The Simultaneous Removal of Copper and Lead onto Banana Peels (Musa Acuminata) Using A Central Composite Design

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***Abstract*—**The simultaneous removal of Cu^{2+} and Pb^{2+} was studied to determine the effect of the co-existence of the metal ions since it is very rare for wastewater to contain a single metal ion. Also, for industrial-scale treatment of wastewater, the study of the co-existence of metal ions with the operating parameters is important. This study investigated the interactive effects of operating parameters (initial metal concentration, adsorbent dosage, and particle size) using response surface methodology (RSM) with central composite design (CCD) for the prediction of the bio-sorption capacity of banana peels for the binary removal of Cu^{2+} and Pb^{2+} ions from wastewater. The quadratic regression model equations gave a high level of confidence (95 %) with a 5 % significance level and a degree of freedom of 9. The regression models showed that the predicted R^2 is in reasonable agreement with the adjusted R^2 with a difference of less than 0.2. The coefficient of determination R^2 for the percentage removal of Pb^{2+} and Cu^{2+} ions gave 0.995 and 0.9738 respectively. The study contributes to the search for eco-friendly and sustainable solutions to the treatment of water contaminated with low concentrations of Cu^{2+} and Pb^{2+} metal ions.

***Keywords*—**Bio-sorption, Wastewater, Central composite design, Optimization, Response surface methodology

I. Introduction

Among the pollution challenges faced by the global community, environmental pollution of water bodies has become alarming due to its detrimental effects on human health. Water is an essential resource for irrigation, recreation, household use, industrial purposes, and animal use. Clean water is an essential resource needed for human survival. The demand for clean water increases as the population increases which invariably impacts on industrialization and urbanization. Unfortunately, the annual rainfall projection has predicted uncertainty of water accessibility in the nearest future [1]. Therefore, the treatment of wastewater generated from various industrial activities can help to reduce the impact of water scarcity.

Wastewater generated from industrial activities contains different types of pollutants depending on the processes involved. The wastewater generated from food industries is less harmful compared to wastewater from chemical industries. The wastewater contains hazardous or toxic metals generated from industries such as; mining, smelting, battery waste, and electroplating [2]. These toxic metals are non-biodegradable and pose a serious threat to human health when it enters the human system. These heavy/toxic metals include; lead, copper, iron, cadmium, manganese, arsenic, and chromium [3]. Copper and lead are mostly found in wastewater and excessive exposure to these metals can lead to nervous system breakdown, insomnia, liver, and kidney failure [4]. The effects of these metals in water at low concentrations cannot be underestimated; therefore, the Environmental Protection Agency (EPA) has set the permissible limit for copper and lead in wastewater before discharge to 1.3 and 0.05 mg/L respectively [5]. These rules and regulations imposed by the EPA and World Health Organization (WHO) have made it mandatory for industries to treat wastewater to the level at which it is safe for plant, animal, and human beings before discharge.

Various techniques have been explored for wastewater treatment which include; membrane separation (reverse osmosis, ultrafiltration, microfiltration), electrodialysis, coagulation/flocculation, ion exchange, chemical precipitation, and adsorption [6]. All these methods have drawbacks and the common one being the production of large volumes of sludge. However, adsorption technology is simple, easy, cost-effective and produces less sludge hence it has gained the interest of researchers over the past decade. In addition, the use of biodegradable wastes for adsorption also known as biosorption has become a focus area which helps to reduce wastes in circulation and reinforces the concept of a circular economy. Agricultural wastes have been proven to contain high molecular weight compounds such as; hemicellulose, pectin, lignin, carbohydrates, etc. Which make them good adsorbents for adsorption studies [7, 8]. Some of the agricultural wastes that have been used for heavy metal removal include; orange peels [9-11], sawdust, watermelon rind [12], rice husks [13], sugarcane bagasse [14, 15],

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eggshells [16], lentil husks [17], banana peels [18, 19], and apple pomace [20] etc.

In this study, banana peels were used for the simultaneous removal of copper and lead ions in an aqueous solution. The effects of operating parameters such as initial metal concentration, adsorbent dosage and particle size were studied in a batch mode using a binary system of copper and lead ions. Many studies have focused on the interactive effects of operating parameters using a single solute system however, it is rare for wastewater to contain a single metal ion. Therefore, this study explored mathematical software such as design expert (11.1.0.1) and MATLAB (2019a) for the interactive graphical representation of the operating parameters.

II. MATERIALS AND METHOD

A. Preparation of bio-sorbent and adsorbate

Banana peels were collected from household wastes and prepared as discussed previously in [21]. The stock solution containing the required amount of copper and lead nitrate salts ($Cu(NO_3)_2 \cdot 3H_2O$ and $Pb(NO_3)_2$) was prepared in a 1000 mL volumetric flask using deionized water. The solution samples with different initial metal concentrations (10 – 100 ppm) were prepared from the stock solution by serial dilution. The solution pH was measured with the aid of a digital pH meter (edge^{pH} HI 2002, USA) while aqueous H_2SO_4 or NaOH (0.1 M each) were used to adjust the solution pH.

B. Batch experiments

All the batch experiments were conducted at room temperature (270C). The experiments were conducted using a 250 mL conical flask with a 100 mL solution containing a mixture of copper and lead nitrate ions ranging from 10 – 100 ppm initial concentration. The adsorbent dosage and particle size varied from 0.1 – 1 g and 75 – 455 µm respectively. The pH of the solution was maintained at 5.5 being the optimum pH value obtained for a single solute system of copper and lead as stated in our previous studies [19]. The solution was agitated using a linear shaker (Orbital shaker 262, Johannesburg, South Africa) at a speed of 180 rpm and mixing time of 120 min. After the adsorption process, the supernatant portion of the solution was filtered using Whatman filter paper and syringe filters of 0.45 µm. The final concentration of the metal ion in the solution was analyzed using a micro-plasma atomic emission spectrophotometer (MP-AES, MY 18379001, Agilent, Santa Clara, CA, USA). The amount of metal ions adsorbed and the removal efficiency of the metal ions onto banana peels was calculated using Equations (1) and (2), respectively.

$$q = \frac{(C_o - C_e)V}{m} \qquad (1)$$

$$\%\,Removal = \frac{(C_o - C_e)}{C_o} * 100 \qquad (2)$$

Where q (mg/g) is the quantity of metal ions adsorbed, C_o and C_e are the initial and final concentrations in mg/L, V (mL) is the volume of the solution and m (g) is the mass of the adsorbent.

C. Experimental design and analytical methods

The design expert software (V.11.0.5.0, Stat-Ease Inc., Minneapolis, MA, USA) was used to generate the statistical design of the experimental runs. In this study, three operating parameters were selected for the biosorption of Cu^{2+} and Pb^{2+} ions onto banana peels, namely, initial metal ion concentration, adsorbent dosage, and particle size at specified factor levels (Table 1). The pH of the solutions was maintained at 5.5, being the optimized pH value for the single metal system. The CCD generated a total of 20 experimental runs with 3 operating parameters using the face-centered design. In the optimization study, the second-order polynomial equation was used to explain the interactive effects of the independent variables. The quadratic model used to optimize the process variables is shown below.

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1} \sum_{j=i+1} \beta_{ij} X_i X_j + \varepsilon \qquad (3)$$

where Y is the predicted response, X_i and X_j are the independent variables, β_o, β_i, β_{ii}, and β_{ij} are the regression coefficient and ε is the residual error. The interpretation of the experimental results and the significant variables are explained using mathematical functions called the analysis of variance (ANOVA).

TABLE 1: EXPERIMENTAL DESIGN CONDITIONS AND VARIABLE LEVELS USING CCD

Bio-sorbent	**Solution**	**Input variables**	**Coded levels**		
			-1	**0**	**1**
Banana peels	Binary solute Cu^{2+}- Pb^{2+}	X1: Initial concentration (mg/L)	10	55	100
		X2: Adsorbent dosage (g)	0.1	0.55	1
		X3: Particle size (µm)	75	250	455

III. RESULTS AND DISCUSSION

A. Adsorption of Cu^{2+} and Pb^{2+} onto banana peels from a binary solute solution using CCD

The experimental design matrix for the biosorption of Pb^{2+} and Cu^{2+} ions onto banana peels is presented in **Table 2** which shows the experimental data simulated together with corresponding predicted responses. The second-order polynomial (Equation 3) with multiple regression analysis was used to generate the responses (percentage removal of Pb^{2+} and Cu^{2+}) using the three design factors.

TABLE II: CCD EXPERIMENTAL MATRIX AND RESPONSES OF PB^{2+} AND CU^{2+} REMOVAL ONTO BANANA PEELS.

Std	Run	Initial Conc. (mg/L)	Adsorbent dosage (g)	Particle size (µm)	Responses			
					Lead % removal		Copper % removal	
					Exp	Pred	Exp	Pred
15	1	55	0.55	265	86.4	86.36	79.62	80.43
5	2	10	0.1	455	85.05	84.57	60.9	60.58
2	3	100	0.1	75	72.04	71.58	80.19	80.46
17	4	55	0.55	265	86.24	86.36	79.6	80.39
12	5	55	1	265	91.2	91.05	82.4	82.35
4	6	100	1	75	92.1	92.48	86.49	86.72
6	7	100	0.1	455	83.89	83.97	81.58	81.58
20	8	55	0.55	265	86.1	86.36	85.26	81.43
9	9	10	0.55	265	87.65	87.61	84.16	85.27
3	10	10	1	75	91.63	91.46	97.6	97.51
16	11	55	0.55	265	86.68	86.36	79.62	80.39
1	12	10	0.1	75	80.64	80.97	76.01	75.67
19	13	55	0.55	265	87.2	86.36	79.59	80.01
8	14	100	1	455	97.85	97.43	87.21	87.47
18	15	55	0.55	265	86.15	86.32	79.61	80.09
10	16	100	0.55	265	87.4	87.82	91.14	90.38
14	17	55	0.55	455	87.9	88.35	75.12	75.54
13	18	55	0.55	75	84.15	84.08	82.79	82.72
11	19	55	0.1	265	78.54	79.07	68.09	68.49
7	20	10	1	455	87.25	87.62	82.41	82.05

The experimental results obtained were analyzed with the second-order polynomial equation which gave the responses (% Pb^{2+} and % Cu^{2+} removal). The application of the Design Expert software resulted in a quadratic model for the responses which were expressed as a function of the three main independent variables; initial concentration (A), adsorbent dosage (B) and particle size (C), together with three quadratic effects (A^2, B^2, C^2) and three interactive effects (AB, AC, BC). The order of the influence of the interactive effects on the yield of Pb^{2+} removal is AB > AC > BC and AC > BC > AB in the case of percentage removal of Cu^{2+}. The quadratic regression model equations for the % Pb^{2+} and % Cu^{2+} removal are expressed in terms of coded factors as indicated in Equations 5.4 to 5.5 respectively.

$$Y_1(Pb) = 86.36 + 0.106A0 + 5.99B + 2.14C + 2.6AB + 2.2BC - 1.86BC + 1.35A^2 - 1.3B^2 - 0.1495C^2 \quad (4)$$

$$Y_2(Cu) = 80.43 + 2.55A + 6.93B - 3.59C - 3.9AB + 4.05AC - 0.0938BC + 7.39A^2 - 5.01B^2 - 1.3C^2 \quad (5)$$

B. Analysis of variance (ANOVA) for the models

The ANOVA was used to determine the significant variables that fitted well with the regression models as presented in **Table 3** and **Table 4**. The ANOVA helps to identify the relationship between the main, quadratic, and interactive effects and the responses. The models gave a high level of confidence (95 %) with a 5 % significance level and a degree of freedom of 9. The significance of the models was evaluated with the sum of squares, F-values, P-values, adequate precision, and the lack of fit values were all within limits and in reasonable agreement. The P-values of the models gave <0.0001 which shows a good fit between the regression models and the experimental data. In the case of percentage removal of Pb^{2+}, the significant terms with P-values < 0.05 are B, C, AB, AC, BC, $A^{2,}$ and B^2 while A and C^2 are not significant. The lack of fit (LOF) F-value of the model was greater than 0.05 which implies that one or more model terms are not significant. The significant terms for the percentage removal of Cu^{2+} are A, B, C, AB, AC, A^2, and B^2 while BC and C^2 are not significant. Also, the LOF F-value of 0.10 implies the Lack of Fit is not significant relative to the pure error.

Furthermore, the overall performance of the models was evaluated based on the coefficient of determination (R^2) together with the values of the adjusted R^2 and the predicted R^2. All the R^2 values of the models were found to be close to 1, which suggests a reasonable agreement between the data presented with a straight line in **Figure 1**.

The regression models showed that the predicted R^2 is in reasonable agreement with an adjusted R^2 with a difference of less than 0.2. The coefficient of determination R^2 for the percentage removal of Pb^{2+} and Cu^{2+} ions gave 0.995 and 0.9738 respectively.

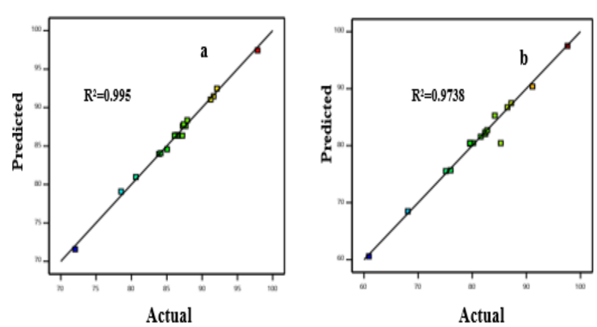

Fig. 1 Predicted versus actual plots (a) Pb^{2+} and (b) Cu^{2+} removal using banana peels.

TABLE III: ANOVA FOR % PB REMOVAL USING BANANA PEELS

Source	Sum of squares	Df	Mean square	F-value	P-value Prob>F	Comments
Pb Model	531.93	9	59.10	222.70	< 0.0001	significant
A-Initial concentration	0.1124	1	0.1124	0.4234	0.5299	
B-Adsorbent dosage	358.44	1	358.44	1350.59	< 0.0001	
C-Particle size	45.71	1	45.71	172.23	< 0.0001	
AB	54.24	1	54.24	204.36	< 0.0001	
AC	38.59	1	38.59	145.40	< 0.0001	
BC	27.71	1	27.71	104.42	< 0.0001	
A^2	5.02	1	5.02	18.90	0.0015	
B^2	4.68	1	4.68	17.63	0.0018	
C^2	0.0615	1	0.0615	0.2317	0.6406	
Residual	2.65	10	0.2654			
Lack of Fit	1.83	5	0.3660	2.22	0.2009	not significant
Pure Error	0.8241	5	0.1648			
Cor Total	534.58	19				
Std. Dev. 0.515	**R^2 0.9950**	**Adjusted R^2 0.9906**	**Predicted R^2 0.9472**	**Adeq. Precision 70.96**	**Mean 86.31**	**C. V. % 0.596**

The tables show the influence of each operating parameter on the adsorption process. The p-value of each parameter contributed greatly to the significance of the model.

TABLE IV: ANOVA FOR % CU REMOVAL USING BANANA PEELS

Source	Sum of squares	Df	Mean square	F-value	P-value Prob>F	Comments
Cu Model	1093.77	9	121.53	41.31	< 0.0001	significant
A-Initial concentration	65.18	1	65.18	22.16	0.0008	
B-Adsorbent dosage	480.80	1	480.80	163.45	< 0.0001	
C-Particle size	128.59	1	128.59	43.72	< 0.0001	
AB	121.45	1	121.45	41.29	< 0.0001	
AC	131.30	1	131.30	44.64	< 0.0001	
BC	0.0703	1	0.0703	0.0239	0.8802	
A^2	150.20	1	150.20	51.06	< 0.0001	
B^2	69.15	1	69.15	23.51	0.0007	
C^2	4.68	1	4.68	1.59	0.2358	
Residual	29.42	10	2.94			
Lack of Fit	2.79	5	0.5588	0.1050	0.9864	not significant
Pure Error	26.62	5	5.32			
Cor Total	1123.18	19				
Std. Dev. 1.72	**R^2 0.9738**	**Adjusted R^2 0.9502**	**Predicted R^2 0.9505**	**Adeq. Precision 30.45**	**Mean 80.97**	**C. V. % 2.12**

C.3D representation of the interactive effects on the responses

The 3D graphical representations of the interactive effects of the operating parameters on the responses are presented in **Figure 2** to **Figure 4**. The plots depict the percentage removal of Pb^{2+} and Cu^{2+} in a binary system with the interaction of the operating parameters. The graphs were obtained with the aid of MATLAB (MATLAB 2019a). **Fig.** represents the interaction between initial concentration and adsorbent dosage with percentage removal of Pb^{2+} and Cu^{2+} ions. The percentage removal of Pb^{2+} and Cu^{2+} increased with increasing adsorbent dosage. This result is reasonable because an increase in adsorbent dosage means an increase in active sites which leads to a corresponding increase in adsorption capacity. The graph depicting the percentage removal of Cu^{2+} showed a saddle point which implies that the highest bio-sorption capacity lies between the maximum and the minimum points. However, increased initial concentration led to an increase in the removal of Cu^{2+} ions.

The interactive effects of initial concentration and particle size on the bio-sorption capacity of Pb^{2+} and Cu^{2+} are shown in Figure 3. The percentage removal of Pb^{2+} increased slightly with an increase in initial concentration while no changes occurred with increasing particle size for Pb^{2+} and Cu^{2+} removal. The percentage removal of Cu^{2+} increased significantly with increasing initial concentration. This shows that initial concentration and adsorbent dosage greatly influenced the bio-sorption efficiency of Cu^{2+}.

The interaction between the adsorbent dosage and the particle size on the bio-sorption efficiency of Pb^{2+} and Cu^{2+} is represented in Figure 4. The percentage removal of Pb^{2+} and Cu^{2+} increased with increasing adsorbent dosage. This result is acceptable because an increase in adsorbent dosage increases the active sites thus enhancing the efficiency of the bio-sorbent. Also, the bio-sorption of Pb^{2+} increased slightly with increasing particle size while particle size had no effect on the biosorption of Cu^{2+}.

Finally, the adsorption efficiency of Pb^{2+} and Cu^{2+} in a binary system onto banana peels was greatly influenced by adsorbent dosage then followed by the initial concentration while the particle size had little or no significant impact on the process.

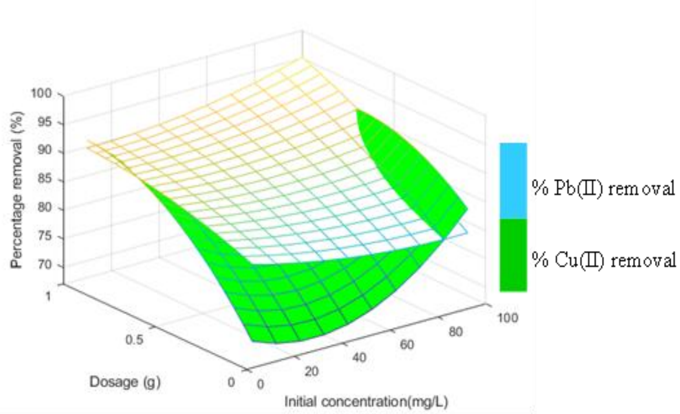

Fig. 2 Effect of initial concentration and dosage on % Pb^{2+} and % Cu^{2+} removal in a binary system onto banana peels.

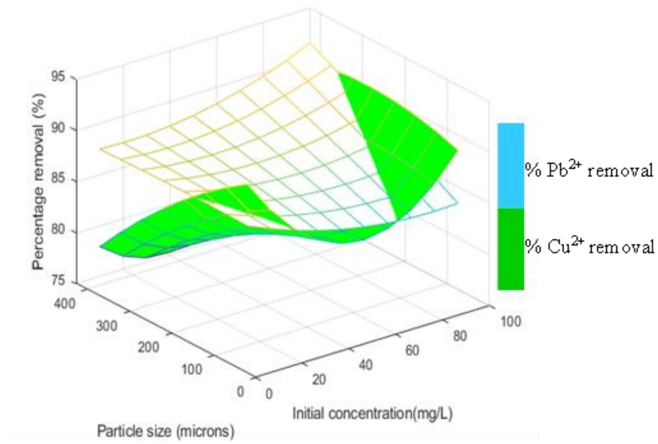

Fig 3 Effect of initial concentration and particle size on % Pb^{2+} and % Cu^{2+} removal in a binary system onto banana peels.

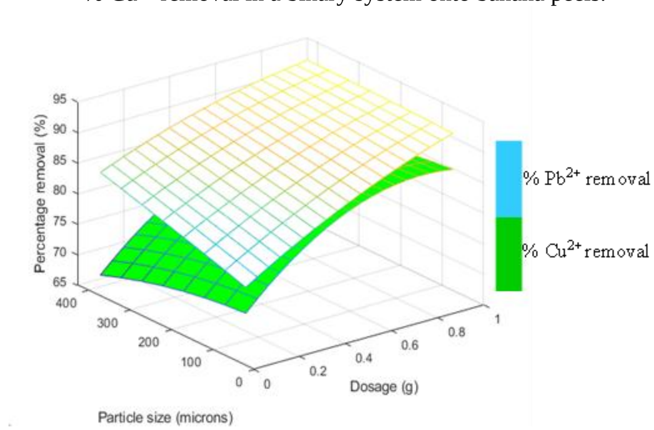

Fig. 4 Effect of adsorbent dosages and particle size on % Pb^{2+} and % Cu^{2+} removal in a binary system onto banana peels.

IV. CONCLUSIONS

The interactive effect of operating variables was studied in a binary system using RSM. The effect of the co-existence of Cu^{2+} and Pb^{2+} ions in solution and the interactive effect with the operating variables showed that Pb^{2+} was more adsorbed than Cu^{2+} which implies that banana peels have a higher affinity for Pb^{2+}. The adsorption process was greatly influenced by the adsorbent dosage and initial metal concentration. The percentage removal increased with an increase in adsorbent dosage. The ANOVA showed that the regression model was significant with a low probability (p-value). The agreement between the experimental and predicted values confirmed the validity of second-order polynomial equations for the biosorption of Cu^{2+} and Pb^{2+} in binary systems using banana peels.

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