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SURFACE CHARACTERIZATION AND POINT OF ZERO CHARGE OF A CU^{2}+ BASED COORDINATION COMPOUND AND ITS POTENTIAL FOR ADSORPTION OF POLLUTANTS

***[1]Sunday E. Elaigwu, [2]Vincent O. Adimula, [2]Olanrewaju A. Rasheed, [2]Bisola R. Efunwole and [1]Adedibu C. Tella**

[1]Department of Chemistry, University of Ilorin, Kwara State, Nigeria.
[2]Department of Industrial Chemistry, University of Ilorin, Kwara State, Nigeria.

*Corresponding authors' email: sunnietrinex@hotmail.com

ABSTRACT

Coordination polymers or compounds have been used as multifaceted materials for different application due to their porosity and high surface area, availability of multicomponent organic ligands and the possibility of using different inorganic metal atoms in their fabrication. Thus, in this study, a Cu^{2}+ based coordination compound was synthesized from 1,2,4,5–benzenetetracarboxylic acid, 4–aminomethylbenzoic acid, NaOH and copper (II) acetate monohydrate by refluxing and characterized for its potential application in the removal of pollutants. Characterization techniques studied includes Fourier Transform Infrared (FTIR) spectroscopy, Transmission Electron Microscopy (TEM), Brunauer–Emmett–Teller (BET) surface area analysis, and X-ray Photoelectron Spectroscopy (XPS). FTIR confirmed successful metal–ligand coordination through O–H, C=O, and Cu–O vibrations, while TEM revealed a porous and heterogeneous morphology, the BET analysis gave a surface area of 72.20 m^2g^{-1}. XPS spectra showed the coexistence of Cu^{2}+ and Cu+ oxidation states. The point of zero charge (PZC) determined by the pH drift method was 5.7, suggesting selective adsorption of anionic pollutants below this pH and cationic pollutants above it. These results demonstrate that the Cu2+ based coordination compound exhibits typical characteristics that favours adsorption of pollutants.

Keywords: Adsorption, Coordination Compound, Copper, Pollutants, PZC, Surface Chemistry

INTRODUCTION

Water pollution caused by industrial effluents, heavy metals, and organic dyes has become a major global concern due to their persistence and harmful impacts on ecosystems and human health (Gupta and Suhas 2009). Conventional water treatment methods such as chemical precipitation, membrane separation, and coagulation often suffer from drawbacks including high cost, incomplete removal, and the generation of secondary waste (Fu & Wang, 2011).

Adsorption on the other hand has been recognized as one of the most effective method for the removal of pollutant owing to its efficiency, simplicity, and potential for regenerability (Wang & Peng, 2010). A crucial factor influencing adsorption efficiency is the surface chemistry of the adsorbent, as well as the point of zero charge (PZC) that represents the pH at which the net surface charge of an adsorbent is zero (Kosmulski, 2009). At solution pH values below the PZC, adsorbent surfaces become positively charged favoring the uptake of anionic species, while above the PZC, the surface becomes negatively charged enhancing uptake of cationic species (Sposito, 1998).

Recently, coordination compounds have emerged as promising candidates for adsorption due to their structural tunability, stability, and functionalizable ligands (Adimula et al., 2025; Lei et al., 2020). Transition metal complexes in particular, provide multiple active sites for surface interactions and can be tailored towards specific adsorption process. Among coordination compounds, the copper-based coordination compounds have gained interest because of their unique redox properties, high affinity for donor ligands, and potential to form porous structures that enhance surface interactions (Nazari-Arshad and Mohammadikish, 2022). However, while numerous studies have reported the synthesis and structural characterization of Cu^{2+} complexes, limited attention has been given to their surface properties and point of zero charge (PZC) determination, which are crucial for understanding their adsorption mechanisms.

Therefore, in this study, we synthesized a Cu^{2+} based coordination compound and investigated the surface properties and point of zero charge, in order to provide insights into its potential application as adsorbents in wastewater treatment. The surface characterization was performed using Fourier transform infrared (FTIR) spectroscopy, Transmission Electron Microscopy (TEM), Brunauer–Emmett–Teller (BET) surface area analysis, and X-ray Photoelectron Spectroscopy (XPS), while the point of zero charge was determined using the pH drift method.

MATERIALS AND METHODS

Analytical grade chemicals and reagents used for the synthesis were obtained from a commercial supplier in Ilorin, Nigeria and were used without further purification. They include dimethylformamide (DMF), sodium hydroxide (NaOH), NaCl, hydrochloric acid (HCl), copper (II) acetate monohydrate, a common precursor used for copper coordination chemistry and the ligands namely 1,2,4,5–benzenetetracarboxylic acid (HBTC), and 4–aminomethylbenzoic acid (AMB). The ligands for the coordination process were selected based on their ability to form stable chelate with the copper metal center, which enhances the material's adsorptive properties.

Synthesis of Cu^{2+} Coordination Compound

The Cu^{2+} coordination compound was synthesized via a reflux method. Briefly, 0.25 g of HBTC was dissolved in 5 ml of DMF, 0.151 g of AMB in 10 ml of distilled water, and 0.04 g of NaOH in 5 ml of DMF. These solutions were heated on a magnetic stirrer to promote complete dissolution. In a separate beaker, 0.199 g of Copper (II) acetate monohydrate was dissolved in 5 ml of distilled water. The ligand solutions were combined in a round-bottom flask, followed by the copper solution. The reaction mixture was refluxed at 110 °C for 2 h, cooled, and filtered to obtain the coordination compound (bluish precipitate). The product was washed with distilled water, air-dried, and stored in a desiccator.

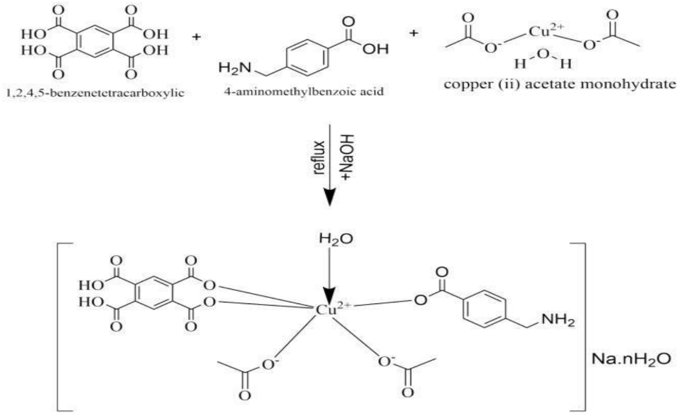

Figure 1: Synthesis of Cu^{2+} Based Coordination Compound from 1,2,4,5–benzenetetracarboxylic acid, 4–aminomethylbenzoic acid, NaOH and copper (II) acetate monohydrate by Refluxing

Surface Characterization

The functional groups of the synthesized Cu-based coordination compound were analyzed using a PerkinElmer Spectrum Two FTIR spectrometer in the wavenumber range of 4000–400 cm^{-1}. The spectra were collected in attenuated total reflectance (ATR) mode with a resolution of 4 cm^{-1}. The surface morphology of the compound was examined using a Tecnai 12 Transmission Electron Microscope (FEI) operated at an accelerating voltage of 120kV. Micrographs were obtained at different magnifications to assess particle distribution and morphology. BET analysis was performed at 77 K using **a** Micromeritics ASAP 2020 surface area and porosity analyzer**.** The samples were degassed at 150 °C under vacuum for 12 h prior to analysis. The surface composition and oxidation states of copper in the compound were analyzed using an ESCALAB 250Xi XPS spectrometer (Thermo Fisher Scientific).

Determination of Point of Zero Charge (PZC)

The PZC was determined by the pH drift method. Briefly, 0.1 M NaCl solution was prepared as the background electrolyte, and its pH was adjusted to values between 2 and 12 using 0.1 M HCl or 0.1 M NaOH. 0.05 g of the Cu^{2+} coordination compound was added to 50 mL of each pH-adjusted solution in conical flasks. The suspensions were equilibrated for 24 h at room temperature under constant stirring. Final pH values were measured using a (Jenway 3510) pH meter, and the PZC was identified as the point where the ΔpH (final pH – initial pH) equaled zero.

$$\Delta pH = pH_f - pH_i = 0 \quad (1)$$

Where pH_f is the final pH and pH_i is the initial pH.

RESULTS AND DISCUSSION

FTIR Analysis

The FTIR spectrum of the Cu^{2+} based coordination compound is shown in Fig. 2. The broad absorption band around 3420 cm^{-1} corresponded to O–H stretching vibrations, which may have come from the coordinated water molecules or surface hydroxyl groups. A strong band near 1630 cm^{-1} is attributed to C=O stretching vibrations, indicating the presence of coordinated carbonyl or carboxylate functionalities. The distinctive absorption at 530 cm^{-1} corresponds to Cu–O stretching, confirming the coordination of Cu^{2+} ions with the ligand framework. The observed shifts compared to free ligand spectra suggest strong metal–ligand interactions, which is consistent with previous reports on Cu-based coordination materials (Lei et al., 2020). Such coordination is critical, as it imparts surface activity and influences adsorption behavior.

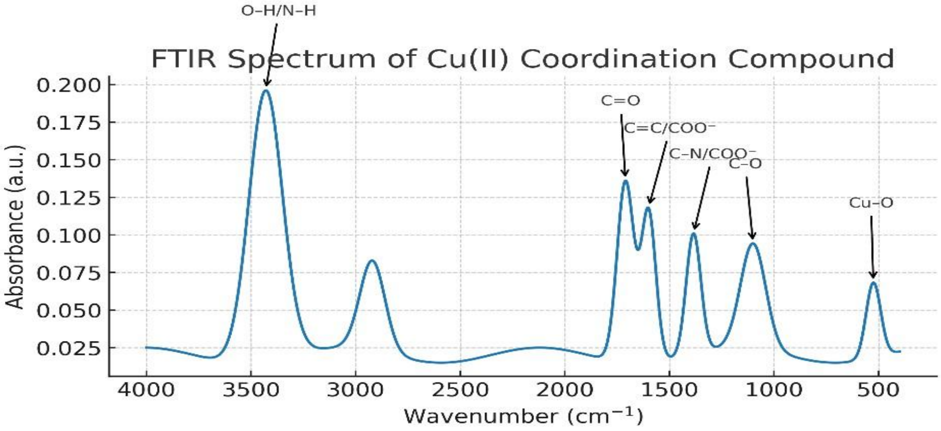

Figure 2: FTIR Spectrum of Cu^{2+} Coordination Compound Showing O–H, C=O, and Cu–O Vibrations

TEM Analysis

The TEM micrographs of the Cu-based coordination compound at varying magnifications is shown in Fig. 3 a–c. The images revealed that the particles in the compound exhibit significant agglomeration, forming large irregular clusters at low magnification (Fig. 3a), while the closer inspection at intermediate magnification (Fig. 3b) showed that these agglomerates are composed of smaller, discrete nanoparticles arranged in a porous network with visible inter-particle voids. At higher magnification (Fig. 3c), the boundaries of the nanoparticles became clearer, with some regions displaying faint lattice fringes, indicating partial crystallinity. This morphology is consistent with other Cu-based adsorbents where irregular nanoparticle shapes and mesoporosity enhance pollutant adsorption (Baruah, 2022).

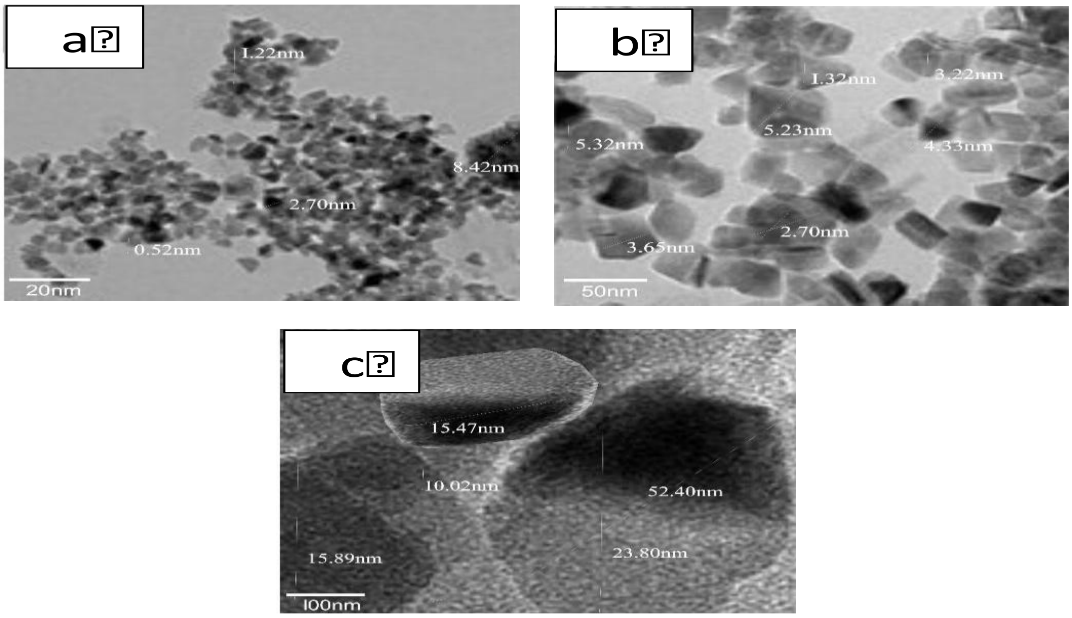

Figure 3: TEM Micrograph of the Cu²⁺ Coordination Compound Illustrating Porous and heterogeneous morphology at (a) 20 nm, (b) 50 nm, and (c) 100 nm.

BET Surface Area Analysis

The BET surface area analysis of the Cu^{2+} based coordination compound shown in Fig. 4 displayed typical characteristic of a mesoporous material. The calculated BET surface area is 72.20 m^2g^{-1}, which is moderate when compared to high surface area materials like MOFs (often > 500 m^2g^{-1}). The surface area may not be exceptionally large, however, it exceeds many natural adsorbents such as untreated clays or zeolites, which often exhibit surface areas below 50 m^2g^{-1} (Wang & Peng, 2010). This suggests that the adsorption efficiency of this compound may not be governed solely by surface area, but also by the presence of chemically active sites, such as hydroxyl and carbonyl groups. This finding is supported by the notion that materials with moderate porosity can still achieve significant adsorption when complemented by favourable surface chemistry (Mane et al., 2024).

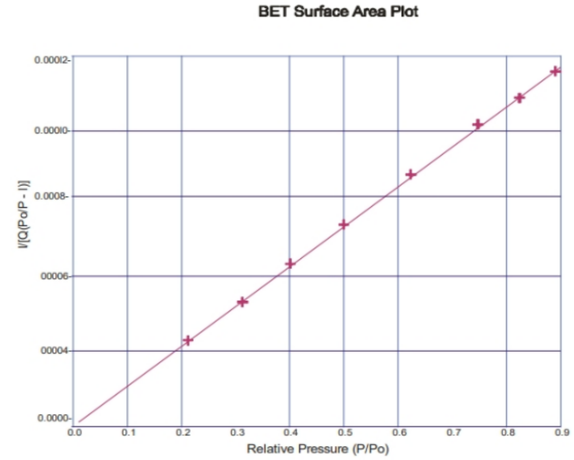

Figure 4: BET Surface Area Analysis of Cu^{2+} Coordination Compound (BET Surface Area = 72.20 m^2g^{-1})

XPS Characterization

The XPS spectra of the Cu^{2+} coordination compound shown in Fig. 5 provided further information on the surface composition and oxidation states of copper in the compound. Peaks at ~933 eV and ~953 eV were assigned to Cu^{2+} species. Additionally, the satellite peaks indicate the coexistence of Cu^{+} states, suggesting a mixed-valence copper environment. Such mixed oxidation states are often associated with enhanced redox activity and electron-transfer capability, which may improve interactions with both cationic and anionic pollutants (Bonthula et al., 2023).

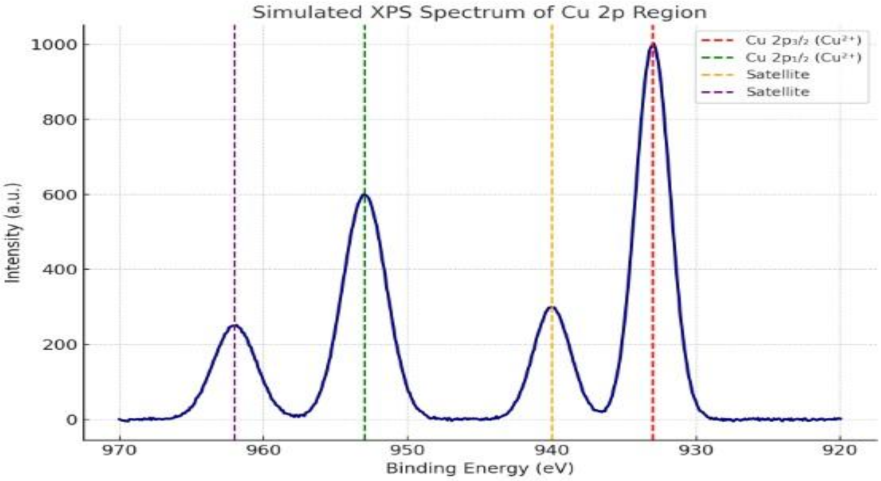

Figure 5: XPS Spectrum of the Cu^{2+} Coordination Compound Showing Mixed Oxidation States of Copper

Point of Zero Charge (PZC)

The PZC of the synthesized Cu^{2+} compound was determined by the pH drift method and found to be pH 5.7 (Fig. 6). At solution pH values below 5.7, the surface carries a net positive charge, favoring the adsorption of anionic species such as dyes and oxyanions. Conversely, at pH values above 5.7, the surface becomes negatively charged, promoting the uptake of cationic pollutants (Baruah, 2022). This dual-charge behavior provides flexibility in adsorption applications depending on the nature of the contaminant. Similar PZC values have been reported for other Cu-based oxides and coordination compounds (Kosmulski, 2009; Sposito, 1998). Thus, the surface charge characteristics of this compound align with established convention for adsorbents, making it a promising candidate for pollutant removal applications (Baruah, 2022).

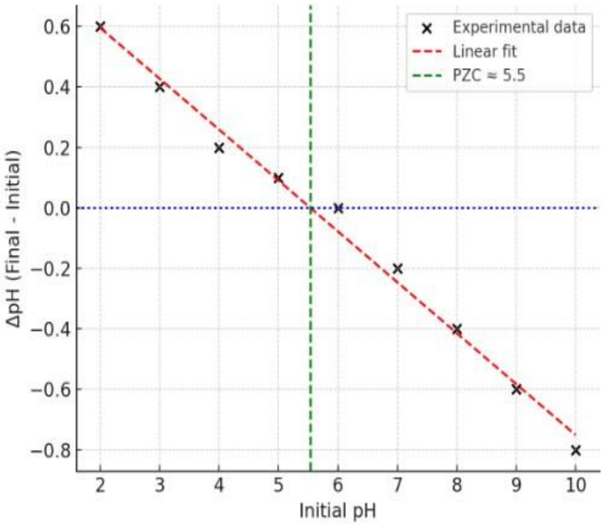

Figure 6: ΔpH Versus Initial pH Curve used to Determine the Point of Zero Charge (PZC = 5.7)

CONCLUSION

The Cu^{2+}-based coordination compound synthesized in this study exhibits promising characteristics for adsorption of pollutants. FTIR analysis confirmed the coordination between Cu^{2+} and the ligand donor groups, while TEM micrographs revealed a porous and heterogeneous morphology favourable to adsorption. BET analysis gave a surface area of 72.20 m^2g^{-1}, indicating moderate porosity. XPS analysis indicated the coexistence of Cu^{2+} and Cu^{+} oxidation states, providing insight into the surface chemistry of the compound. The point of zero charge (PZC) found to be pH 5.7 demonstrates selective adsorption behavior implying that anionic pollutants will be preferentially adsorbed below this pH, while cationic pollutants will be adsorbed above it. These findings suggest that the Cu^{2+}-based coordination compound is a viable candidate for wastewater treatment, with potential applications in removing both anionic and cationic pollutants under different pH conditions. It also suggest that the compound's adsorption efficiency will not only be dependent on surface area but also on its surface chemistry. Future work on its practicality in real-world environmental remediation scenarios is ongoing.

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