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Chitosan Beads as Eco-Friendly Biosorbent for the Biosorption of Au(III) and Cu(II) from Aqueous Solutions

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Abstract
This work aimed to study the biosorption of metal ions by chitosan beads, conducted at room temperature by adding chitosan beads into a conical flask containing different initial pH. The flask was agitated, and the concentrations of metal ions were determined using inductively coupled plasma-optical emission spectrometry (ICP-OES). It was found that the sorption percentage and capacity on the effect of pH for metal ions increased with pH and hit a plateau at pH 3 for single-metal solutions, while binary-metal solutions hit a plateau at pH 2. A consideration separation of Au(III) from Cu(II) could also be achieved at pH 3-5.

Keywords: biosorption; chitosan beads; Au(III); Cu(II)

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1.0 Introduction

Gold and copper consumption are steadily increasing in conjunction with the development of the global economy, and this trend is expected to continue as more people and societies strive to achieve a higher standard of living, which will necessitate access to electricity, sewage systems, and modern appliances. Gold (precious metal) and copper (base metal) are in high demand due to their extraordinary properties, which include high electrical conductivity, malleability, and ductility; excellent corrosion resistance; high catalytic activity; ease of alloying, casting, and recycling; high thermal conductivity and workability; and antibacterial effect (S. H. Chang, 2021). The needs of today's community increased the amount of wastewater while also limiting the supply of gold and copper, driving the focus on recovering gold and copper from metal-containing aqueous solutions.

About 1636 million tonnes of municipal solid waste, including e-waste, are generated annually worldwide, and the amount keeps increasing (Owusu-sekyere et al., 2022). Usually, e-waste generated from used electronic devices and household appliances is dumped in the landfill without treatment because of its complexity and non-biodegradability, which poses severe environmental hazards and health risks. Nevertheless, e-waste contains a high metal content (28 %), such as copper (12.60 %), iron (1.20 %), zinc (5.60 %), nickel (2.41 %), gold (0.0014 %), silver (0.0003 %), and lead (3.10 %), which have rendered it an invaluable secondary source for metal recovery.

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Hence, there has been an increasing interest in recovering these metals, particularly precious metals due to their high economic values, from e-waste.

Among various metal ion recovery methods, adsorption is one of the preferable approaches due to its simplicity, high efficiency, and economic feasibility (Kong et al., 2022). The adsorption process is one of the prevalent methods employed in separation and purification, particularly for removing pollutants from wastewater and industrial product recovery. Adsorption is an accumulation of solutes on the solid adsorbent surface resulting from either electrostatic forces or/and chemical interactions between the solutes and the adsorbents. Various adsorbents that separate and purify pregnant leach solutions include activated carbon and sawdust. However, the performance of these adsorbents is often limited by pre-treatment cost, recycling, disposal method, temperature adjustment, regeneration problem, and high cost. Recently, researchers have embarked on many innovative green initiatives to utilize green materials in metal recovery processes following the paradigm shift towards green growth and sustainable development. In this regard, adsorbents of biological origin, i.e., biosorbents, have drawn significant attention as green adsorbents for metal recovery. Biosorption employs a diverse range of biosorbents derived from natural materials such as activated carbon, zeolite, clay minerals, organic polymers, biological biomass, water treatment residuals, and biochar, all of which play an essential role in the efficient recovery of metals.

Chitosan ((1-4)2-amino-2-deoxy-D-glucose) is a cationic and hydrophilic natural biopolymer composed of long linear chains derived from chitin (1-4)-linked, 2-amino-2-deoxy—D-glucopyranose with varying degrees of alkaline deacetylation. The presence of cation polyelectrolyte nature of reactive hydroxyl (-OH) & amino (NH_2) groups with higher adsorption capacity of chitosan-based biosorbents have proven it to be an effective metal ion recovery method and are gaining popularity as a biosorbent. These are due to its physicochemical properties, excellent chelation performance, high reactivity, high selectivity towards metal ions, high hydrophilicity, flexible polymer chain, availability of numerous adsorption sites, cost-effectiveness, eco-friendliness, inexhaustible, non-hazardous, operational ease, and reusable nature (Vakili et al., 2019). It is a biodegradable, biocompatible, and non-toxic biopolymer derived from the deacetylation of chitin and available in different forms like powder and flake (S. H. Chang, 2021). Most researchers had transformed chitosan in powder or flake form into beads to increase its porosity and surface area, as well as to reduce its crystallinity, before using it to adsorb metal ions such as Al(III), Co(II), Fe(II) and Fe(III). To our knowledge, no prior studies have explored the sorption efficiency and selectivity of Au(III) and Cu(II) sorption by chitosan beads. Since previous studies have shown that metal sorption by chitosan beads is a pH-dependent process, this work aimed to investigate the effect of pH on the sorption efficiency and selectivity of Au(III) and Cu(II) and determine the optimum pH for further studies.

Nomenclature

Au(III)	Gold(III) ion
Al(III)	Aluminum(III) ion
Cu(II)	Copper(II) ion
Co(II)	Cobalt(II) ion
Fe(II)	Iron(II) ion
Fe(III)	Iron(III) ion
NH_2	Amino groups
- NH^{3+}	Ammonia
- OH	Hydroxyl groups
v/v	Volume to volume ratio
h	Hour
rpm	Rotation per minute
ICP-OES	Inductively coupled plasma-optical emission spectrometry
HCl	Hydrochloric acid
NaOH	Sodium hydroxide
C_o	Initial concentration of metal ion (mg/L)
C_e	Equilibrium concentration of metal ion (mg/L)
V	Volume of solution (L)
m	Mass of adsorbent dry weight (g)
q_e	Sorption capacity (mg/g)
%sorption	Percentage of adsorption (%)

2.0 Literature Review

2.1 Conventional recovery methods for Au(III) and Cu(II) from aqueous solution

The enormous amount of leachate carrying assorted metal ions was generated from industry. The leachate-containing metals will either be discharged into the environment through industrial effluents to surface water or deposited as sludge to soil. Hence, the metals tend to accumulate in living organisms besides threatening human health and the environment since they are non-biodegradable. Currently, the most conventional methods of removing metals include ion exchange, membrane filtration, chemical precipitation, and electrochemical removal. Nevertheless, the deficiency of these methods was due to their restriction of low efficiency, high operational cost, high energy needed, incomplete removal, sensitive operating condition (Burakov et al., 2018), as well as production of toxic sludge throughout the process that require additional treatment (Kim et al., 2018).

Table 1 shows conventional methods with different efficiency in recovering metal ions, yet they suffer some drawbacks. Most conventional methods can recover different types of heavy metals up to 90%. Nevertheless, all of the approaches have their benefits and weakness that become the limitation for the whole process of achieving high metal ion recovery. For instance, the ion exchange method is susceptible to the solution pH, which can cause fouling of resin with solid contaminants in the wastewater. Besides, maintenance and operation costs are high due to high energy consumption.

Table 1: Comparison of Main Conventional Methods Use for Heavy Metal Recovery from E-Waste

Method	Process description	Advantages and Disadvantages	References
Ion Exchange (Up to 80% recovery)	Ions present in the aqueous solution exchange with other ions either on the surface or through the interior of the solid ion exchange resin	**Advantages:** High treatment capacity and high removal efficiency **Disadvantages:** Weak binding affinity and very sensitive to pH	(; Lebron et al., 2021)
Membrane filtration (84% - 99.99% recovery)	Separating suspended particulate from soluble components and solution with different sizes and characteristics by using pressure to carry the solution through a porous structure of semi-permeable membrane	**Advantages:** Need small space to operate and less production of solid waste **Disadvantages:** Clog of membrane and high energy demand	(Crini & Lichtfouse, 2019)
Chemical precipitation (Up to 90% recovery)	Addition of precipitant reagent to precipitate chemical from the dissolved substance in wastewater resulting in the formation of an insoluble compound	**Advantages:** High selectivity level and low capital cost **Disadvantages:** High amount of precipitate agent and operational cost higher for sludge disposal	(Abdullah et al., 2019)
Electrochemical removal (Up to 97% recovery)	Electricity is applied to allow current to pass through an aqueous metal solution and create an electric field between electrodes comprising the insoluble anode	**Advantages:** Electric field migration allows fast removal for low concentration heavy metals and can treat metal ions multiple times at one time **Disadvantages:** High equipment cost and need for coagulants and salt flocculants	(Crini & Lichtfouse, 2019; C. Liu et al., 2019)
Solvent extraction (Up to 98% recovery)	The solvent extraction progress through the following stages: i. The solvent penetrates the aqueous matrix ii. The solute is diffused out of the aqueous matrix iii. The extracted solutes are collected	**Advantages:** Able to handle desired pH and high resistance towards an aqueous solution **Disadvantages:** Less selective and other chemical reactions can occur	(Abdul Halim et al., 2019)
Adsorption (Up to 99% recovery)	In the mass transfer process, the material is transferred directly to the surface of the solid phase from the liquid phase.	**Advantages:** High efficiency and ease of regeneration **Disadvantages:** Highly dependent on the solution environment	(Bui et al., 2020; M. Zhao et al., 2020)

2.2 Recovery of Au(III) and Cu(II) by various biosorbents

Chitosan is a biopolymer-based adsorbent that plays a significant role in adsorption as it is biocompatible and reusable. Nevertheless, the employment of chitosan is limited because it is insoluble in an aqueous solution but only soluble in acidic media. One of the chitosan modification techniques preferred is blending the chitosan with ionic liquid where the chitosan pillar can be regulated through chitosan modification to obtain high adsorption efficiency and improve mechanical stability via the introduction of different functional groups that are prone to combine with amine groups of chitosan matrix (Wang & Zhuang, 2022). These are due to the specific physicochemical characteristics, excellent chelation performance, high reactivity, high metal ions selectivity, high hydrophilicity, more adsorption sites available, and flexible polymer chain structure of chitosan-based biosorbent (Vakili et al., 2019).

Table 2 below shows a variety of ionic liquids utilized to recover metals. Several ionic liquids have been applied as an alternative extractant for chitosan modification through the physical modification method. Different types of ionic liquid were utilized under different conditions, including pH, temperature, agitation speed, contact time, initial concentration, and adsorbent dosage, to obtain the highest metal recovery. Over other solvents that are used in the extraction process, incorporating ionic liquid as an environmental-friendly solvent offers several advantages such as solvent power, potentiality in adjusting solubility depends on the option of either cation or anion to enhance transport properties, provide ion exchange interactions, and improve metal ion adsorption through effective electrostatic interaction.

Table 2: Chitosan-based Biosorbent for Metal Recovery from Aqueous Solution

Chitosan-based biosorbent	Condition	Adsorption capacity ($mg.g^{-1}$)	References
Poly aniline modified chitosan embedded with ZnO-Fe_3O_4	pH = 6.5 Contact time = 51 minutes Temperature = 31°C Initial concentration = 82 mg/L	328.4	(Kavosi Rakati et al., 2019)
Chitosan impregnated with 1-ethyl-3-methyl imidazolium chloride (Aqueous solution)	pH = 2.5-2.8 Contact time = 1.5 hours Temperature = 25°C Initial concentration = 20 mg/L	2.45 1.44	(Lupa et al., 2018)
$MnFe_3O_4$/chitosan	pH = 2 – 9 Contact time = 7 minutes Temperature = 15 – 40°C Initial concentration = 10 – 250 mg/L	62.30	(Zhang et al., 2020)
Magnetic chitosan / sodium alginate gel beads	pH = 3 – 5 Contact time = 180 minutes Temperature = 25 – 35°C Initial concentration = 80 mg/L	124.5	(Shuo et al., 2018)

2.3 Chitosan sorption on Au(III) and Cu(II)

Au(III) sorption by N-carboxymethyl chitosan found that the maximum adsorption is at pH 4 to pH 6. From pH 4 to pH 6, it is recognized that chitosan amine groups can serve as electron donors, and it shows chelating potential against several metal ions (Vedula & Yadav, 2021). In the amine groups, the nitrogen electrons present will create bonds with transition metal ions to allow sorption to happen. The reaction between Cu(II) and chitosan favored the protonation of the amine groups to form $-NH^{3+}$ at lower solution pH values (Mallik et al., 2022). As more NH_2 transformed to $-NH^{3+}$, there were only fewer NH_2 sites usable for Cu adsorption on the surface of the beads. This results in a decrease of Cu(II) adsorption on the beads. Figure 1 shows the chemical structure of chitosan. The existence of cation polyelectrolyte nature of reactive hydroxyl (-OH) & amino (NH_2) groups with more adsorption capacity than chitosan-based biosorbents have proven it as an effective metal ion recovery (Mallik et al., 2022). Chitosan is insoluble in alkaline solution, having the degree of deacetylation less than 50% and molecular weight more than 5 kDa. However, chitosan is pH sensitive, and it is soluble in dilute organic acids.

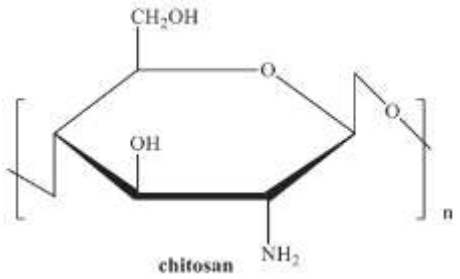

Figure 1: Chemical structure of chitosan

3.0 Methodology

3.1 Material

Copper (II) chloride dihydrate ($CuCl_2.2H_2O$) (R&M >98% purity), sodium chloride (NaCl) (QRec, >99.8 purity), gold(III) chloride trihydrate ($HAuCl_4.3H_2O$) (Sigma-Aldrich, ≥99% purity), hydrochloric acid (HCl) (QRec 37% purity) (Fisher Scientific ≥ 37% purity), sodium hydroxide (NaOH) (Qrec ≥ 99%), ammonia solution (NH_4OH, R&M Chemicals, 30% purity), methanol (CH_3OH) (QRec, 99.8% purity), chitosan (Sigma Aldrich, >75% deacetylated), and acetic acid (QRec).

3.2 Synthesis of chitosan beads

Chitosan solution was prepared by dissolving 3.0 g of chitosan powder in 100 mL of 1 % (v/v) acetic acid solution. The viscous solution was left stirred for 24 hours at room temperature to ensure complete dissolution of chitosan powder and form chitosan gel like solution. The solution was then added drop wise using a dropper into a precipitation bath containing methanol/ammonia (9:1 v/v). The chitosan beads were then filtered and rinsed with distilled water to remove any methanol or ammonia residue. The chitosan beads were filtered, rinse and finally oven-dried at 50°C for 24 hours (Youcefi et al., 2022).

3.3 Batch adsorption experiment

The batch adsorption experiments were conducted in conical flasks containing 0.6 g of chitosan beads in 120 mL of single- or binary-metal solutions. The initial metal concentration was kept at 25 mg/L, and the pH values of the solutions were adjusted to 1, 2, 3, 4, and 5 with 1 M HCl or 1 M NaOH. The flasks were agitated for 5 h on an orbital shaker (SK-300, JEIO TECH) at 150 rpm. Upon completion of the agitation, the solutions were filtered with filter papers, and the filtrates were analyzed by an inductively coupled plasma-optical emission spectrometry (ICP-OES) (Optima 7000 DV, PerkinElmer). The sorption percentage (%sorption) and equilibrium sorption capacity (q_e, mg/g) of chitosan beads were calculated by Equations (1) and (2) (Ayub et al., 2020):

$$\%\ sorption = \frac{C_o - C_e}{C_o} \times 100\% \quad (1)$$

$$q_e = \frac{\left(C_o - C_e\right)V}{m} \quad (2)$$

Where C_o and C_e are the initial concentration and the equilibrium concentration of metal ion aqueous solution (mg/L), respectively, V is the volume of the solution (L), and m is the dry weight of biosorbent (g).

4.0 Findings and Discussion

4.1 Sorption of Au(III) and Cu(II) from single-metal solutions

Since chitosan beads are pH-sensitive sorbents, as reported by Zeng et al. (Zeng et al., 2022), the effects of initial pH on the adsorption of Au(III) and Cu(II) were studied by chitosan beads. Fig. 2. shows the percentage of sorption by chitosan beads at different initial pH of Au(III) and Cu(II) in single metal ion solutions. Effects of pH on the sorption of Au(III) and Cu(II) were studied at an initial concentration of 25 mg/L, and the sorption experiments were performed within 5 hours. As can be seen from Fig. 2, Au(III) sorption in single metal ion solutions at pH 1 was at the lowest and started to increase at pH 2, then plateaued from pH 3 to 5. The least Au(III) metal ion sorption at pH 1 was due to the competition of chlorine ion of the acid and gold species in a very acidic condition, thus reducing the adsorption efficiency (S. H. Chang, 2021). For Cu(II), the trend was slightly similar to the sorption trend of Au(III). However, pH 2 of Cu(II) sorption (83.7%) showed an increment of chitosan beads sorption performances by 36% compared to Au(III) adsorption (53.0%). Thus, the best pH for Cu(II) and Au(III) metal ion solution adsorption was at pH of 3, with sorption of 99.5% and 98.4%, respectively.

Fig. 3. shows the equilibrium sorption capacity for Au(III) in single metal ion solutions on chitosan beads. It reveals a consistent trend with that reported by Zhao et al., 2020 in their previous work. There is a slight increase in capacity after pH 2 for both Au(III) and Cu(III) ion sorption by the chitosan beads. The highest sorption capacity of Au(III) in single metal ion solutions is at pH 3, with a value of 5.09. The lowest sorption capacity is at pH 1, with a value of 0.02 by chitosan beads. In the sorption by Gibbsite 400 (Yang et al., 2020), the sorption capacity values for both pH 3 and 1 were less than 2. For Cu(II) ion, the highest sorption capacity is at pH 3 (5.04), the lowest is at pH 1 (0.36), and sorption by phosphogypsum reported zero sorption capacity for both pH 1 and 3 (L. Zhao et al., 2020).

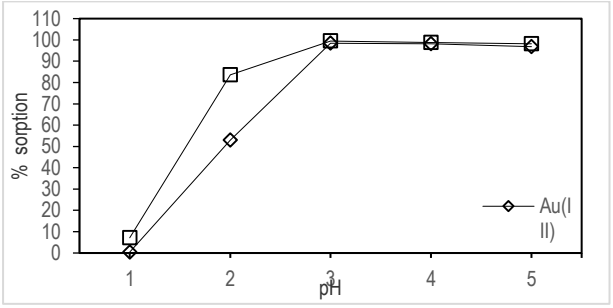

Fig. 2: Effect of initial pH on Au(III) and Cu(II) sorption in single metal ion solutions.

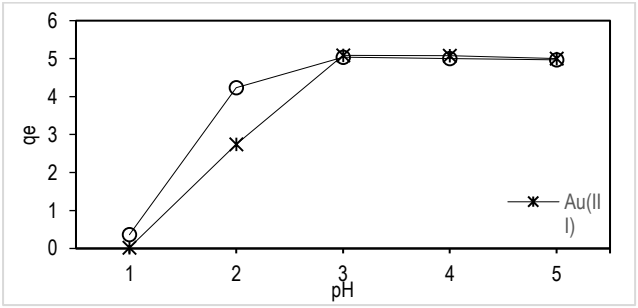

Fig. 3: Equilibrium sorption capacity of Au(III) and Cu(II) in single metal ion solutions.

4.2 Sorption of Au(III) and Cu(II) from binary-metal solutions

Fig. 4. shows the percentage of sorption by chitosan beads at different initial pH of Au(III) and Cu(II) in binary metal ion solution. The effect of pH on the adsorption of Au(III) and Cu(II) was studied at an initial concentration of 25 mg/L for each metal ion, and the adsorption was performed for 5 hours. The trend of binary solution shows an almost similar adsorption trend as a single metal ion solution reaction. From Fig. 4., Au(III) sorption in binary metal ion solution exhibits the highest percentage of adsorption (99.4%) compared to Cu(II) metal ion (78.4%) at pH 4. Kavosi Rakati et al. (Kavosi Rakati et al., 2019) reported similar findings, where the adsorption efficiency of Au(III) metal ions strongly increased from pH 1 to 2. This occurrence demonstrated that the pH of the solution has a significant impact on Au(III) recovery. According to literature, $AuCl_4^-$ is the main substance of Au(III) at pH < 5.0. The species of $AuCl_4^-$ varies with pH. When the pH is lower than 5.0, the decrease of the sorption amount of gold ions is due to the competition between $AuCl_4^-$ and Cl^- for sorption site and hydrogen ion (H^+) preferentially combine with $AuCl_4^-$.

Fig. 5. shows the competition of Au(III) and Cu(II) on chitosan beads in binary metal ion solution at an initial concentration of 25 mg/L for each metal ion for 5 hours. It shows that the selectivity value towards Au(III) over Cu(II) is more than 1. From Fig. 5., Au(III) and Cu(II) sorption capacity plateau from pH 3 to 5. However, Au(III) sorption capacity is highest at pH 4 with a value of 7.86, and Cu(II) shows the lowest sorption capacity at pH of 1 by 0.80 from Au(III).

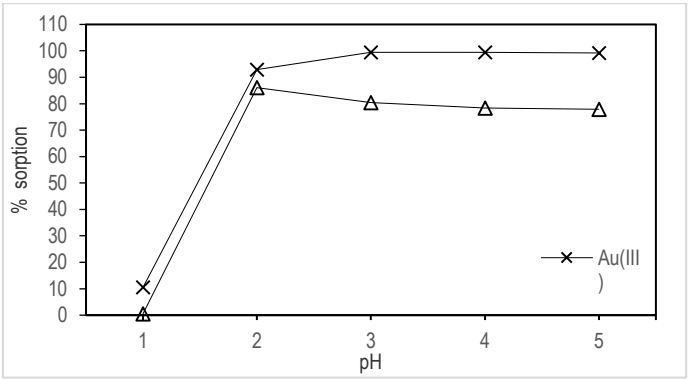

Fig. 4: Effect of initial pH on Au(III) and Cu(II) sorption in binary metal ion solutions.

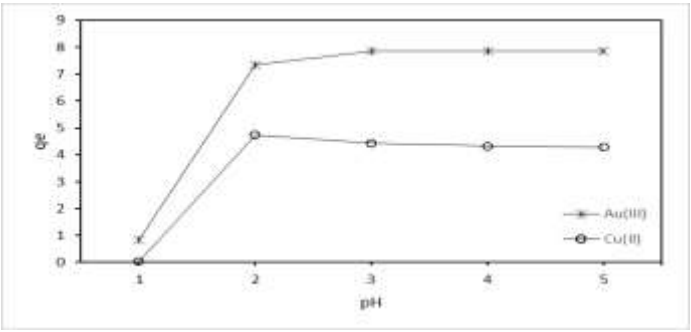

Fig. 5: Equilibrium sorption capacity of Au(III) and Cu(II) in binary metal ion solutions.

5.0 Conclusion and Recommendations

The biosorption of Au(III) and Cu(II) in single- and binary-metal solutions by chitosan beads was optimum at pH 3 to 5, with more than 78% sorption. The sorption capacities of chitosan beads for Au(III) and Cu(II) were 5.09 and 5.04, respectively. Results of the binary-metal solutions revealed that considerable separation of Au(III) from Cu(II) could be achieved from pH 3 to 5. Following this experiment, we suggest further research into modifying chitosan-based adsorbents to be more selective towards gold over copper across the initial pH range.

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Paper Contribution to Related Field of Study

Following the paradigm shift towards green growth and sustainable development, researchers have embarked on green and eco-friendly materials in metal recovery processes. The findings are expected to benefit public health and the environment in line with the National Key Priority Area of Water Security and the Environmental Quality Act 1974.

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