

# **SORPTION OF HEAVY METAL IONS ( $\text{Cu}^{2+}$ and $\text{Cd}^{2+}$ ) FROM SIMULATED AND INDUSTRIAL WASTEWATERS USING SUGARCANE BIOMASS**

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## **ABSTRACT**

The widespread use of heavy metals in different manufacturing processes has resulted in their presence in the air, soil and aquatic ecosystems. Many manufacturing processes release their effluents into nearby streams and rivers due to non-availability of waste treatment technologies in the developing world. Industrialization has therefore been reported to be a major anthropogenic source of these elements and their compounds waterbodies. Conventional methods that are currently being used in the developed world (such as chemical precipitation, ultra-filtration, reverse osmosis, etc) are expensive and so, unaffordable to small-scale businesses prevalent in developing economies. The need for alternative, cheaper and available technologies therefore, cannot be over-emphasized. This study attempted to remove heavy metals ( $\text{Cu}^{2+}$  and  $\text{Cd}^{2+}$ ) from simulated and industrial (paint and textile) wastewaters using sugarcane biomass. A synthetic resin (Chelex) was used as control. Batch sorption experiments were conducted to assess the influence of contact time, adsorbent dose, adsorbate concentration, pH, agitation speed, temperature and particle size. Increases in the parameters (except particle size) gave corresponding increases in percentage adsorption of both metals. Optimal temperature for adsorption was achieved at 50°C above which adsorption declined considerably. Experimental data for  $\text{Cu}^{2+}$  and  $\text{Cd}^{2+}$  fitted well into freundlich and langmuir isotherms. In textile effluent, 100% adsorption was achieved for both metals using sugarcane biomass in contrast to 89% and 91% for  $\text{Cu}^{2+}$  and  $\text{Cd}^{2+}$  respectively in chelex, a synthetic resin. Similarly, adsorption was 100% for both metals using the biosorbent. Sugarcane biomass may therefore be utilized for water and wastewater remediation in the developing world. Its biodegradability gives comparative advantage over alternative synthetic and expensive resins since its disposal will be more environmentally friendly.

## **KEYWORDS**

Copper; cadmium; sugarcane biomass; wastewaters; remediation.

## **1. INTRODUCTION**

Increasing shortage of freshwater resources due to global climatic changes poses greater demand on water supplies. Water needs for domestic, industrial and recreational purposes however increases by day worldwide. The need for remediation and reuse of large volumes of industrial wastewaters being generated globally therefore becomes imperative. Pollution of

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the various environmental compartments (water, soil, and air) is an important consequence of industrial processes and human activity. Metals are naturally distributed in the environment both by geological and biological cycles. The metal contamination of the environment reflects both natural sources and the contribution from industrial activity [1]. Heavy metals released by a number of industrial processes are some of the major pollutants in soil and water resources [2]. Main industrial sources of pollution include electroplating, battery and mining industries discharging a variety of toxic metals such as lead, copper, nickel, zinc and cadmium [3, 4]. Heavy metals are toxic and environmentally harmful substances; they have accumulating characteristics in nature as they cannot be degraded. Accumulation of these metals in human body causes brain, skin, pancreas and heart diseases [4, 5]. Heavy metal pollution is therefore, a serious problem and this has led to the development of new and improved methods for treating wastewaters [6].

Conventional methods for removal from waters include electrodialysis, chemical precipitation, ultrafiltration, reverse osmosis, etc. [3,7,8]. Technical and economic constraints often pose limitations on the industrial applicability of these methods especially in developing countries [6, 9, 10]. Adsorption, ion exchange, chemical settling and reverse osmosis are the most frequently preferred methods. Among them, adsorption receives considerable interest with high efficiency in heavy metal removal [4]. Different materials have been utilized for heavy metals removal from wastewaters. They include bacteria [1], algae [3], clay materials [4, 11, 12], chitosan [13] and animal bi-products [14]. Cellulosic materials that have been used include wheat bran [2], pine sawdust [5], sour orange [6], sugar beet pulp [15] and maize cob [16].

Sugar cane is a highly versatile plant, and can be grown successfully under a wide range of conditions. The by-products of sugar manufacture (molasses, bagasse and filter mud) are used in many ways including food as well as the basic raw material for burgeoning the sucro-chemical industry [17]. In most developing countries of Africa, sugarcane fibre is usually discarded and presently, there is no known use of this waste material in this part of the globe. The objective of this work therefore is to investigate the adsorption potential of sugarcane biomass for heavy metal ions ( $\text{Cu}^{2+}$  and  $\text{Cd}^{2+}$ ) removal from aqueous matrices, its desorption and re-use potentials.

## **2. MATERIALS AND METHODS**

### **2.1 Reagents, biomass preparation, effluent collection and instrumentation**

Deionized water was used to prepare all solutions and all chemicals used were of analytical grade (Merck, Germany). The laboratory glassware was kept overnight in a 5% (v/v) nitric acid solution. Afterwards, it was rinsed thoroughly with deionized water and dried. Working solutions in the range of 5 – 200 mg/L were prepared daily by diluting corresponding stock solutions (1000 mg/L of  $\text{Cu}^{2+}$  or  $\text{Cd}^{2+}$ ). The synthetic resin used was Chelex (Bio-Rad, France) France; batch samples of sugar cane fibre were collected as waste materials from dumps. The fibres were cut into smaller sizes, thoroughly washed, first with tap water and then, with distilled deionized water and then oven-dried at 90°C for 24 h. The dried samples were then ground using a wooden mortar, sieved using 100, 200, 500 and 2000 mesh screens. These particle size fractions of the biomass were washed twice with 0.01 M HCl to remove any metals or debris that might be in the biomass prior to experimental metal ion exposure. The acid washed biomass samples were again washed twice with distilled- deionized water to remove acid and then, oven-dried at 70°C for 48 h. This pretreated biomass was then stored in plastic containers at room temperature for further experimentation [18].

Batch samples of paint and textile effluents were collected into clean plastic bottles from points of discharge at Ikeja Industrial Estate, Lagos – Nigeria. Effluents pH values were measured using a pH meter (Jenway Ltd., Dunmow Essex, England, model 3015). Metal ion concentrations were analysed using Alpha 4 Atomic Absorption Spectrophotometer (Chemtech Analytical, UK) with hollow cathode lamps. The operating conditions of the AAS instrument for metals' ions analyses (copper and cadmium) were 3.0, 0.5, 324.7 and 3.0, 0.5, 228.8 for HC lamp current (mA), Slit width (nm) and Wavelength (nm) respectively.

## **2.2 Experimental run**

### **Adsorption Experiments**

Equilibrium conditions of adsorbents weight (capacity studies), contact time (kinetics), pH and metals' concentrations (equilibrium isotherm), temperature, particle size and agitation speed were studied using respective metal standard solutions and industrial (textile and paint) effluents [16].

### **Kinetic Studies**

Kinetic parameters for the adsorption process were studied on batch adsorption of 100 mg/L solutions at 32<sup>o</sup>C. Six sets of a mixture of sugarcane biomass (0.4 g) in 25 mL of 100 mg/L metal ion in 100 mL conical flasks were allowed to stand at different time intervals of 30, 60, 90, 120, 150 and 180 min respectively for adsorption to take place. The mixtures were then filtered to separate the adsorbent and the filtrate to allow for analysis of residual metal ions.

### **Capacity studies**

Different weights of each of the adsorbents (sugarcane biomass and chelex) in the range of 0.025 g and 0.6 g in 25 ml solution were used to remove metals from 100 mg/L standard solutions. A 25 ml aliquot each of the metals' solutions was accurately measured into 100 ml conical flask and the adsorbent added to the flask; a contact time of 2 h was maintained for all the experiments. After 2 h contact time, the mixture was filtered through Whatman No 1 Filter paper (medium flow rate and porosity with particle retention of 11µm) and the filtrate analysed for residual metal by AAS.

### **Effect of pH on adsorption capacity**

The pH of 100 mg/L metal solution prepared from 1000 mg/L stock solution was adjusted to desired value and kept constant with the addition of either 0.1 M NaOH or 0.1 M HCl. The pH values investigated were 2, 4, 5, 7, 8 and 10. Sugarcane biomass (0.4 g) aliquot was weighed and added to 25 ml of 100 mg/L metal solution at the desired pH and left for 2 h. The mixture was then filtered and the filtrate analyzed for metal ion concentration.

### **Effect of concentration on adsorption**

Metals standard solutions with concentration range of 5 mg/L and 200 mg/L were prepared from the stock solution. Adsorbent weight of 0.4 g in 25 ml solution was also used for adsorption isotherm studies with a contact period of 2 h (earlier established as equilibrium time or adsorption) after which the mixture was filtered for metals analyses.

### **Effect of shaking speeds**

A 0.4 g aliquot of f sugarcane biomass was weighed into 25 ml solution of 100 mg/L metal ion in a 100 ml conical flask. The mixtures were shaken in an orbital shaker (Edmund Buhler, GmbH & Co., Germany) at speeds in the range of 50 rpm and 300 rpm. After 30 min, the

mixtures were filtered using Whatman No 1 filter paper and filtrates analyzed for residual metal content.

### Effect of Temperature

Metal ion solutions were equilibrated by rocking a mixture of 0.4 g sugarcane biomass and 25ml of 100 mg/L for 30 min at 250 rpm at different temperatures (30°C, 40°C, 50°C, 60°C, 70°C and 80°C in a water bath (Techne Cambridge Ltd, Duxford Cambridge, England). These were centrifuged immediately at 2500 rpm for 5 min and the supernatant decanted. The supernatants were then analyzed for metal concentrations using AAS.

### Effect of Particle Size

Zero point four grammes (0.4 g) of 100, 200, 500 and 2000 mesh adsorbents were weighed and transferred to separate tubes; 25 ml of 100 mg/mL metal solution was then added to each tube and equilibrated by rocking for 30 min at 250 rpm. The mixture was centrifuged at 2500 rpm for 5 min and the supernatant used for the analysis of residual metal ions.

### Adsorption studies on effluents

Textile and paint effluents (pH values 3.93 and 4.56 respectively at 30°C at the point of discharge) were used for the experiment. For each effluent, 0.4 g each of the adsorbents (sugarcane biomass and chelex) were added to 25 ml of the effluent and left standing for 2 h. At the expiration of 2 h, the mixtures were filtered and the filtrates analyzed for metal ions.

## 3. RESULTS AND DISCUSSION

Table 1: Equilibrium concentration of metals onto sugarcane biomass at different time intervals (Mean±SD) n=3

Time (minutes)	Residual metal concentration in solution (mg/L)	
	Cd <sup>2+</sup>	Cu <sup>2+</sup>
30	12.08±0.04	15.02±0.02
60	9.64±0.01	13.94±0.06
90	4.82±0.02	9.35±0.03
120	4.77±0.01	8.02±0.03
150	4.67±0.06	7.67±0.04
180	4.38±0.05	7.41±0.02

Effects of contact time on adsorption of metals are presented in Table 1. Results obtained in this study showed that at pH 5, optimal adsorption was achieved in 2 h for Cd<sup>2+</sup> and 2 h and 30 min for Cu<sup>2+</sup>. Subsequent adsorption experiments were therefore carried out at the optimal contact time for the respective metals unless otherwise stated. The result is consistent with those of Altundogan *et al.* [15] who reported 2-3 h equilibrium time for Cu<sup>2+</sup> sorption when acid and alkaline modified sugar beet pulp was used as adsorbent. Adsorption rate constant (K<sub>ad</sub>), which was calculated using Lagergren's equation (which is the most widely used sorption equation), followed a second order mechanism for sugarcane biomass. Values were comparable to those of coirpith carbon reported by Kadirvelu and Namasivayam [19], Ilhan *et al.* [20] as well as Shukla and Pai [21].

Adsorption capacity of sugarcane biomass is presented in Table 2. The results obtained for capacity studies established that the amount of adsorbent (sugarcane biomass) used is an important factor in metal removal from solution. Adsorption of both metals increased with increasing biomass weight. Optimum adsorption was achieved with 0.4 g sugarcane biomass for both metals. Sugarcane biomass contained cellulose as a major component in its chemical structure. Shukla and Pai [21] reported that cellulose has a two-phase structure, the crystalline compact structure that cannot be penetrated and the amorphous open structure composed of surfaces, external as well as the internal surfaces, which allow the adsorption of heavy metal cations. The pore size of a material plays an important role in deciding the level of penetration of a chemical entity in the amorphous region followed by its adsorption. Effects of metal concentration on adsorption are presented in Figure 1. Obtained trend is consistent with previous results in the literatures [20, 22-28]. These authors all reported increased adsorption with increasing metal ion concentration. Freundlich and Langmuir isotherm models were used to adjust experimental data (Table 3).

Table 2: Equilibrium capacity of sugarcane biomass for metals' removal (Mean  $\pm$ SD) n=3

Weight of residue (g)	Residual metal ions in solution after equilibrium (mg/L)	
	Sugarcane biomass	
	Cd <sup>2+</sup>	Cu <sup>2+</sup>
<b>0.025</b>	15.75 $\pm$ 0.09	17.27 $\pm$ 0.28
<b>0.05</b>	14.66 $\pm$ 0.40	12.35 $\pm$ 0.02
<b>0.1</b>	11.90 $\pm$ 0.01	9.80 $\pm$ 0.02
<b>0.2</b>	7.29 $\pm$ 0.03	5.82 $\pm$ 0.04
<b>0.4</b>	7.16 $\pm$ 0.02	2.28 $\pm$ 0.02
<b>0.6</b>	7.12 $\pm$ 0.01	1.84 $\pm$ 0.04

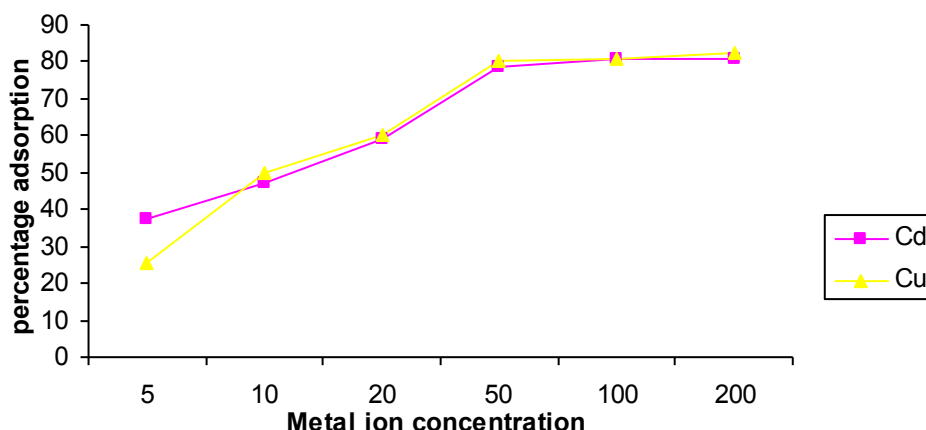


Figure 1. Influence of metal ion concentration on percentage adsorption by sugarcane biomass.

Table 3: Isotherm parameters for metals' adsorption on to sugarcane biomass

Metal	Langmuir			Freundlich		
	$X_m$ (mg/g)	K	$R^2$	$K_f$	n	$R^2$
$Cd^{2+}$	48.91	0.032	0.992	0.70	9.82	1.000
$Cu^{2+}$	35.68	0.029	0.998	0.08	9.72	0.999

The Freundlich model considers the existence of a multilayered structure [26]. Values of  $K_f$  and n, determined from the slope of Freundlich plots were about unity, and this suggests favourable adsorption for the biomass. This agrees with Kadirvelu and Namasivayam [29] who showed that n-values (sorption intensity indicator) between 1 and 10 represent beneficial adsorption. Coefficients of determination were found to be highly positively correlated to sorption ability of residues. Significant adsorption may therefore take place even at high metal concentrations.

In the present study, adsorption was found to be dependent on the pH of adsorption (Figure 2). There was a steady increase in adsorption with increasing pH; the decrease in adsorption at low pH values is due to an increase in competition for adsorption sites by  $H^+$ . Furthermore, adsorption was reported to depend on factors other than pH which include flow rate, resident time, effects of dissolution of some other materials from adsorbents, amongst others. This may explain the continued increase in adsorption of metals by biomass after pH 6 when precipitation of metals is expected to set in. Metals removal by biomass occurs by a combination of physio-sorption and chemisorption. Physio-sorption occurs by trapping of metal ions into the pores of adsorbents while chemisorption is believed to occur at the carboxylic binding sites. However, the exact mechanism of chemisorption of metals onto biomass is not fully understood at very acidic and alkaline pH values [30].

Percentage adsorption was greatly enhanced by shaking and the equilibrium value was attained at 30 minutes (Results not presented). Percentage adsorption of metal ions was

enhanced by shaking both for standard solution and the effluents. In textile effluent, 100% adsorption was achieved in shaken experiments for both metals; adsorption by sugarcane biomass was significantly enhanced by shaking ( $p=0.05$ ). Also in paint effluent, sugarcane biomass had 100% adsorption in shaken experiment.

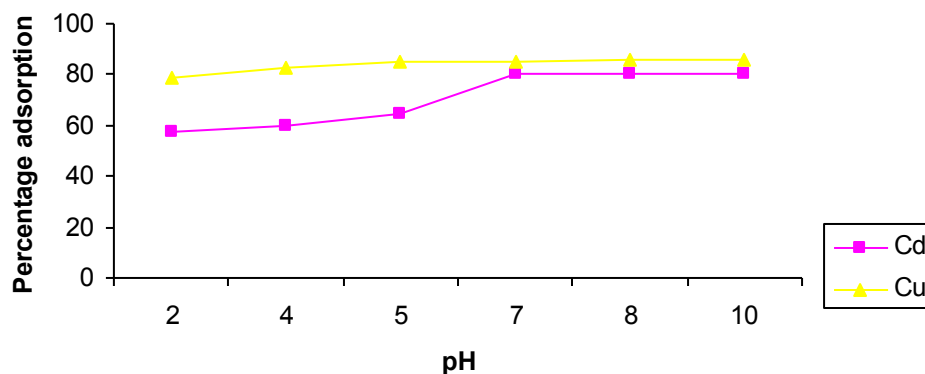


Figure 2. Effect of pH on percentage adsorption of metals by sugarcane biomass

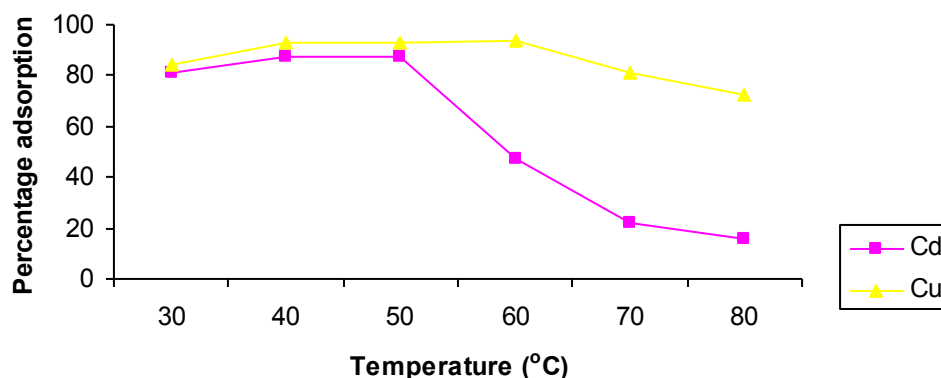


Figure 3. Influence of temperature on percentage adsorption of metal ions by sugarcane biomass.

Optimal temperature for adsorption for both metals was 50°C above which adsorption reduced considerably for  $\text{Cd}^{2+}$ . High temperatures tend to decrease the boundary layer thickness; the metal ions therefore had increased tendency to escape from the biomass surface to the solution phase. The decreased adsorption with increasing temperature also suggests weak adsorption interaction between biomass surface and the metal ion, which supports physisorption [31].

Regression analysis showed that particle density and porosity of adsorbents are important parameters for good adsorption. Particle density and porosity are negatively correlated to percentage adsorption in textile effluent and the former in particular is significant at 95% confidence level. There was enhanced metal adsorption with decreasing particle sizes (Results

not presented). Rate of removal of metals from aqueous solutions has been reported to be particle-diffusion controlled ( $K_p$  is the rate coefficient for particle diffusion control corresponding to the particle size of the adsorbent) [32]. This seems to be due to increase in surface area and exposure of functional groups responsible for metal binding. Results of comparative metals' removal from wastewaters using sugarcane biomass and chelex are presented in Table 4. Under agitation, sugarcane biomass had 100% adsorption for both metals in the two effluent types (Results not presented).

Table 4. Percentage adsorption of metals in textile and paint effluents.

Effluent	$Cd^{2+}$		$Cu^{2+}$	
	Biomass	Chelex	Biomass	Chelex
<b>Textile</b>	94.28	57.10	87.52	61.45
<b>Paint</b>	98.67	53.49	98.89	55.26

#### 4. CONCLUSION

Effects of adsorbent weight, metal concentration, contact time, pH, agitation, temperature, particle size were assessed to determine equilibrium conditions for adsorption. Percentage adsorption of metals was considerable (over 90%) for both metals both textile and paint effluents; comparative assessment of shaken and unshaken experiments revealed that shaking enhanced percentage adsorption for both sugarcane biomass and chelex. The biomass is a better adsorbent for metals since it has greater potential to remove metals from effluents. It also has an added advantage of being biodegradable; hence, disposal will be relatively easier

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