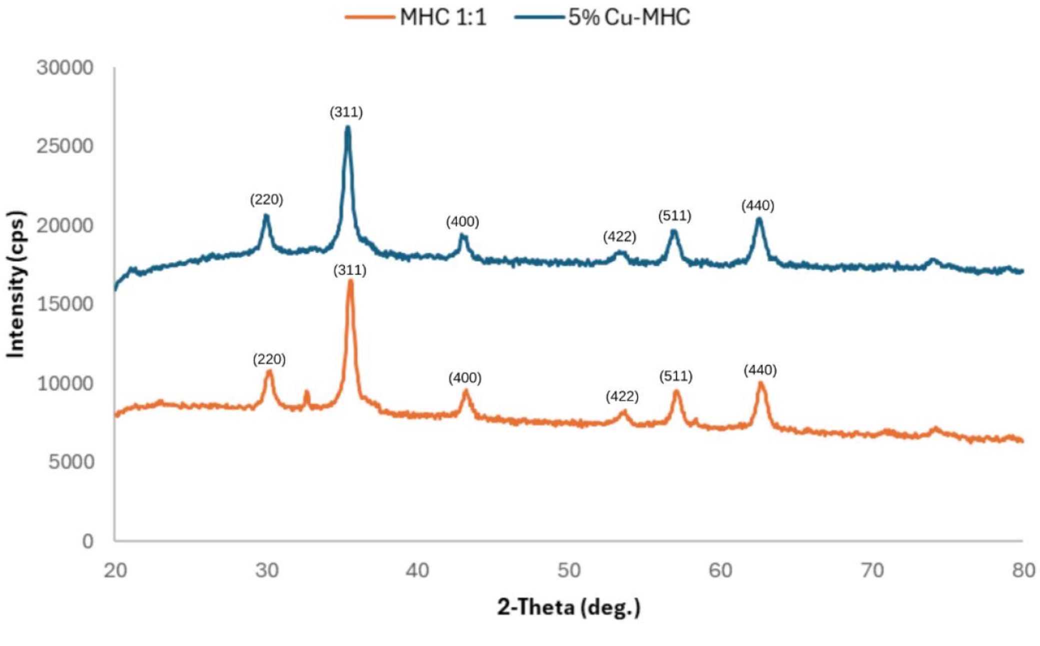

Figure 3: XRD pattern for MHC and 5% Cu-MHC catalyst

3.3 FESEM-EDX analysis

Field Emission Scanning Electron Microscopy (FESEM) coupled with Energy Dispersive X-ray Spectroscopy was performed to investigate the surface morphology and confirm the presence of Cu. FESEM analysis of the hydrochar alone exhibits a rough and compact morphology and is rich with fibrous fragments, almost similar to the biomass-derived hydrochar reported by Ormaza et al. [11] and Shi et al. [12]. Moreover, at higher magnification, the hydrochar revealed wrinkles and folded structures with a central cavity. This indicates that the coconut husk cell-wall structure appears to be mostly collapsed, as shown by the FESEM image in Figure 4.

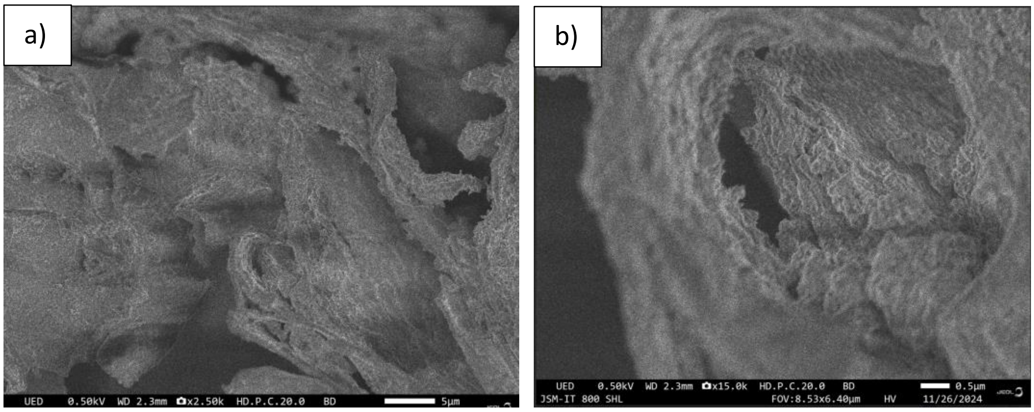

Figure 4: FESEM image of hydrochar at (a) low magnification with scale value is 5 μm and (b) higher magnification with scale value is 0.5 μm

The 1, 5, and 10 % Cu-MHC catalysts were further characterised to provide both morphological and elemental information. Figure 5 illustrates the FESEM images, EDX elemental mappings and EDX elemental spectrums of 1 % Cu-MHC catalyst (Figure 5 (a-c)), 5 % Cu-MHC catalyst (Figure 5 (d-f)) and 10 % Cu-MHC catalyst (Figure 5 (g-i)).

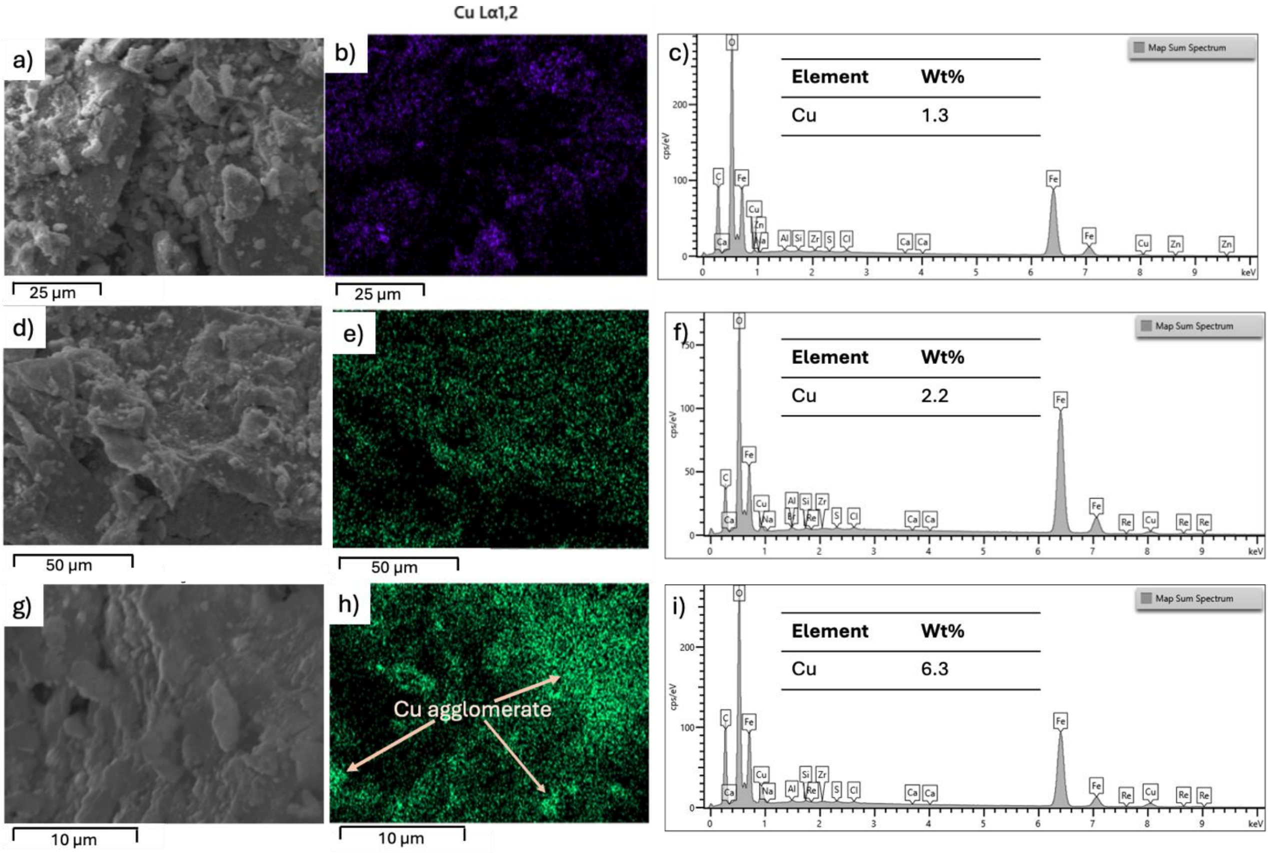

Figure 5: FESEM images, EDX elemental mapping and EDX spectra of (a-c) 1 % Cu-MHC, (d-f) 5 % Cu-MHC and (g-h) 10 % Cu-MHC

From the results, the addition of Cu and magnetic components disrupts the native hydrochar morphology, resulting in a more compact, irregular surface compared to the unmodified hydrochar. At the lowest percentage loading (1 % Cu-MHC), the surface morphology shows a rough and irregular carbonaceous surface, with Cu distribution appearing weak and lightly dispersed, as observed in Figures 5 (a) and (b).

The EDX elemental spectrum reveals a Cu content of 1.3 wt.%, relatively good agreement with the theoretical loading. At 5 % and 10 % Cu-MHC, the surface morphology of the magnetic hydrochar showed a layered plate-like structure, reflecting the intensified interaction between Cu species and the magnetic hydrochar. At 10 % Cu loading, small agglomeration becomes obvious, attributable to the excess of Cu precursor that leads to particle clustering, as further supported by the EDX elemental mapping in Figure 5 (h). While 5 % Cu loading exhibits relatively well-distributed Cu species on magnetic hydrochar, which gives excellent catalytic performance on the reduction of 4-NP. However, as the intended loading increased to 5 % and 10 %, the measured values were significantly lower at 2.2 wt% and 6.3 wt%, respectively. This discrepancy suggests that a portion of the Cu precursor was not incorporated or was lost during the post-synthesis washing and filtration stages. Furthermore, considering the inherent limitation of EDX, the weight percentages obtained reflect localised elemental concentrations on the catalyst's surface rather than the comprehensive bulk loading throughout the magnetic hydrochar matrix.

3.4 TEM Analysis

The microscopic structural details and lattice characteristics of the 5 % Cu-MHC nanocomposite were elucidated through TEM, as presented in Figure 6 (a)-(c). The TEM image reveals a predominantly spherical to sub-spherical morphology, with a dense and high distribution of nanoparticles immobilised across the hydrochar support (Figure 6 (a)). The coconut husk-derived hydrochar serves as an effective platform to anchor copper and magnetic components, thereby preventing the severe agglomeration often associated with unsupported metal nanoparticles. While the particles exhibit a high degree of dispersion, Figure 6(b) also revealed the formation of localised nanoclusters, with the particles retaining their distinct spherical boundaries with slight clustering, allowing them to remain accessible to 4-NP molecules.

Furthermore, Figure 6(c) provides the measurements of the particle size and crystallinity. The individual particles exhibit approximately diameters ranging from 15 to 20 nm, indicating successful formation of nano-sized particles with a relatively narrow size. These nanosized particles are a primary factor in the high activity of the synthesised catalyst. The images display clear and well-defined lattice fringes with interplanar spacings of approximately 629.0 – 647.6 pm, confirming both the crystalline nature of the copper species or magnetite within the hydrochar matrix. This direct observation of crystallinity provides essential evidence that the copper species might be in a crystalline phase. This effectively explains the absence of copper peaks in the XRD pattern with a slight shift of the peaks associated with small crystallite sizes rather than an amorphous state.

In addition, the high catalytic activity of the 5 % Cu-MHC is mostly attributed to a surface-mediated process following the Langmuir-Hinshelwood mechanism. In this model, Cu particles (TEM analysis indicates 15–20 nm) serve as specific active sites where both 4-NP and BH4- ions are simultaneously adsorbed and undergo rapid electron transfer from the borohydride donor to the nitro group of 4-NP. The resulting 4-AP desorbs from the surface as the reaction completes. The magnetic hydrochar support is crucial for preventing the agglomeration of Cu nanoparticles by maintaining the high dispersion of Cu particles, which explains the significant performance increase compared to without Cu particles (MHC alone). To further evaluate the results, the performance of the 5 % Cu-MHC was compared with a previous study by Yu et al. [13] using copper-based catalyst supported on magnetic maize straw. Both catalysts utilise agricultural waste combined with the magnetic properties and follow the same reaction mechanism. In this study, 5 % Cu-MHC achieved 98 % reduction in 3 minutes, which is remarkably faster than the 10 minutes required by the maize-straw-based catalyst. In this study, smaller, finer, and spherical nanoparticles are obtained, while the maize-straw-based catalyst reported much larger, flower-like structures with diameters between 220 and 800 nm. Therefore, the superior performance of 5 % Cu-MHC may be attributed to the smaller particle size, better morphology and high dispersion of Cu particles that provides a significantly enhanced available catalytic surface area, allowing for a more efficient and rapid electron transfer during the reduction of 4-NP.

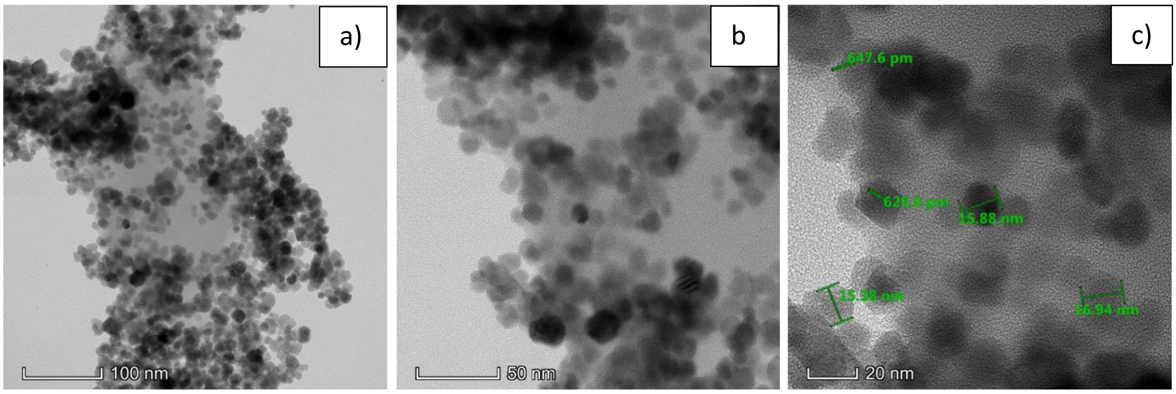

Figure 6: TEM images of the 5 % Cu/MHC catalyst at magnifications with the scale value of (a) 100 nm, (b) 50 nm and (c) 20 nm

4. CONCLUSIONS

The production of Cu-magnetic hydrochar using coconut husks has been successfully synthesised. The magnetic hydrochar acts as a stable support for copper nanoparticles, enhancing their dispersion and stability during the catalytic reduction of 4-NP to 4-AP. The optimal reduction was achieved using 30 mg of 5 % Cu–magnetic hydrochar at a 4-NP concentration of 0.12 mM, resulting in approximately 98 % reduction within 175 seconds. This excellent performance is attributed to the high dispersion of Cu on the magnetic hydrochar. Overall, this study provides a promising approach for the development of biomass-derived catalysts for wastewater treatment. Future research will focus on detailed kinetic analysis and comprehensive recyclability studies to confirm its long-term stability and suitability for large-scale industrial applications. Furthermore, the versatility of the 5 % Cu-MHC catalyst will be evaluated against other toxic organic pollutants, such as organic dyes or different nitro-compound derivatives.

Acknowledgements

This work received financial support by the Universiti Sains Islam Malaysia under the grant scheme of Research Grant USIM (PPPI/USIM/FST/USIM/16424). The authors would also gratefully acknowledge the Faculty of Science and Technology, USIM, for the facilities throughout the work.

Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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