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Vitis Vinifera (Grape Stem Branch Powder) as a Natural Adsorbent for Removal of Cu (II) from Aqueous Solutions

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Abstract: ***Copper (II) contamination in water poses significant environmental and health risks due to its non-biodegradable nature and toxic effects at elevated levels. This study investigates the use of*** *Vitis vinifera* ***(grape stem branch powder), an agricultural waste, as a low-cost and eco-friendly biosorbent for Cu(II) removal from aqueous solutions. Batch adsorption experiments were conducted to evaluate the influence of pH, contact time, adsorbent dosage, initial Cu(II) concentration, and temperature on removal efficiency. Characterization using FTIR confirmed the involvement of hydroxyl and carboxyl groups in Cu(II)binding, while SEM and EDX analyses revealed surface changes and metal deposition after adsorption. The results demonstrate that grape branch powder is an effective and sustainable material for Cu(II) removal, offering a promising alternative for wastewater treatment.***

Keywords: Biosorption, Eco-friendly adsorbent, copper removal, Wastewater treatment, Wastewater treatment

1. Introduction

Copper (II) contamination is an escalating environmental concern due to its extensive industrial use and non-biodegradable nature. Although copper is an essential micronutrient, its excessive intake surpassing physiological requirements leads to severe health problems, including gastrointestinal distress, kidney damage, anemia, and liver accumulation. Chronic copper exposure is further linked to conditions such as hair loss, oxidative stress, Wilson's disease, insomnia, and neurodegenerative disorders (Suja et al., 2024). The sources of copper pollution are diverse, ranging from industrial effluents, mining, and battery manufacturing to the excessive use of copper-based fertilizers and fungicides. These agrochemicals accumulate in soils and leach into nearby rivers and ground water through surface runoff, particularly during heavy rainfall events (Abubakar, 2024). Another major contributor is the corrosion of copper pipes and plumbing systems in older buildings, which introduces high copper levels into drinking water supplies (Hussain et al., 2010).

Copper (II) contamination is also toxic to aquatic ecosystems, causing oxidative stress, impaired osmoregulation, metabolic disturbances, and gill damage in fish (Bhat et al., 2019). Unlike organic pollutants, copper does not degrade but persists in the environment, bioaccumulates in organisms, and biomagnifies through the food chain, posing long-term threats to ecological and human health (Alloway, 2013; Radisson et al., 2023). Factors such as soil pH, organic matter, and redox potential further affect copper's mobility and bioavailability, complicating its removal (Olaniran et al., 2013). Historical records also reveal significant copper levels in ice cores dating back to early industrialization, reflecting its enduring environmental presence (Driscoll et al., 2013). The problem has been intensified by unregulated disposal of copper-containing waste from industrial and domestic sources (Kakkar et al., 2010). Agencies like the EPA and WHO have set permissible limits for copper in soil and water to mitigate health and environmental risks (Mustafa & Hassan, 2024).

Traditional copper remediation methods, including ion exchange, membrane filtration, chemical precipitation, and electrochemical treatments, although effective, are costly and generate hazardous by-products (International Atomic Energy Agency, 2002; 2004; Hawaas et al., 2023). As a sustainable alternative, biosorption using natural adsorbents has gained attention for being cost-effective, eco-friendly, and efficient. Among such materials, *Vitis vinifera* (grape branch powder), an agricultural byproduct, shows great promise due to its high surface area, porosity, and abundance of functional groups such as hydroxyl, carbonyl, and carboxylic acids. These active sites facilitate strong interactions with Cu(II) ions via ion exchange, electrostatic attraction, and complexation mechanisms.

To understand the surface characteristics and adsorption mechanisms, the adsorbent was characterized using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX). FTIR analysis confirmed the presence of functional groups responsible for copper binding. SEM images revealed morphological changes before and after adsorption, indicating Cu (II) ion deposition on the adsorbent surface. EDX analysis verified the elemental presence of copper post-adsorption, supporting the successful biosorption process.

This research highlights *Vitis vinifera* as an effective, sustainable biosorbent for Cu(II)removal from water and offers an environmentally friendly solution for mitigating heavy metal pollution in wastewater treatment applications.

2. Materials and Methods

In this study, Vitis vinifera (grape stem branch powder) was employed as a biosorbent for the removal of copper (Cu II) ions after treatment from aqueous solutions. The grape branches were collected, washed thoroughly with distilled water to remove impurities, sun-dried, crushed, and sieved to obtain uniform particle size. This powder was treated with 39% formaldehyde and 0.1 mol L^{-1} sulphuric acid at 80^0 C for 30 minutes. After cooling and washing with dobble distilled water, substrate was allowed to dry for over night in open air. (Kuchekar et al. 2020) Driedpowder was stored in an airtight container and used for adsorption study. A 1000 ppm Cu (II) standard solution was used to prepare the desired concentrations for batch adsorption studies. The effects of various parameters such as pH (3.0–12.0), contact time (15–120 minutes), adsorbent dosage (0.1–3.0 g), initial metal ion concentration, and temperature (30–60°C) were investigated to determine optimal conditions for Cu (II) removal. Adsorption experiments were conducted in 100 mL Erlenmeyer flasks containing 25 mL of Cu (II) solution and a measured quantity of biosorbent, shaken on a rotary shaker to ensure proper interaction. After equilibrium, the solutions were filtered using Whatman No. 41 filter paper, and residual Cu (II) concentrations were analyzed using UV-Visible Spectrophotomer. Percentage removal and adsorption capacity were calculated using standard formulas. The surface morphology and elemental composition of the biosorbent before and after adsorption were analyzed using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX), while functional groups involved in Cu binding were identified through Fourier Transform Infrared Spectroscopy (FTIR), providing insights into the adsorption mechanism.

3. Results and discussion

1) Effect of Contact Time

The removal efficiency of copper (II) ions increased with contact time, reaching a maximum of 78.1% at 75 minutes. This rapid uptake during the initial phase was due to the availability of numerous active sites on the adsorbent surface. After 75 minutes, a slight decrease in efficiency was observed, likely due to saturation of binding sites or possible desorption at longer durations. Thus, 75 minutes was identified as the optimum contact time for effective adsorption (Fig. 1).

2) Effect of Adsorbent Dosage

Copper (II) adsorption increased with increasing adsorbent dosage, attaining the highest removal efficiency of 81.0% at 0.2 g. This improvement is attributed to the greater number of available binding sites. However, further increasing the dosage beyond 0.2 g led to a decrease in efficiency, likely caused by particle aggregation and reduced surface area exposure. Therefore, 0.2 g was considered the optimal dosage for maximum uptake (Fig. 2).

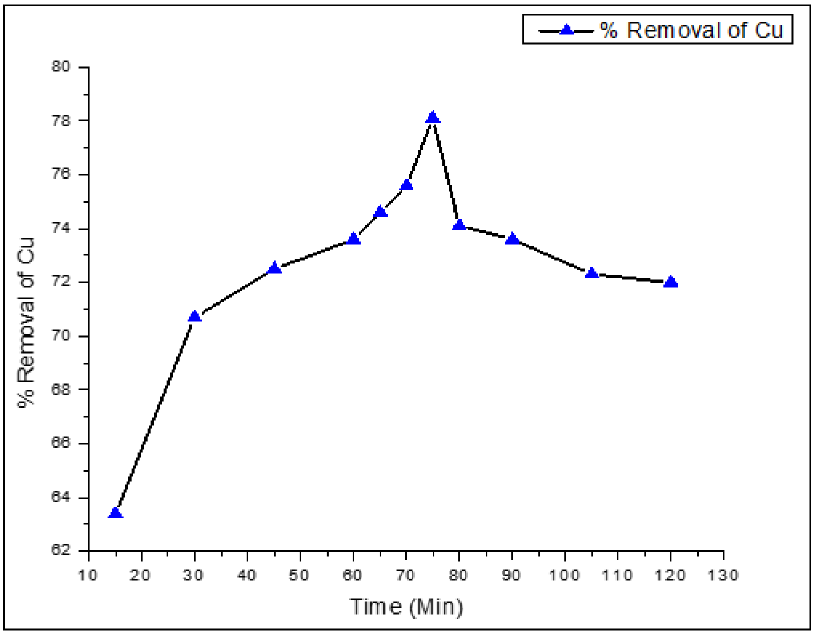


Figure 1: Effect of contact time on adsorption of Cu(II)

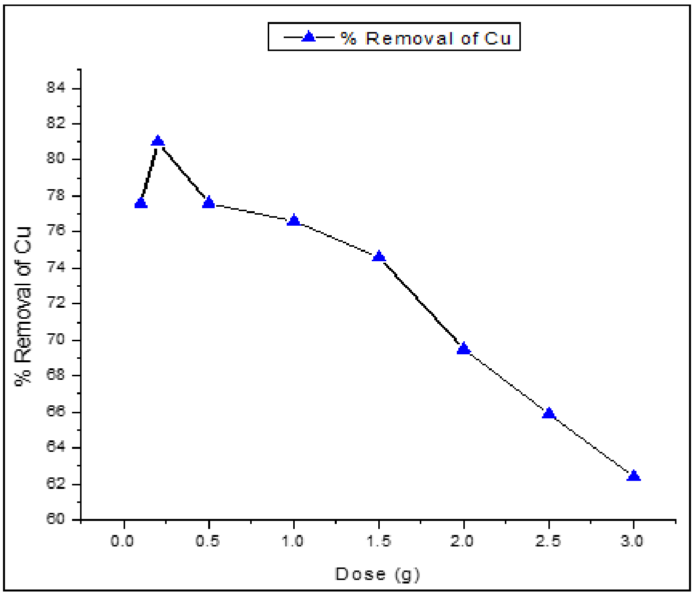


Figure 2: Effect of adsorption dose on adsorption Cu(II)

3) Effect of pH

The pH of the solution significantly influenced copper (II) adsorption. The removal efficiency increased with rising pH, peaking at 86.1% at pH 5. At low pH, excess H^+ ions competed with Cu(II) for adsorption sites, lowering efficiency. Beyond pH 5, reducing the amount of free Cu(II) ions available for adsorption. Hence, pH 5 was found optimal for copper (II) removal (Fig. 3).

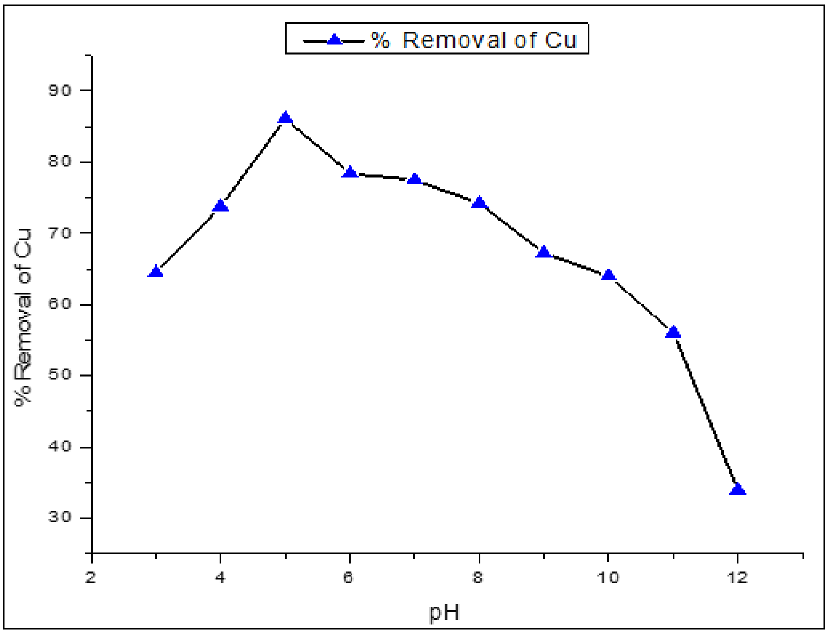


Figure 3: Effect of pH on adsorption of Cu(II)

4) Effect of Temperature

Temperature variation showed that copper (II) removal increased with temperature, reaching 82.3% at 40°C. The rise in efficiency is due to enhanced mobility of metal ions and better interaction with active sites. However, temperatures above 40°C caused a gradual decline in adsorption, likely due to thermal desorption or alteration of the adsorbent structure. Therefore, 40°C was identified as the optimal temperature (Fig. 4).

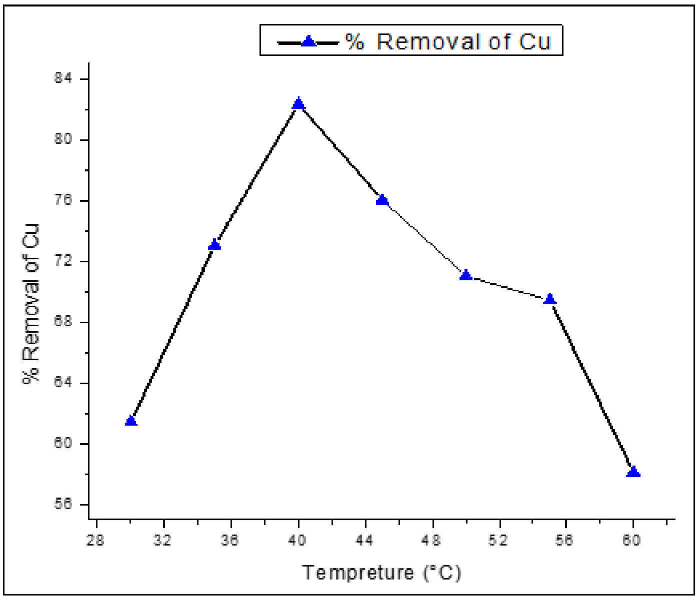


Figure 4: Effect of temperature on adsorption of Cu(II)

5) Effect of Initial Metal Ion Concentration

Adsorption efficiency increased with initial copper (II) concentration, with maximum removal of 81.1% observed at 650 µg. This was due to the higher driving force for mass transfer at elevated concentrations. However, concentrations above 650 µg led to decreased efficiency, indicating that available adsorption sites became saturated. An optimum concentration is essential to ensure efficient removal without overloading the adsorbent (Fig. 5).

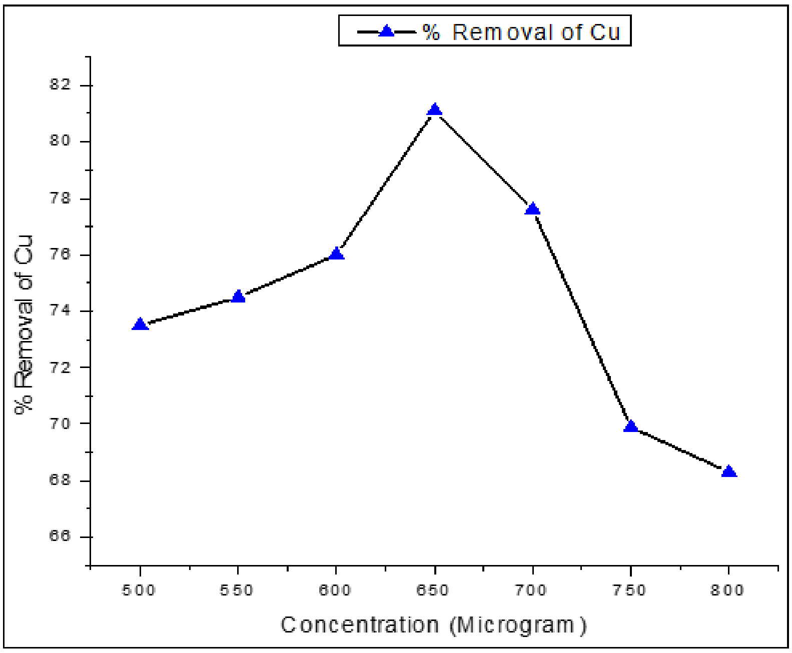


Figure 5: Effect of initial metal ion conc. on adsorption of Cu(II)

6) FTIR Spectra of Vitis Vinifera of Cu (II) Adsorption

Fourier Trasnsform Spectra obtained from vitis vinifera before and after absorption of Cu (II) metal ion reported in Fig. 6 and 7 respectively.The strong band observed in 1318 cm^{-1} 1368 cm^{-1} indicating –CN starching vibration of protein fractions.Thepeak at 1420 cm^{-1} showing symmetric banding of CH_3 of acetyl moiety. The band at 1583 cm^{-1} due to C=O stretching vibrations, the absorption band at the region between 2912 cm^{-1}, 3328 cm^{-1}, showed the presence of CH Stretching vibration pack at 2232 cm^{-1} to 1880 cm^{-1} and 1050 cm^{-1} to 800 cm^{-1} indicates C-X bond.The –NH group at variable wave number 1318 cm^{-1}, 2232 cm^{-1}, attributing vibrations of NH_2.

The spectra obtain due to interaction of functional group on the vitis vinifera with Cu (II), the FTIR peak might shift to lower to higher wave numbers after absorption of Cu (II) (Fig.7). The peak obtainedin vitis vinifera at 2912 cm^{-1} shifted to 3313 cm^{-1} in Cu(II) loaded adsorbent. The peaks around 1325 cm^{-1}, 1592 cm^{-1}, 1156 cm^{-1} suggest that carbonyl and amine groups may participate in adsorption.The peaks at 1325 cm^{-1}, 3410 cm^{-1} were attributed to metal chelate of amine, carboxyl and hydroxyl group respectively.

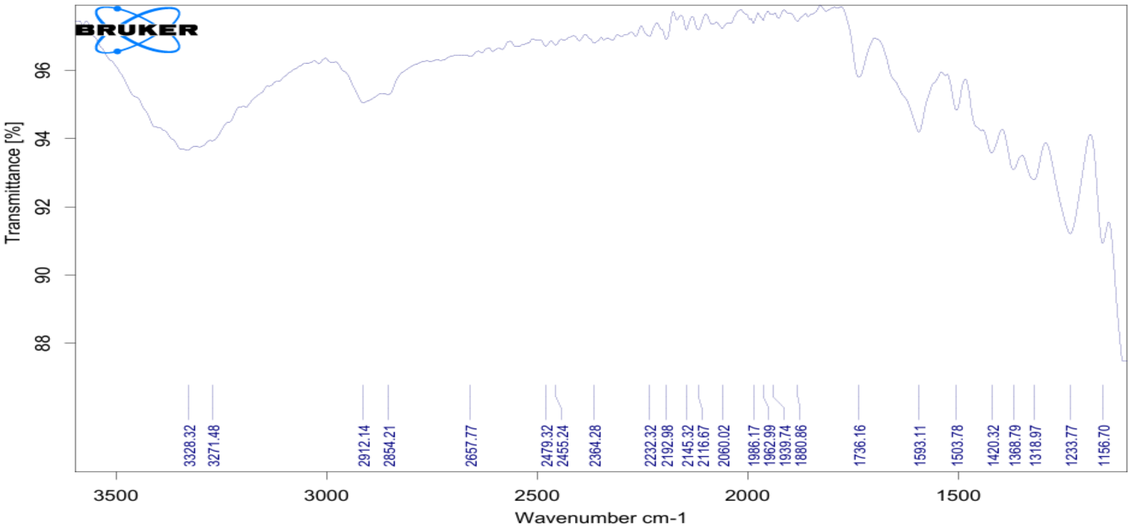


Figure 6: FTIR Spectra of VitisViniferaWithout Cu (II) Adsorption

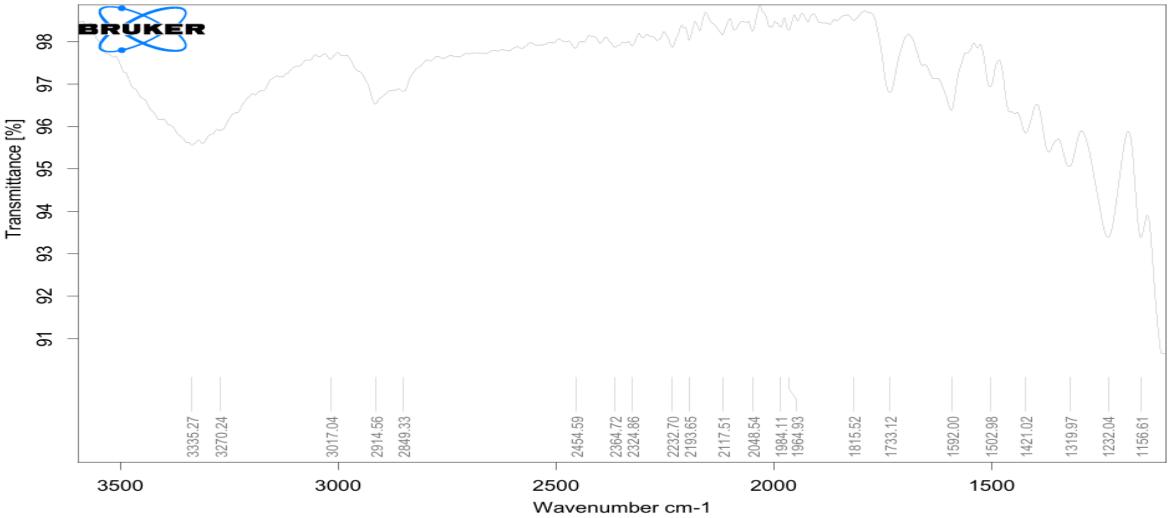
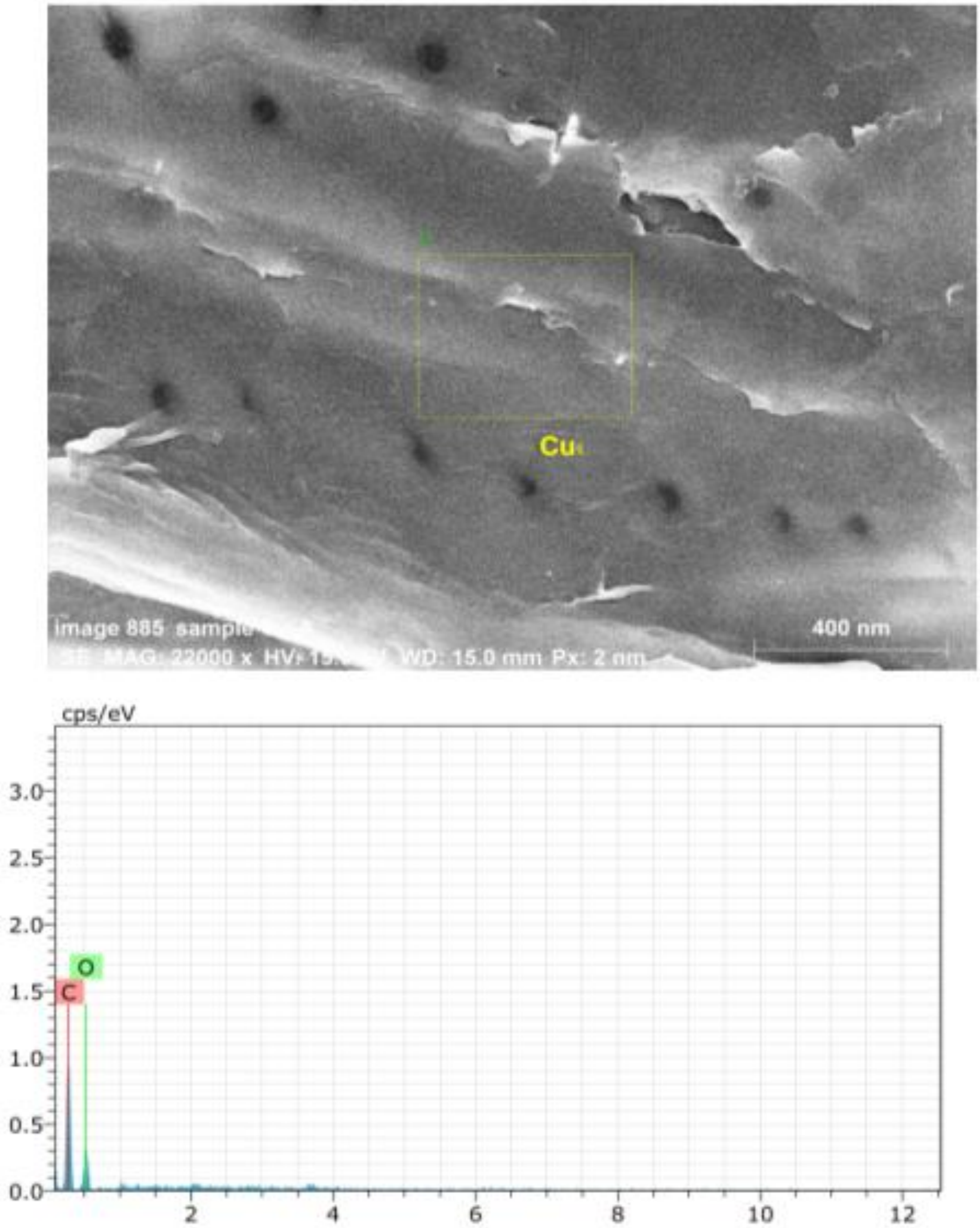


Figure 7: FTIR Spectra of VitisViniferaWith Cu (II) Adsorption

7) SEM micrograph and Energy Dispersive X-ray Analysis of Cu (II)

The morphological analysis of phenol formaldehyde treated with vitis vinifera branch steam powder was performed by as shown in Fig. 8. Many small pores and particles are observed on the surface of adsorbent. In Fig. 9 pores are not observed. It clearly indicates that biosorption of Cu(II) on vitis vinifera adsorbent. EDX spectrum from Fig. 9 also showed peak of 0.8 Kev and 8.0 KeV which conform that Cu (II) was adsorbed on adsorbent. Which was absent in Fig. 8. It support that the 0 reactions of metal ion and phenolic –OH group on treats adsorbent powder, surface may be partly ion exchange or complexation.



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Element   Series   unn. C norm. C Atom. C Error (3 Sigma)
                   [wt.%]  [wt.%]  [at.%]           [wt.%]
-----------------------------------------------------------
Carbon   K-series  61.55   61.55   68.07            36.56
Oxygen   K-series  38.45   38.45   31.93            31.40
-----------------------------------------------------------
          Total:  100.00  100.00  100.00
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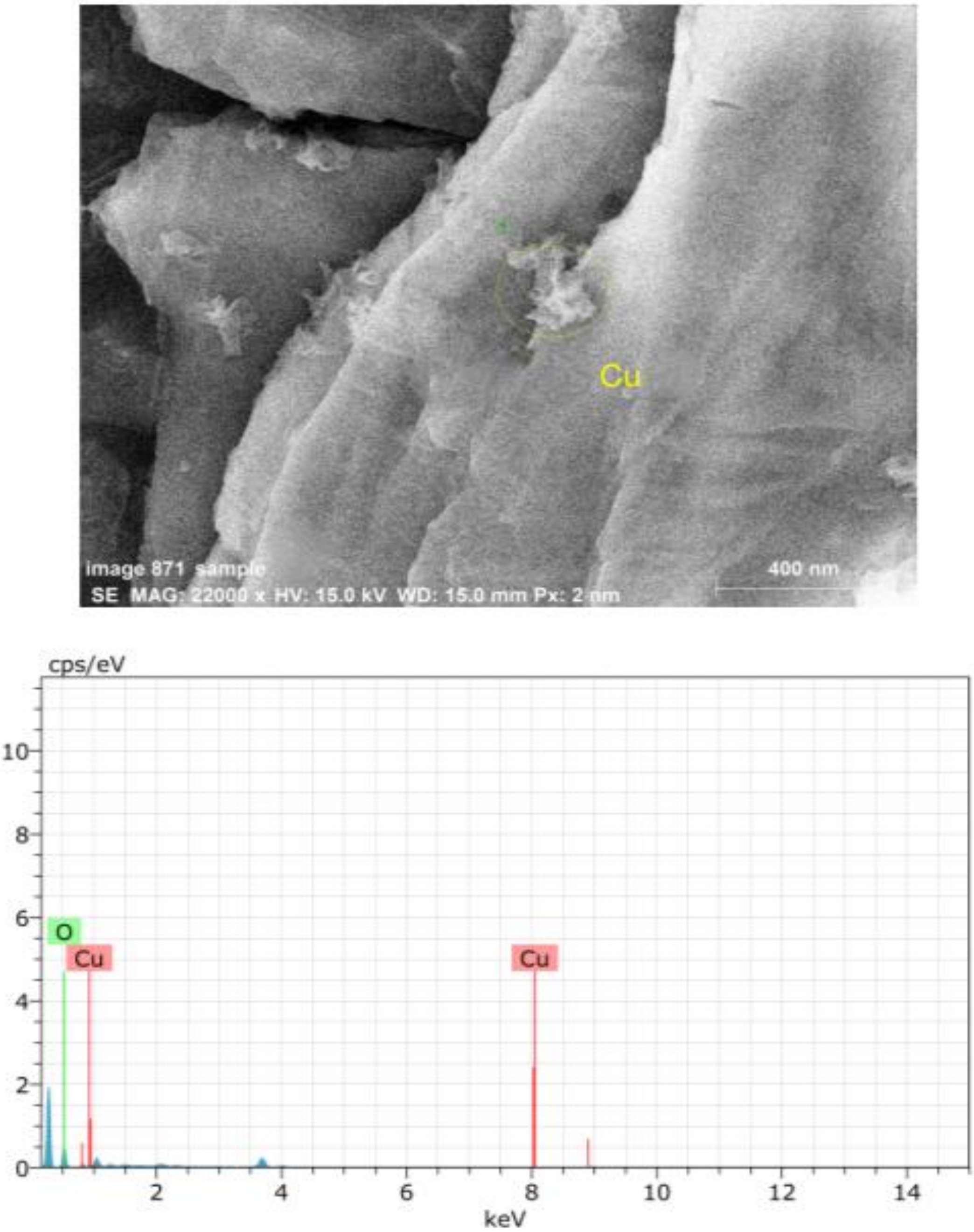
Figure 8: SEM micrograph and Energy Dispersive X-ray Analysis of Adsorbent Before Adsorption.



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Element   Series   unn. C  norm. C  Atom. C Error (3 Sigma)
                   [wt.%]  [wt.%]   [at.%]           [wt.%]
-----------------------------------------------------------
Copper   K-series    8.79     8.79     2.37            4.76
Oxygen   K-series   91.21    91.21    97.63           48.88
-----------------------------------------------------------
           Total:  100.00   100.00   100.00
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Figure 9: SEM micrograph and Energy Dispersive X-ray Analysis of Adsorbrnt After Cu(II) Adsorption

4. Conclusion

The study revealed that copper (II) adsorption using Vitis vinifera (grape stem branch powder) was strongly influenced by contact time, dosage, pH, temperature, and initial metal concentration. Maximum removal (81.0%) occurred at 75 minutes and 0.2 g dosage, with pH 5 proving optimal due to reduced competition from H^+ ions and minimal metal precipitation. The best temperature was 40°C, suggesting an endothermic process, while 650 µg/L was the ideal initial concentration for highest efficiency (81.1%). Overall, the adsorbent demonstrated strong metal-binding ability and high efficiency at low dosages, making it a cost-effective and eco-friendly option for copper removal in wastewater treatment. Further studies on reusability could improve its large-scale application.

The findings highlight the potential of utilizing agricultural waste for environmental remediation. This approach aligns with sustainable practices and supports circular economy principles in wastewater management.

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