Statistical Design Analysis for Adsorption of Cu(II) and Pb (II) onto Kaolinitic Clay.

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ABSTRACT

Clay from Ikpoba in Benin city was tested as low-cost adsorbents for Cu(II) and Pb(II) removal from aqueous solutions. A full factorial experimental design was utilized to assess the effect of four factors on the batch adsorption of Cu(II) and Pb(II) with the clay (particle size < 0.02mm). The chosen experimental factors were adsorbent dose, temperature, shaking speed, and contact time. The results indicated that only adsorbent dose had significant effect for both ions. The best conditions for uptake of ions were respectively time of 90 minutes and 60 at 0.2g adsorbent.

(Keywords: adsorption, statistical design of experiments, low-cost adsorbents, heavy metals, clay)

INTRODUCTION

With the rapid development of modern industries, the environment has faced more and more contamination than in the past (Sheela and Nayaka, 2012). Environmental pollution due to the discharge of heavy metals from various industries, including metal plating, mining, painting, and agricultural sources such as fertilizers and fungicidal sprays are of significant concern because of their toxicity and threat to human life, especially when tolerance levels are exceeded (Patterson, 1985).

A number of treatment methods for the removal of metal ions from aqueous solutions have been reported, mainly reduction, ion exchange, electrodialysis, electrochemical precipitation, evaporation, solvent extraction, reverse osmosis, chemical precipitation and adsorption (Gupta *et al.*, 2009). Most of these methods suffer from drawbacks such as high capital and operational costs or the disposal of the residual metal sludge Due to its simplicity and easy operational conditions, adsorption is a widely-used process (Mabrouk *et al.,* 2009).

Activated carbon has been the most employed adsorbent for heavy metal removal from aqueous solution, but it is however, expensive (Okieimen, 2008). Many reports have appeared on the development of low-cost activated carbon adsorbents developed from cheaper and readily available materials (Okieimen *et al.,* 1991; Badel and Kurniawan, 2003; Bailey *et al*., 1999; Pollard *et al.*, 1993).

Adsorption is dependent on various factors: pH, adsorbent content, initial adsorptive concentration, contact time, temperature, particle size, and ionic strength. The interaction among factors has been scarcely considered. In this respect, a statistical experimental design can be employed to determine the effects of operational variables and their interactions and to optimize the adsorption process, avoiding the traditional one-factor-at-a-time experiments (Carmona *et al*., 2005; Huang *et al*., 2008).

The design determines which factors have important effects on a response as well as how the effect of one factor varies with the level of the other factors (Carmona *et al*., 2005; Montgomery, 2001; Box *et al*., 1978; Brasil *et al*., 2005; Pavan *et al*., 2007; Annadurai *et al*., 2002). The determination of factor interactions could only be attained using statistical designs of experiments (Box *et al*., 1978; Montgomery, 2001), since it cannot be observed when the system optimization is carried-out by varying just one factor at the time and fixing the others. Response surface methods are often employed after a screening of important factors.

In this study, Ikpoba clay was used as an adsorbent for Cu(II) and Pb(II) from aqueous solutions. A 2^4 factorial design was applied to investigate the individual and interactive effects of the adsorbent dose, shaking speed, contact time and temperature on the uptake. The most important factor affecting the heavy metal was also determined.

EXPERIMENTAL

Collection and Preparation of Adsorbents

Clay samples were collected from the clay deposits at Ikpoba River in Benin; and at depths of up to 10 cm with the aid of a plastic shovel and digger and hand-picked to minimize the possibility of contamination. About 4.0 kg of sample was collected and placed in small polythene bag and then dried at 80^oC for 24h, pulverized and sieved to geometric mean size of <0.02 mm before analysis.

Preparation of Aqueous Solution

The salts used for the preparation of the aqueous solutions were: Lead (II) Nitrate and Copper (II) Sulphate. Other chemicals used were: sodium hydroxide and hydrochloric acid. All reagents used were of analytical grade. Distilled water was used for preparing stock solutions (1000 mg/L) from their salts. All working solutions were obtained by diluting the stock solutions with appropriate amounts of distilled water. The pH of working solutions was adjusted to 11 by adding HNO_3 and NaOH aqueous solutions. This was monitored using pH meter (Suntex, model SP-701). The concentration of metal ions in aqueous solution was analyzed by Atomic Absorption Spectrophotometer (Buck scientific, model 210 VGP). A duplicate was analyzed for every sample to track experimental error and show capability of reproducing results (Marshall and Champagne, 1995).

Characterization of Ikpoba Clay

The pore size distribution and specific surface area of the Ikpoba clay samples were carried by N_2 adsorption using Micromeritics instrument (Tristar 3000) and by using Brunauer–Emmett–Teller (BET) method.

The specific surface area of Ikpoba clay was determined from the Brunauer, Emmett and Teller (BET) multipoint method (Bruanuer *et al.*, 1938) and the pore size distribution were obtained using Barret, Joyner, and Halenda (BJH) method (Barret *et al*., 1951). This was carried out by N_2 adsorption using Micromeritics instrument (Tristar 3000). Also pore volume was determined using the surface area analyzer which utilizes the BET theory for the analysis and plots of each sample data and then presents the results of pore volume (cm^3/g). .The cation exchange capacity (CEC) was determined by the procedure described by Mclean and Pratt (Mclean and Pratt, 1961). The mineralogical composition of the clay was obtained by XRD studies (PAN analytical X'Pert PRO MPD, PW 3040/60) using the Kx radiation of Cu.

Batch Equilibrium Experiments and Analytical Method

Adsorption was performed in batch experiments where 50 mL of Pb(II) solutions at the concentration of 30mg/L were added to 0.2 g and 0.8g of Ikpoba clay (size <0.02mm) in conical flasks and mixed with vapor bathing constant temperature vibrator (B. Bran Scientific and instrument corporation, England; Model: THZ-82);agitated at a speed of 100 and 300 rpm for 10 to 60 minutes at temperature range of 303 to 333K. Solution and clay were separated by filtering through a filter paper (Whatmann no. 42). Adsorption was monitored at each selected concentration. Similar procedure was performed for Cu(II) at the concentration of 3 mg/L. The residual metallic ion concentrations were determined using an Atomic Absorption Spectrophotometer (Buck scientific, model 210 VGP).A duplicate was analyzed for every sample to track experimental error and show capability of reproducing results (Marshall and Champagne, 1995).

$$q = \frac{(C_0 - C_e) * V}{m} \quad \textbf{(1)}$$

Where q is the specific uptake in mg/g at equilibrium, C_o and C_e are the initial and final concentration in mg/L, respectively, V the volume in liters of aqueous solution and m is the adsorbent dose in grams.

Statistical Design of Experiments

For the 2^4 experimental design, the four independent variables, adsorbent dose, contact time, shaking speed and temperature were respectively coded A, B, C and D at two levels. The high level was designated as +1 and the lower level as −1 (Table 1).

After performing a screening of the factors with the full factorial design for the adsorption of Cu(II) and Pb(II) onto clay, two independent response surface analysis statistical procedure (Box et al., 1978; Montgomery, 2001) were employed sequentially, in order to achieve the highest amount of copper(II) and Pb(II) adsorbed by the adsorbent. Minitab Statistical Software, Release 14.12.0, was employed for the statistical design of experiments and the optimization of adsorption of Pb(II) and Cu(II) onto the clay sample. The experimental set was carried out according to Tables 4 and 5.

In order to study the influence of the adsorbent dose and time on the sorption of Cu(II) and Pb(II) onto Ikpoba clay, a central composite design (CCD) was used. For the two factors, this design was made up of a full 2^2 factorial design with its four cube points, augmented with three replications of the center points and the four star points, that is, points having an axial distance to the center of (± 1.414) for one factor, whereas the other factor is at level 0, resulting in a total of 13 experiments. Thus, the influence of all experimental variables, factors and interaction effects on the response was investigated. The independent variables, experimental range and levels investigated for the CCD is given in Table 6.

Table 1A: The High and Low Levels of Experimental Factors for Cu(II).

Factor	Low Levels (-1)	High Levels (+1)
Adsorbent dose, g (A)	0.2	0.8
Contact Time, min (B)	10	60
Shaking Speed, rpm (C)	100	300
Temperature, K (D)	303	333

Table 1B: The High and Low Levels of Experimental Factors for Pb(II).

Factor	Low Levels (-1)	High Levels (+1)
Adsorbent dose, g (A)	0.2	0.8
Contact Time, min (B)	10	60
Shaking Speed, rpm (C)	100	300
Temperature, K (D)	303	333

RESULTS AND DISCUSSION

Table 2: Physico-Chemical Properties of Ikpoba Clay.

Parameters	Ikpoba Clay
Specific surface area - BET (m^2/g)	8.6932
Cation Exchange Capacity (meq/100 g)	4.8
Average Pore Diameter (nm)	26.22612
Pore Volume (cm^3/g)	0.101267

Table 3: Mineralogical Composition of Ikpoba Clay.

Mineral	Ikpoba clay Composition (%)
Kaolinite	32.56
Quartz	31.45
Anatase	14.11
Goethite	13.42
Violarite	8.46

DISCUSSION

Adsorbent Characterization

Table 2 shows the surface area, average pore diameter and pore volume for the adsorbent used for this study. The Specific surface area using the BET method was 8.6932m^2/g, the average pore diameter was 26.22612nm and pore volume was 0.101267 cm^3/g.

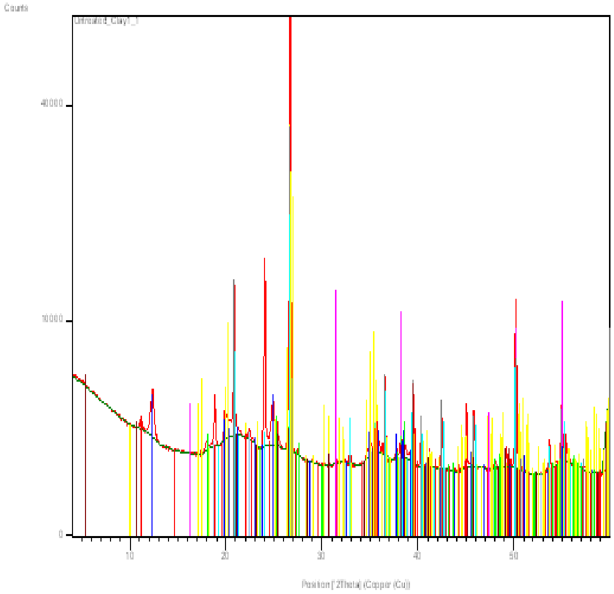

Figure 1: XRD Patterns for Ikpoba Clay.

Table 4: Optimization of Cu^{2+} and Pb^{2+} Adsorption on Clay: Full 2^4 Factorial Design with Two Pseudo-Central Points

Run	Factor				q	
	A	B	C	D	Cu^{2+}	Pb^{2+}
1	-1	-1	-1	-1	0.7125	6.0475
2	1	-1	-1	-1	0.180625	1.76125
3	-1	1	-1	-1	0.72	6.1525
4	1	1	-1	-1	0.181875	1.786875
5	-1	-1	1	-1	0.715	6.0625
6	1	-1	1	-1	0.18125	1.7625
7	-1	1	1	-1	0.7225	6.1625
8	1	1	1	-1	0.1825	1.788125
9	-1	-1	-1	1	0.71	6.0475
10	1	-1	-1	1	0.180625	1.71625
11	-1	1	-1	1	0.72	6.1525
12	1	1	-1	1	0.18125	1.786875
13	-1	-1	1	1	0.7175	6.0625
14	1	-1	1	1	0.180625	1.7625
15	-1	1	1	1	0.7275	6.16
16	1	1	1	1	0.183125	1.7875
17	0	0	0	0	0.289	2.535
18	0	0	0	0	0.287	2.576

As observed, the surface area for the clay is relatively low; below the range of 10 to 20 m^2/g established for kaolinite (Bohn *et al*., 1985; Yong *et al.*, 1992; Uehara *et al*., 1972).This may be due to the high quartz content of the clay. The CEC was also observed to be low (4.8 meq/100 g). This is however within the range of values of 3 and 15 meq/100 g for Kaolinite given elsewhere (Grim, 1986). With a pore diameter of 26.22612nm, the clay sample is mesoporous based on IUPAC classification (Rodriguez—Reinoso and Linares-Solano, 1989); hence it's rather low pore volume of 0.101267cm^3/g is understandable.

From XRD analysis presented in Table 2 and Figure 1, the results show that kaolinite has the highest percentage of 32.56 while the lowest mineral in the clay was Violarite with 8.46%.

The kaolinite mineral content (32.56 %) obtained is much less than the percentage reported for Kaolinitic clay from elsewhere (Miranda-Trevino and Coles, 2003; Ketcha Mbadcam, *et al*., 2011); in the same context, the high percentage (31.45 %) of free quartz is comparable to the value recorded in the literature for free quartz of Kalabsha (Talaat et al., 2011).

Statistical Design of Experiments

In this work four variables, adsorbent dose, contact time, shaking speed and temperature have been selected to optimize the adsorption process using the statistical design of experiments mentioned earlier.

Table 5 shows the experiment main, interaction effect, coefficients of the model, and standard deviation of each coefficient, and probability for the full 2^4 factorial designs for the adsorption of both lead (II) ions and copper (II) ions. As can be seen, only the adsorbent dose had significant effect at a 5% of probability level ($P < 0.05$) for both Cu(II) and Pb(II) sorption onto clay. It can also be seen from Table 4 that no interactive effects of factors was significant as P>0.05.

The ranking of the importance of each factor and interaction in the global processes of batch adsorption will depend on the numerical value of the coefficient of each factor or its interaction in absolute value (Brasil et al., 2005; Pavan *et al*., 2007).

Table 5: Full 2^4 Factorial Estimated Effects and Coefficients for q.

Term	Cu^{2+}				Pb2+			
	Effect	Coeff	StDev Coef	P	Effect	Coeff	StDev Coef	P
Constant	---	0.4318	0.03596	0.007	---	3.784	0.3071	0.007
Ads dose	-0.5366	-0.2683	0.03814	0.02	-4.34	-2.168	0.3258	0.022
Time	0.0051	0.0025	0.03814	0.953	0.069	0.035	0.3258	0.925
Speed	0.0029	0.0014	0.03814	0.973	0.012	0.006	0.3258	0.987
Temp	0.0005	-0.0003	0.03814	0.995	-0.01	-0.003	0.3258	0.993
Ads dose*Time	-0.0037	-0.0018	0.03814	0.966	-0.03	-0.016	0.3258	0.965
Ads dose*Speed	-0.0021	-0.0011	0.03814	0.98	0.00	0.00	0.3258	1.00
Ads dose*Temp	-0.0007	-0.0004	0.03814	0.993	-0.01	-0.003	0.3258	0.994
Time*Speed	0.0002	0.0001	0.03814	0.998	-0.01	-0.004	0.3258	0.992
Time*Temp	0.0007	0.0004	0.03814	0.993	0.005	0.003	0.3258	0.994
Speed*Temp	0.0013	0.0007	0.03814	0.988	0.005	0.003	0.3258	0.994
y	0.0002	0.0001	0.03814	0.998	0	-0.002	0.3258	0.996
Ads*Time*Temp	-0.0005	-0.0003	0.03814	0.995	0.006	0.003	0.3258	0.994
Ads*Speed*Temp	-0.0012	-0.0006	0.03814	0.989	0.006	0.003	0.3258	0.994
Time*Speed*Temp	0.0002	0.0001	0.03814	0.998	-0.01	-0.003	0.3258	0.993
Ads*Time*Speed*Temp	0.0002	0.0001	0.03814	0.998	-0.01	-0.003	0.3258	0.994

Table 6: Optimization of Cu(II) and Pb(II) Adsorption on Clay: Central Composite Surface Design with Two Factors.

Experiment	Factor		q	
	A	B	Cu^{2+}	Pb^{2+}
1	-1	-1	0.7225	6.69
2	1	-1	0.2425	2.351667
3	-1	1	0.7275	6.6975
4	1	1	0.245833	2.3575
5	-1.41421	0	0.725	6.71
6	1.41421	0	0.245	2.356667
7	0	-1.41421	0.36125	3.52
8	0	1.41421	0.364	3.525
9	0	0	0.365	3.525
10	0	0	0.365	3.52375
11	0	0	0.36375	3.52375
12	0	0	0.365	3.525
13	0	0	0.36375	3.525
Independent Variables		Low Levels(-1)	High levels (+1)	
Adsorbent dose, A (g)		0.2	0.6	
Contact Time, B(min)		60	120	

The temperature was fixed at 303K shaking speed fixed at 300 rpm

It can be seen from Table 4 that adsorbent dose was the most important factor for the overall adsorption. The positive values of effects means that an increase in their levels lead to an increase in the metallic ion, q uptake by the adsorbent; the negative values of the effects lead to a diminution of the response, q when their levels were increased.

The second most important factor to the overall optimization of the batch adsorption processes for Cu(II) and Pb(II) was the time of contact between the adsorbents and the adsorbate. Increasing contact times from 60 to 120 min lead to an augmentation of the response, q. probably for low contact times, the batch adsorption system did not reach the equilibrium. Higher times of contact should be explored in further statistical design of experiments in order to optimize this factor.

After performing a screening of factors using a full 2^4 factorial design, a central composite response surface design (containing 13 experiments, divided in four cube points, four axial points and five central points) was carried out according to experiments described in Table 4, in order to achieve the highest amount of Cu(II) and Pb(II) uptaken by the clay adsorbents. The levels of the chosen factors were set based on the previous factorial design described above, varying the adsorbent dose to 0.2 g to 0.6g and the contact time up to 120 min and fixing the shaking speed at 300rpm and temperature at 303K.

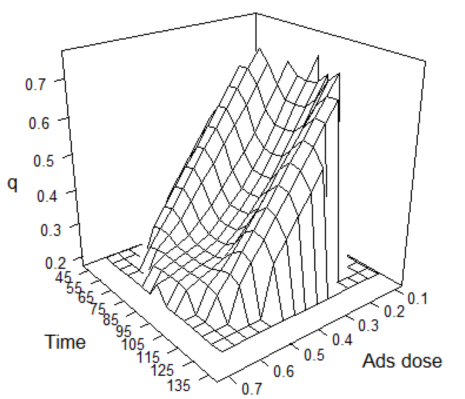

Figure 2a: Response Surface for Cu(II) Adsorption on Clay.

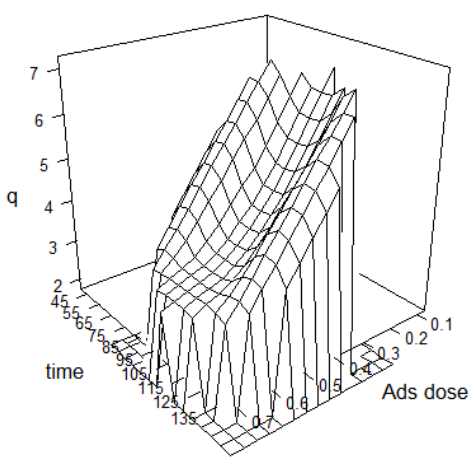

Figure 2b: Response Surface for Pb(II) Adsorption on Clay.

In Figure 2 were shown the contour plots of the response (*q*) for time of contact between the adsorbate and adsorbents and also 3D surface plots, the optimal conditions were temperature of 303K, adsorbent dose of 0.2g, shaking speed of 300rpm for both metal ions and contact time of 60 min and 90 respectively for Cu^2+ and Pb^{2+}.

CONCLUSION

The use of a factorial experimental design has helped in identifying the most important factors influencing the copper ions and lead ions adsorption using Ikpoba clay as adsorbent. From the statistical analysis, adsorbent dose was the only significant factor for both Cu(II) and Pb(II) adsorption on the clay. The optimum adsorption conditions selected were temperature of 303K, adsorbent dose of 0.2g, shaking speed of 300rpm for both metal ions and contact time of 60 min and 90 respectively for Cu^2+ and Pb^{2+}.

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