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## Research Article

### Preparation and Alkaline Activation of Cellulose Extracted from *Salacca zalacca* Midrib for $[\text{AuCl}_4]^-$ Adsorption

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## ABSTRACT

Cellulose from salacca (*Salacca zalacca*) midrib waste shows potential as an adsorbent for metal ions. In this study, cellulose was extracted through drying, grinding, delignification with 5% NaOH, and alkaline activation, followed by Fourier Transform Infrared (FTIR) characterization. Adsorption experiments were conducted by contacting 5 g of activated cellulose with 30 mL of 50 ppm  $[\text{AuCl}_4]^-$  solution at pH 1–4. The remaining Au concentration was measured using AAS to determine adsorption efficiency. The results show very high adsorption percentages: 99.70% (pH 1), 99.28% (pH 2), 97.98% (pH 3), and 97.72% (pH 4). Pearson correlation analysis yielded a significance value of 0.034 and a correlation coefficient of 0.966, indicating that pH influences adsorption and that adsorption efficiency decreases slightly with increasing pH. Overall, alkaline-activated cellulose from salacca midrib is highly effective for Au(III) adsorption, particularly under strongly acidic conditions.

**Keywords:** Cellulose, Salacca Midrib, Alkaline Activation,  $[\text{AuCl}_4]^-$  Adsorption, pH Effect

## 1. INTRODUCTION

Gold is widely utilized in various industrial applications, including electronics, catalysis, and biomedical devices. The increasing global demand for gold has consequently led to a rise in industrial wastewater containing gold ions, particularly Au(III) in the form of tetrachloroaurate  $[\text{AuCl}_4]^-$ . The presence of  $[\text{AuCl}_4]^-$  in aquatic environments not only poses potential environmental concerns but also represents the loss of a valuable resource if not efficiently recovered. Therefore, the development of effective, economical, and environmentally sustainable methods for noble-metal recovery is essential, including biomass-based adsorption approaches that provide greener alternatives to conventional chemical extraction methods [1–3].

Adsorption has emerged as one of the most promising techniques for metal removal due to its operational simplicity, low cost, and high selectivity toward specific ions [4,5]. In recent years, considerable attention has been given to the use of lignocellulosic biomass as a sustainable and low-cost adsorbent alternative. Lignocellulosic materials are mainly composed of cellulose, hemicellulose, and lignin, with cellulose being the most abundant fraction. Cellulose possesses advantageous characteristics such as mechanical strength, biodegradability, and reactive hydroxyl groups that allow interaction with metal ions [6-8]. However, although cellulose inherently contains functional groups capable of binding metal ions, its native structure often exhibits limited porosity, surface area, and accessibility of active sites, which restricts its adsorption capacity—thus motivating the need for performance-enhancing treatments.

Chemical modification is often applied to enhance the adsorption performance of cellulose [9-10]. Alkaline activation, in particular, can increase porosity, expand surface area, and alter functional groups, thereby improving the material's ability to interact with metal ions. This treatment also helps remove residual lignin and hemicellulose, resulting in cellulose with higher purity and enhanced adsorption capability [11]. Accordingly, introducing alkaline activation becomes essential to overcome the inherent limitations of unmodified cellulose and to unlock its full adsorption potential. Consequently, alkaline-activated cellulose has the potential to serve as an efficient adsorbent for  $[\text{AuCl}_4]^-$  ions.

*Salacca zalacca* (snake fruit) midrib is an abundant agricultural waste in Indonesia that remains underutilized despite its high lignocellulosic content [12]. Converting this biomass into functional cellulose not only aligns with circular-economy principles and agricultural waste reduction but also provides opportunities for producing a value-added, locally sourced adsorbent material [13]. To date, studies utilizing cellulose extracted from *Salacca zalacca* midrib, particularly for noble-metal ion adsorption remain scarce. Previous works mainly focus on its general lignocellulosic properties or non-metal adsorption applications, leaving a research gap regarding its structural suitability, adsorption performance, and modification strategies for gold recovery. This limitation forms the bottleneck addressed in the present work and highlights the novelty of applying alkaline-activated *Salacca* cellulose for  $[\text{AuCl}_4]^-$  adsorption.

Based on these considerations, this study aims to extract cellulose from *Salacca zalacca* midrib, perform alkaline activation, and evaluate its adsorption performance toward  $[\text{AuCl}_4]^-$  ions under various pH conditions. The study includes Fourier Transform Infrared (FTIR) spectroscopy to observe functional-group changes and statistical analysis to determine the influence of pH on adsorption effectiveness. The findings are expected to provide scientific insight into the potential of salacca midrib as a sustainable biomass-based adsorbent for gold recovery and to enrich the limited body of literature on modified tropical-biomass cellulose for noble-metal adsorption.

## 2. EXPERIMENTAL SECTION

This section describes the materials, instruments, and experimental procedures employed in this study. All experimental steps were conducted systematically to ensure reproducibility and accuracy of the results.

### 2.1. Materials

The materials used in this study included salacca midrib petiole obtained from Turi, Sleman Regency, Yogyakarta; sodium hydroxide solutions (5% and 10% NaOH), solid NaOH, hydrochloric acid (HCl), and an aqueous  $[\text{AuCl}_4]^-$  solution (50 ppm, Sentra Chemical). Additional materials such as phenolphthalein indicator, filter paper, and distilled water were also utilized. All reagents were used as received without further purification.

### 2.2. Instrumentation

The laboratory equipment used in this study included beakers of various capacities (50, 250, and 500 mL), a 10 mL volumetric pipette, glass funnels, both magnetic and glass stirrers, a vacuum pump, mortar and pestle, a hot-plate stirrer, thermometer, aluminum foil, an analytical balance, a drying oven, measuring cylinders, a pH meter, and standard filtration apparatus. The analytical measurements were conducted using a Shimadzu IRPrestige-21 FTIR spectrophotometer, which operates in the wavenumber range of  $4000\text{--}400\text{ cm}^{-1}$  and is equipped with a high-sensitivity DTGS detector and a KBr beam splitter suitable for transmission measurements using KBr pellets. Elemental analysis of Au ions was performed using an Atomic Absorption Spectrophotometer (AAS) equipped with a hollow-cathode lamp for Au determination and a standard air–acetylene flame atomization system. All instruments were calibrated in accordance with standard laboratory procedures prior to use.

## 2.3. Procedure

### 2.3.1. Sample Preparation

Salak petioles were cleaned, chopped, and dried under sunlight followed by oven drying at 60°C. The dried petioles were cut into smaller pieces and ground using a blender to obtain fine powder. The powder was then sieved using a 60-mesh sieve shaker. The resulting powder was oven-dried again at 60°C for 1 hour to remove excess moisture.

### 2.3.2. Delignification

A total of 25 g of salacca midrib powder was soaked in 500 mL of a 5% NaOH solution for 24 hours to remove lignin. After soaking, the material was thoroughly washed with distilled water until a neutral pH was reached. The neutralized sample was then dried in an oven at 60 °C.

### 2.3.3. Sample Characterization

The cellulose derived from salacca midrib was characterized using FTIR spectroscopy. Solid samples were mixed with KBr to form transparent pellets, and the FTIR spectra were recorded over the wavenumber range of 4000–400 cm<sup>-1</sup>. The analyzed samples included raw salacca midrib cellulose, alkali-activated cellulose, and cellulose after adsorption of [AuCl<sub>4</sub>]<sup>-</sup>.

### 2.3.4. Adsorption of [AuCl<sub>4</sub>]<sup>-</sup>

#### 2.3.4.1. pH Variation Study

A total of 5 g of cellulose was mixed with 30 mL of a 50 ppm [AuCl<sub>4</sub>]<sup>-</sup> solution. Prior to adsorption, the solution pH was adjusted to 1, 2, 3, and 4. After the adsorption process, the mixture was filtered using filter paper, and the filtrate was subsequently analyzed by AAS to determine the remaining Au ion concentration.

#### 2.3.4.2. AAS Analysis

Gold ion concentrations were quantified using an Atomic Absorption Spectrophotometer. Residual Au concentrations in the filtrate were determined through interpolation of a standard calibration curve. The amount of adsorbed Au was calculated as the difference between the initial concentration and the residual Au concentration.

#### 2.3.4.3. Statistical Analysis

The influence of pH on [AuCl<sub>4</sub>]<sup>-</sup> adsorption by cellulose was evaluated using Pearson correlation analysis. Statistical processing was performed with SPSS version 25 to assess the relationship between pH variation and adsorption efficiency.

### 3. RESULTS AND DISCUSSION

#### 3.1. Characteristics of Salacca Midrib Cellulose

Fourier-transform infrared (FTIR) spectroscopy was employed to assess the chemical transformation of Salacca midrib biomass following NaOH-mediated delignification. The spectral changes confirm the selective removal of lignin and hemicellulose, yielding a cellulose-enriched material with surface functionality conducive to the adsorption of tetrachloroaurate  $[\text{AuCl}_4]^-$  ions, an important consideration for gold recovery from electronic or mining waste streams.

In the untreated Salacca midrib, a broad absorption band centered at  $3340\text{ cm}^{-1}$  was observed (Figure 1), assigned to O–H stretching vibrations from hydroxyl groups in lignin, hemicellulose, and cellulose [14]. After NaOH treatment, this band shifted to  $3218\text{ cm}^{-1}$  and exhibited reduced intensity as shown in Figure 1, indicating partial deprotonation and loss of non-cellulosic hydroxyl groups. This modification is consistent with lignin solubilization under alkaline conditions and enhances the exposure of cellulose hydroxyls, which can serve as coordination sites for metal complexes under controlled pH [15].

A prominent peak at  $1728\text{ cm}^{-1}$  in the raw biomass attributed to C=O stretching in ester and carboxylic acid functionalities of hemicellulose and lignin side chains [16] disappeared after NaOH activation. This confirms the saponification of ester linkages, a key mechanistic step in alkaline delignification that effectively removes acetyl and uronic acid groups from hemicellulose [17]. The elimination of such acidic functionalities reduces surface charge competition and may improve selectivity toward anionic gold species in acidic media.

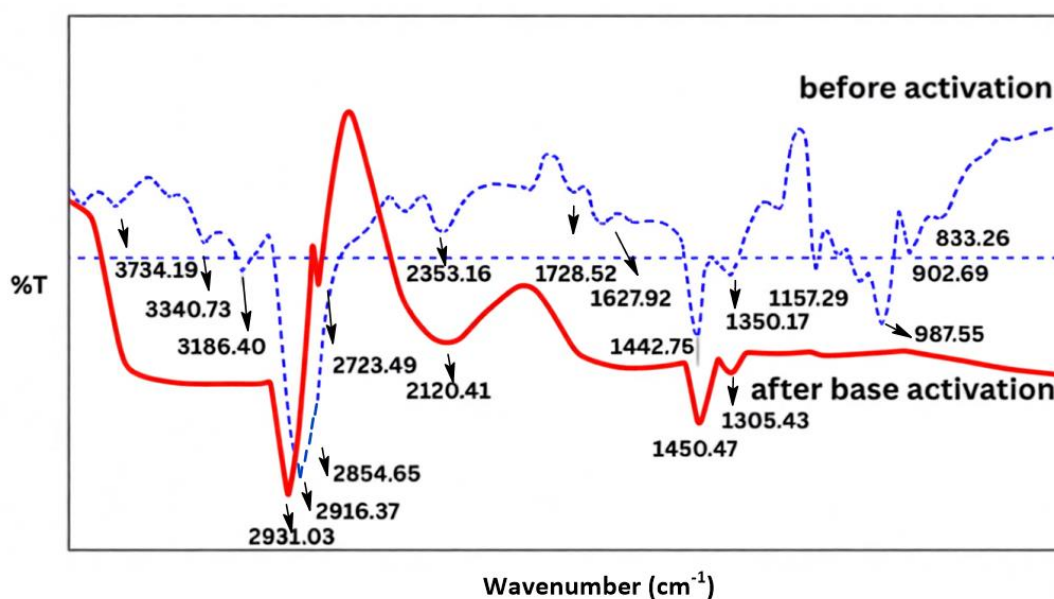
The aromatic skeletal vibrations of lignin, typically detected at  $1600\text{--}1510\text{ cm}^{-1}$  (e.g.,  $1628\text{ cm}^{-1}$  and  $1512\text{ cm}^{-1}$  in untreated biomass), were significantly attenuated post-treatment. This reflects the cleavage of  $\beta\text{-O-4}$  ether bonds and solubilization of phenylpropane units under alkaline conditions [18]. While a faint band near  $1600\text{ cm}^{-1}$  persists, suggesting trace residual lignin, the overall spectral profile aligns with successful delignification, as reported in other lignocellulosic systems treated with 5–10% NaOH [19].

Additionally, aliphatic C–H stretching vibrations at  $2931\text{ cm}^{-1}$  and  $2855\text{ cm}^{-1}$ , associated with methyl/methylene groups in lignin and hemicellulose, markedly diminished after treatment, further corroborating the extraction of non-cellulosic [20]. Concurrently, cellulose-characteristic bands, such as the C–O–C glycosidic bond vibration at  $1160\text{ cm}^{-1}$  and C–O

stretching between 1030–1060  $\text{cm}^{-1}$  became relatively more pronounced, indicating cellulose enrichment [21].

The resulting delignified *Salacca* midrib is thus a cellulose-rich biopolymer with enhanced surface homogeneity and accessible hydroxyl groups. Although native cellulose surfaces are negatively charged above pH 3, the adsorption of anionic  $[\text{AuCl}_4]^-$  can be facilitated under highly acidic conditions (pH 1–2), where protonation of surface  $-\text{OH}$  groups generates positively charged sites capable of electrostatic attraction. Moreover, the absence of competing lignin-derived phenolics minimizes unwanted redox reactions that could prematurely reduce  $\text{Au(III)}$  to metallic  $\text{Au(0)}$ , thereby preserving the gold complex for controlled recovery [22].

These findings position delignified *Salacca* midrib as a sustainable, low-cost precursor for gold adsorbents. Its performance can be further optimized via mild functionalization (e.g., quaternary ammonium grafting), but even in its native post-delignification state, it offers structural and chemical attributes aligned with effective anionic metal complex capture.

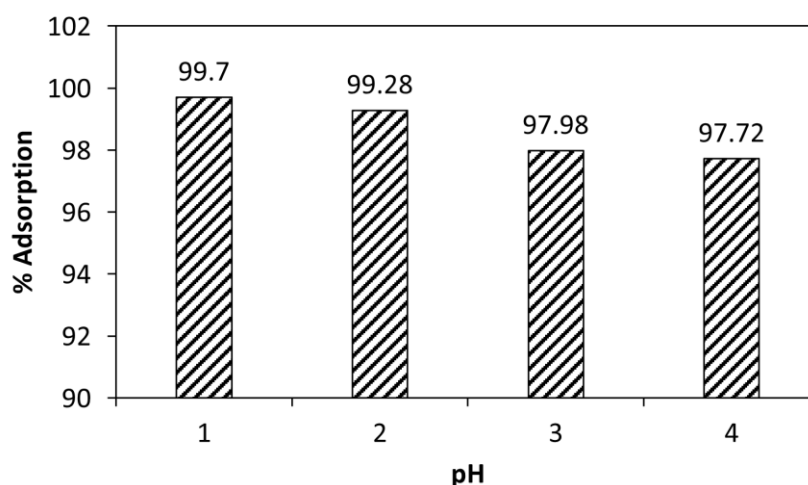


**Figure 1. FTIR spectra of *Salacca* midrib biomass before and after base activation with NaOH**

### 3.2. Effect of pH on $[\text{AuCl}_4]^-$ Adsorption

The ability of delignified cellulose from *Salacca* midrib to adsorb  $[\text{AuCl}_4]^-$  ions was evaluated over a pH range of 1–4, as shown in Figure 2. The results demonstrate that this material achieves exceptionally high adsorption efficiency (>97%) under acidic conditions, with optimal performance at pH 1 (99.70%) and pH 2 (99.28%). A slight but consistent

decrease in adsorption efficiency occurs as the pH increases to 3 (97.98%) and 4 (97.72%), indicating strong pH dependence of the adsorption process.



**Figure 2. The adsorption efficiency of  $[\text{AuCl}_4]^-$  onto NaOH-activated Salacca midrib**

This behavior aligns with the central hypothesis of this study: that NaOH treatment effectively delignifies Salacca midrib, yielding a cellulose-rich material with a surface abundant in hydroxyl ( $-\text{OH}$ ) groups. Under highly acidic conditions ( $\text{pH} \leq 2$ ), these  $-\text{OH}$  groups undergo partial protonation to form positively charged sites ( $-\text{OH}_2^+$ ), enabling electrostatic attraction toward the anionic  $[\text{AuCl}_4]^-$  species [23]. Furthermore, the high concentration of  $\text{H}^+$  suppresses surface deprotonation, minimizing electrostatic repulsion between the adsorbent and the gold complex.

Conversely, at higher pH values ( $\geq 3$ ), hydroxyl groups tend to deprotonate, rendering the cellulose surface increasingly negatively charged. This generates electrostatic repulsion against  $[\text{AuCl}_4]^-$ , directly reducing adsorption affinity (Zhao et al., 2019). Additionally, above pH 2, gold species in chloride media may undergo hydrolysis to form less adsorbable hydroxo-complexes such as  $[\text{Au}(\text{OH})_4]^-$  or  $\text{Au}(\text{OH})_3$ , which exhibit lower compatibility with the hydrophilic cellulose surface [24].

Notably, the delignified cellulose maintains adsorption efficiency above 97% even at pH 4, a remarkable performance compared to many conventional lignocellulosic adsorbents. This is likely attributed to the success of the delignification process, which not only enhances cellulose purity but also exposes more reactive  $-\text{OH}$  groups and improves surface accessibility, as confirmed by FTIR analysis (Section 3.1). Residual lignin, if not removed, could otherwise interfere with adsorption by promoting uncontrolled reduction ( $\text{Au}^{3+} \rightarrow \text{Au}^0$ ) or blocking active sites.

Thus, these findings strongly support the initial hypothesis that NaOH-mediated delignification of Salacca midrib produces high-purity cellulose suitable as an adsorbent for anionic precious metal complexes. The high affinity for  $[\text{AuCl}_4]^-$  under acidic conditions highlights its potential for gold recovery from electronic waste leachates or acidic hydrometallurgical streams.

### 3.3. Adsorption Mechanism

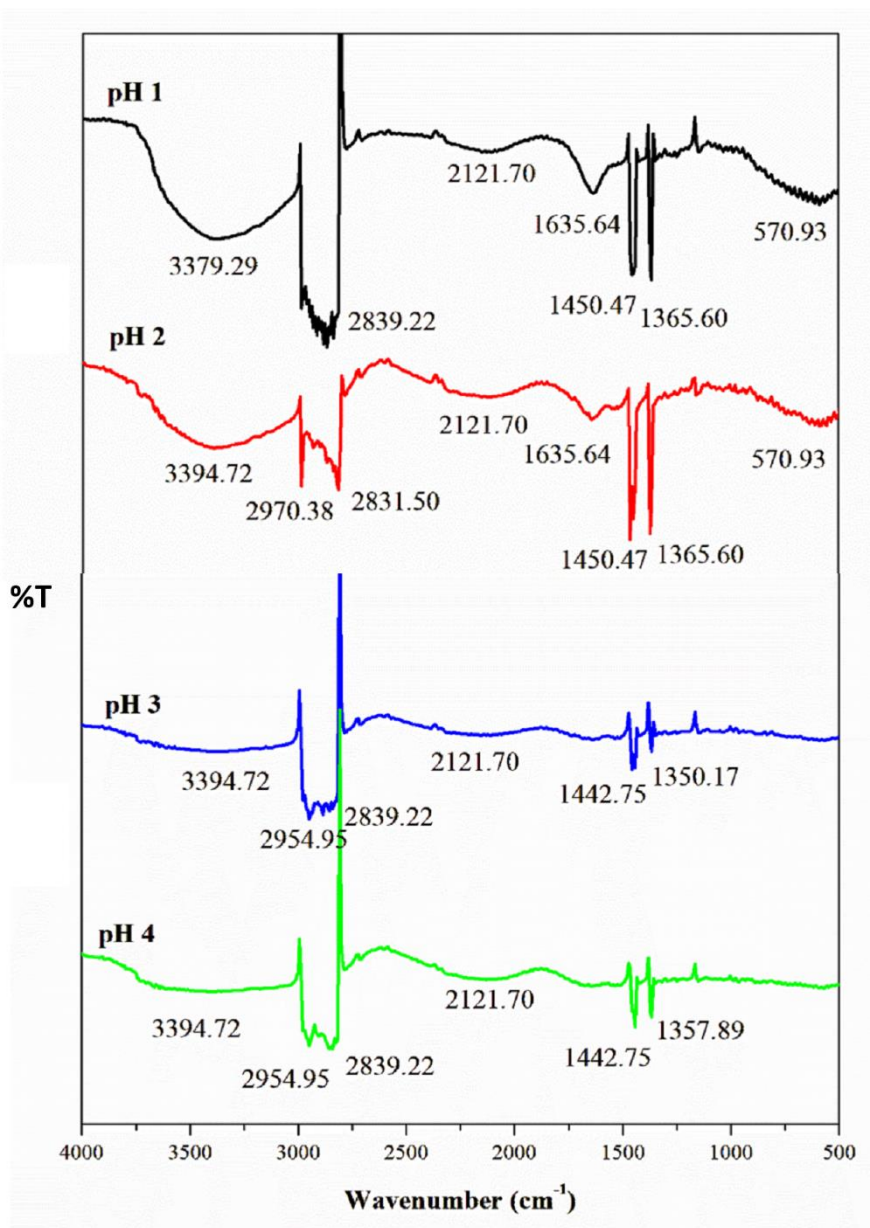
FTIR analysis of delignified Salacca midrib cellulose after  $[\text{AuCl}_4]^-$  adsorption at various pH values (Figure 3) provides important insights into the nature of the interaction between the adsorbent surface and gold species. Overall, no new peaks appeared in the post-adsorption spectra that would indicate the formation of covalent bonds between gold and the cellulose surface. This suggests that the adsorption process does not involve permanent chemical reactions but is instead dominated by reversible physical interactions. Nevertheless, subtle shifts and intensity changes in several characteristic peaks reflect modifications in the electronic environment of surface functional groups due to the proximity of  $[\text{AuCl}_4]^-$  ions.

One of the most noticeable changes occurred in the O–H stretching region around  $3394\text{ cm}^{-1}$ . In samples used for adsorption at pH 1 and 2, this band appeared slightly broader and less intense compared to the spectrum of the pristine adsorbent. This phenomenon indicates that hydroxyl groups on the cellulose surface are involved in interactions with  $[\text{AuCl}_4]^-$  ions, likely through hydrogen bonding between the oxygen of surface –OH groups and the chloride ligands of the gold complex, or through electrostatic attraction if the –OH groups are protonated ( $-\text{OH}_2^+$ ). In contrast, at pH 3 and 4—where adsorption efficiency slightly decreased, the O–H band became sharper, reflecting lower protonation levels and weaker interactions, consistent with a pH-dependent adsorption mechanism.

The C–H stretching region ( $2839\text{--}2955\text{ cm}^{-1}$ ), originating from the aliphatic backbone of cellulose, remained largely unchanged after adsorption in both peak position and intensity. This confirms that the cellulose carbon framework does not directly participate in gold binding, thereby ruling out significant hydrophobic interactions or reactions involving the polysaccharide main chain. In the fingerprint region ( $1500\text{--}1000\text{ cm}^{-1}$ ), minor shifts were observed near  $1450\text{ cm}^{-1}$  (C–H bending), and a subtle feature emerged around  $1350\text{--}1365\text{ cm}^{-1}$  in all post-adsorption samples. These changes may result from perturbations in C–O–C glycosidic bond vibrations due to the electrostatic field of adsorbed  $[\text{AuCl}_4]^-$  ions, or, in some



cases, could reflect weak Au–Cl vibrational modes though this region is typically difficult to interpret without complementary techniques such as XPS.



**Figure 3. FTIR spectra of delignified *Salacca* midrib cellulose after  $[\text{AuCl}_4]^-$  adsorption at different pH values (pH 1–4)**

Overall, the absence of significant new peaks particularly below  $600\text{ cm}^{-1}$ , where metal–oxygen or metal–chloride vibrations typically appear strongly supports the conclusion that  $[\text{AuCl}_4]^-$  adsorption onto delignified cellulose is physical rather than chemical in nature. The most plausible mechanism is outer-sphere surface complexation, wherein  $[\text{AuCl}_4]^-$  remains hydrated and binds to the surface via electrostatic attraction to positively charged sites generated by protonation of hydroxyl groups under acidic conditions. This interaction is

further enhanced by hydrogen bonding between chloride ligands and surface –OH groups. This mechanism explains why adsorption efficiency is highest at pH 1–2 and gradually declines as pH increases, and it also accounts for the adsorbent's structural stability and potential for regeneration. Thus, delignified cellulose from *Salacca midrib* proves to be an efficient, stable, and potentially regenerable biosorbent for gold recovery from acidic solutions.

### 3.4. Statistical Correlation Between pH and Adsorption Efficiency

To quantitatively assess the relationship between solution pH and the adsorption efficiency of  $[\text{AuCl}_4]^-$  onto NaOH-activated *Salacca midrib* cellulose, Pearson's correlation analysis was performed. The data satisfied the prerequisite assumptions for parametric correlation testing, including normality and linearity, thereby justifying the use of Pearson's method.

The analysis yielded a Pearson correlation coefficient ( $r$ ) of  $-0.966$  with a significance level ( $p$ -value) of  $0.034$ . Since the  $p$ -value ( $0.034$ ) is less than the significance threshold ( $\alpha = 0.050$ ), the correlation is statistically significant. The negative sign of the coefficient indicates an inverse relationship: as pH increases, adsorption efficiency decreases. Furthermore, the magnitude of  $r = -0.966$  suggests a very strong negative linear correlation between pH and  $[\text{AuCl}_4]^-$  adsorption efficiency.

This result statistically corroborates the experimental observation that the NaOH-activated cellulose exhibits optimal gold uptake under highly acidic conditions (pH 1–2), with a gradual decline in performance as the solution becomes less acidic. The strong inverse correlation underscores the critical role of pH in modulating surface charge and speciation of gold complexes, thereby directly governing adsorption behavior.

## 4. CONCLUSION

This study confirms that cellulose isolated from *Salacca zalacca* midrib waste via NaOH delignification and alkaline activation acts as a highly effective biosorbent for  $[\text{AuCl}_4]^-$  ions. The adsorbent exhibits excellent performance, achieving 99.70% removal at pH 1 and sustaining efficiencies above 97% up to pH 4. The statistically significant and strongly negative correlation between pH and adsorption efficiency underscores the critical role of surface hydroxyl group protonation in facilitating the capture of anionic gold complexes.

The conversion of agricultural biomass into a functional adsorbent underscores its potential as a cost-effective and environmentally benign material for gold recovery applications. Nevertheless, as the adsorption tests were performed using synthetic solutions, future work should include evaluations using real waste matrices (e.g., electronic waste leachates or mining effluents) to assess selectivity, robustness, and real-world feasibility. Such investigations will also enable meaningful comparison with commercial adsorbents and recently reported biosorbent systems, providing a clearer outlook on the competitiveness and practical deployment of this material.

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## **AUTHOR CONTRIBUTIONS**

Mujahidul Mubaraq designed and conducted the experimental work, performed the data analysis, and prepared the initial dataset. Maya Rahmayanti contributed to the conceptual framing and interpretation of results. Both authors jointly wrote the manuscript and critically reviewed its content, ensuring scientific accuracy and coherence. All authors have read and approved the final version of the manuscript.

## **CONFLICT OF INTEREST**

The authors declare no conflict of interest related to the publication of this manuscript. There are no financial, personal, or professional relationships that could be perceived as influencing the impartiality of the research presented herein.

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