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KOLANUT POD HUSK: A POTENTIAL BIOSORBENT FOR Cd^{2+}, Ni^{2+} AND Pb^{2+}

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ABSTRACT: *Wastewaters from industries should be treated before discharge. The pollution problems posed by toxic heavy metals to the environment have been a concern. The use of unmodified and mercaptoacetic acid modified kolanut pod husk as biosorbents in detoxifing Cd^{2+}, Ni^{2+} and Pb^{2+} ions from aqueous solutions were investigated using a batch sorption process. Biosorption was carried out in a batch process at various contact time and dose with initial metal ions concentration of 100 mg/l using 250 µm size of the of the unmodified and modified kolanut pod husks respectively at a temperature of 25 ^{0}C and pH of 7.5. Maximum biosorption capacities of unmodified kolanut pod husk were observed at 3g, values given as 98.999 mg/g, 89.870 mg/g for Cd^{2+}, Ni^{2+} and 89.890 mg/g for Pb^{2+} at 2g while that of modified kolanut pod husk indicated 99.952 mg/g at 2g for Cd^{2+} and 99.776 mg/g, 99.021 mg/g for Ni^{2+} and Pb^{2+} ions at 3g. More so at 60 minutes for Cd^{2+}, 90 minutes for Ni^{2+} and 10 minutes for Pb^{2+}, values given as 99.986 mg/g, 99.999 mg/g and 99.999 mg/g by the unmodified kola nut pod husk while modified kolanut pod husk gave 99.666 mg/g at 30 minutes for Cd^{2+}, 99.664 mg/g at 30 minutes for Ni^{2+} and 96.164 mg/g at 90 minutes for Pb^{2+}. Generally maximum biosorption were all favoured at low doses and at low contact time. The kinetic of metal ions biosorption on unmodified and modified kola nut pod husks have also been studied by fitting the data in Lagergren's first-order, Ho-Mckay's pseudo-second-order kinetics hypothesis and Elovich adsorption model. It was observed that the removal of metal ions over the biosorbent showed a better fit with the pseudo-second-order process and Elovich adsorption model than the pseudo -first-order. From this work kolanut pod husk had proven to be a good biosorbent for Cd^{2+}, Ni^{2+} and Pb^{2+}.*

KEYWORDS: Adsorption Kinetics, Biosorbent Dose, Contact Time, Heavy Metal Ions, Kolanut Pod Husk

INTRODUCTION

The mobilization of heavy metals in the environment through anthropogenic activities such as industrial and agricultural activities is of serious concern because of the toxicity of these metals to the environment such as aquatic ecosystems. Pollution of an aquatic environment can alter its chemical, physical and biological characteristics and equally contaminate the quality of water for human consumption [1]. Heavy metals are trace metals with an atomic density greater than 5g/cm^3 [2]. They have been described as "the most common pollutants in wastewater [1]. Although they are found naturally in the earth's crust, some anthropogenic activities have resulted in elevated concentrations of heavy metals in the environment [3]. Significant anthropogenic sources of heavy metals in the environment include: metalliferous mining, smelting operation, road and transportation runoff, landfill sites, agriculture, fossil fuel combustion, surface finishing, photography, electric appliance manufacturing, aerospace and atomic energy installations [4, 5, 6]. Heavy metals of concern include: Hg, Cd, Ni, Pb, Cu, Zn, Cr, Co, As etc. Some are highly toxic to flora and fauna even at a very low concentration [4, 5] and due to their non-biodegradability and persistence of some of them, can accumulate in the environment such as food chain, and thus may pose a significant danger to human health [7, 8]. According to [9] and [10], the severe form of cadmium toxicity in human is “ita-itai” a disease characterized by excruciating pain in the bone. Toxic levels of lead in man have been associated with encephalopathy, anemia, seizures, coma, mental retardation and bizarre behavior [11, 12]. Acute inhalation exposure to nickel may produce headache, nausea, respiratory disorders and death [13, 14]. Epidemiological studies have shown that occupational inhalation exposure to nickel dust at refineries have resulted in increased incidences of pulmonary and nasal cancer [15, 16]. In other words, the inadequate discharged of these liquid wastes may not be wholesome hence, their removal is vital from the stand point of environmental pollution control [17, 18]. Application of physico-chemical wastewater treatment processes were sometime restricted because of technical or economical constraints [19, 20]. Hence, the use of biosorbents as more economical means for the metal removal have been sought for and highly advantageous [21]. Various biosorbents have been utilized in biosorption of heavy metal from wastewaters [22, 23, 24, 25, 26, 27, 28, 29], and many others. This work highlights the use of kolanut pod husk as a biosorbent in adsorbing Cd^{2+}, Ni^{2+} and Pb^{2+} from their aqueous solutions using the dose and particle size of the biosorbent as the important experimental parameters.

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METHODOLOGY

The *Cola nitida* was obtained from Gariki Market Okigwe in Imo State and processed to get the husk. The husk was grounded into tiny particle size using manual grinder and sieved through a test-sieve shaker after washing with de-onized water and drying in oven at 50 ^{0}C for 12hrs to get various mesh sizes (125, 250, 375, 600, 625, 850) µm. It was then activated by soaking in 2% (v/v) dilute nitric acid solution for 24 hours, filtered, rinsed severally with de-ionized water and allowed to dry in the oven at 105^{0}C for about 6 hours. Hence labeled unmodified sample. About 10 g portion of the activated sample was modified using mercarptoacetic acid by soaking the sample into 1000 cm^3 of 0.3 mol mercarptoacetic acid for 2hrs at 25^{0}C, filtered, rinsed with de-ionized water and finally dried at 50^{0}C for 12hrs. 100 mg/l concentration of Cd^{2+}, Ni^{2+} and Pb^{2+} prepared as an aliquot from the stock solution of 1000 mg/l was used. For the particle determination via batch biosorption process, 1g of the various sizes (125, 250, 375, 600, 625, 850) µm of both unmodified and modified samples were put into a 250cm^3 conical flasks containing 50 cm^3 i.e. (5 ml aliquot of 1000 mg/l stock solutions + 45 cm^3 de-ionized water) portion of the test solutions and allowed to stand for 1 hr with intermittently shake. The solutions were filtered and the filtrates were analyzed for residual metals using atomic absorption spectrophotometer (Buck model 200 A). For the time effect, 50 cm^3 of each of the metal ion solutions of 100 mg/l were placed into various flasks containing 1g (250 µm) size of the sample. The flasks were agitated for various time intervals of 10, 30, 60, 90 and 120 minutes, after which the mixtures were filtered and the filtrates were analyzed for residual metal using AAS (Buck model 200A). Triplicate analysis were made and the amounts of Cd^{2+}, Ni^{2+} and Pb^{2+} adsorbed during the series of batch investigations were determined using a simplified mass balance ($Qe = C_o - C_e$), where Q_e = amount adsorbed (mg/g) by the adsorbents at equilibrium or metal ion concentration on adsorbent at equilibrium, C_e = metal ion concentration (mg/l) (final concentration) in the solution (of the filtrate) at equilibrium while C_o = initial metal ion concentration (mg/l) in solution used.

RESULTS AND DISCUSSION

Effect of particle size on adsorption:

Table 1: Amount of heavy metal ions adsorbed by various sizes of kolanut pod husk (KNPH) from aqueous solution at 298 K.

	Unmodified KNPH			Modified KNPH		
Particle size(μm)	Cd^{2+} (mg/g)	Ni^{2+} (mg/g)	Pb^{2+} (mg/g)	Cd^{2+} (mg/g)	Ni^{2+} (mg/g)	Pb^{2+} (mg/g)
125	99.977± 0.039	97.615± 0.322	98.557± 0.150	97.520± 0.088	97.789± 0.048	98.420± 0.168
250	99.982± 0.041	99.999± 0.552	99.748 ±0.336	98.919± 0.463	97.816± 0.059	98.970 ± 0.393
375	99.859± 0.009	99.961± 0.636	99.696± 0.315	97.824± 0.016	97.765± 0.038	97.800 ± 0.085
500	99.855± 0.010	97.646± 0.309	98.671± 0.103	97.397± 0.158	97.555± 0.048	97.640 ± 0.150
625	99.810± 0.029	97.600± 0.328	98.444± 0.196	97.365± 0.171	97.504± 0.068	97.612 ± 0.162
850	97.596± 0.329	98.428± 0.202	97.204± 0.237	97.601± 0.029	97.608 ± 0.163	99.800± 0.033

Table 1 shows concentrations of Cd^{2+}, Ni^{2+} and Pb^{2+}adsorbed by various sizes of unmodified and modified kolanut pod husk from aqueous solutions. The data generally revealed that the adsorption capacities of both unmodified and modified kolanut pod husk increases with decrease in particle size indicating that the adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by unmodified and modified kolanut pod husk increases with increase in the surface area of contact. Slight differences were observed between the adsorption capacities of both. Generally, modification was found to decrease the adsorption capacities of the unmodified and modified kolanut pod husk. However, adsorption capacities were fairly better for Cd^{2+} and Pb^{2+} than for Ni^{2+}. Fig. 1 below shows the variation of amount of Cd^{2+}, Ni^{2+} and Pb^{2+} adsorbed with various sizes of the unmodified and modified kolanut pod husk. From the Fig. 1, adsorption of Ni^{2+} and Pb^{2+} by unmodified and modified kolanut pod husk first increases with particle size but decreases as the particle size increases. Similar observation was found for the adsorption of Cd^{2+} by unmodified kolanut pod husk but for modified kolanut pod husk, Cd^{2+} adsorption decreases linearly with particle size.

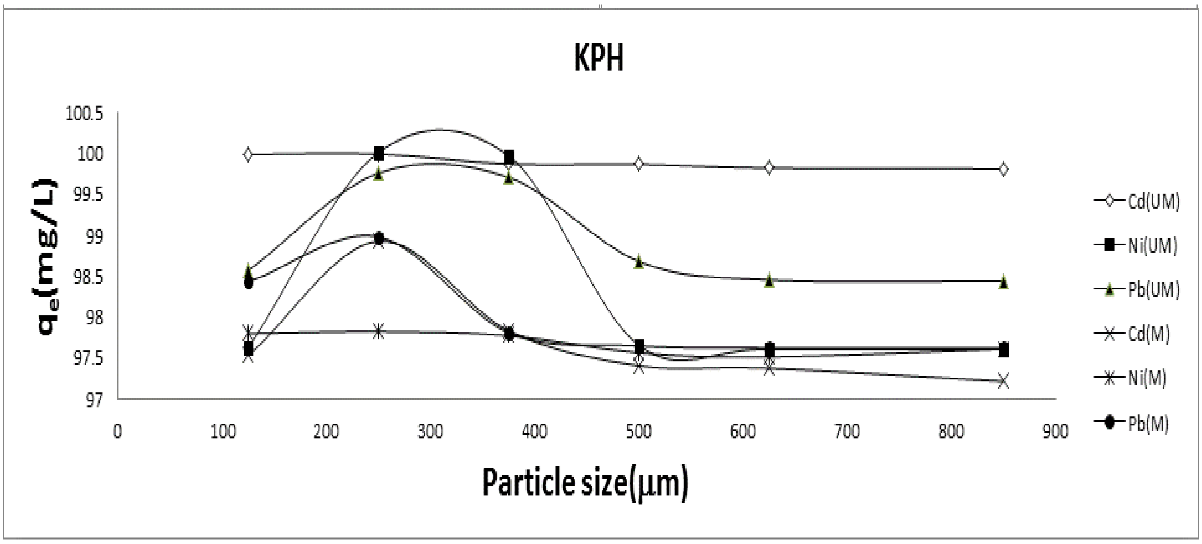

Fig. 1: Plot for the Effect of Particle Size on the Adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by Unmodified and Modified Kolanut Pod Husk.

Effect of Contact Time on Adsorption:

Table 2: Concentrations of Cd^{2+}, Ni^{2+} and Pb^{2+} adsorbed by kolanut pod husk (KNPH) from aqueous solutions at various time and at 298 K.

	Unmodified KNPH			Modified KNPH		
t (min)	Cd^{2+} (mg/g)	Ni^{2+} (mg/g)	Pb^{2+} (mg/g)	Cd^{2+} (mg/g)	Ni^{2+} (mg/g)	Pb^{2+} (mg/g)
10	99.878± 0.181	99.999± 0.412	99.999 ± 0.070	99.470± 0.025	99.502± 0.035	85.190± 2.904
30	99.624± 0.067	97.730 ± 0.602	99.899 ± 0.026	99.666± 0.063	99.664± 0.038	90.033± 0.738
60	99.986± 0.229	97.657 ± 0.635	99.834± 0.003	99.226± 0.134	99.540± 0.018	92.531± 0.379
90	99.958± 0.217	99.999 ± 0.412	99.748 ± 0.042	99.640± 0.051	99.654± 0.033	96.164± 2.004
120	97.923± 0.694	99.999 ± 0.412	99.727 ± 0.062	99.626± 0.045	99.538± 0.019	94.501± 1.260

Table 2 shows the variation of the concentrations of Cd^{2+}, Ni^{2+} and Pb^{2+} adsorbed by kolanut pod husk from aqueous solutions at various time intervals. From TABLE 2, it can be seen that except for the adsorption of Cd^{2+} by unmodified kolanut pod husk, concentrations of heavy metal ions adsorbed were found to increase at a shorter time (10 min) though maximum adsorption of Ni^{2+} was also seen at 90 and 120 minutes. However, adsorptions of Cd^{2+}, Ni^{2+} and Pb^{2+} by kolanut pod husk on modification were also found to decrease with increase in time interval though the adsorptions of did not closely follow a definite pattern indicating that modification had effect on the adsorption capacity of these adsorbents.

From the results (TABLE 2), it is indicative that the concentrations of heavy metal ions adsorbed do not vary regularly with time. In some cases, the amount increases with time while in others, it decreases with time. The average impact of time on the adsorption capacity of heavy metal ions depends on the number of adsorption sites available and on the strength of adsorption and desorption mechanism.

Kinetic Studies:

Several kinetic models including pseudo-first-order, pseudo-second-order and intra-particle diffusion models are common kinetic models that can be used to examine the controlling mechanism involved in the adsorption of Cd^{2+}, Pb^{2+} and Ni^{2+} by unmodified and modified kolanut pod husk.

The pseudo-first-order equation is given as [30]:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1 t}{2.303} \quad (1)$$

Where q_e and q_t are the amount of heavy metal ions adsorbed at equilibrium and at time, t (respectively mg/g) and k_1 is the first-order rate constant (min^{-1}). From equation 1, a plot of log (q_e – q_t) versus t should be linear if a pseudo-first-order kinetic is obeyed. However, when adsorption data obtained for unmodified and modified kolanut pod husk were fitted into the model expressed by equation 1, values of R^2 obtained from the plots (plots not shown) were very low indicating that the adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by nmodified and modified kolanut pod husk is not consistent with a pseudo-first-order kinetics.

According to [31], a pseudo-second-order adsorption rate equation can be expressed as follows,

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (2)$$

Where k_2 is the rate constant of pseudo-second-order adsorption ($gmg^{-1}min^{-1}$), q_e and q_t are the adsorption capacity at equilibrium and at time, t, respectively. Introducing boundary conditions to equation 1, i.e t = 0 to t=t and q_e = 0 to q_t = q_t, integrated form of equation 2 was obtained (equation 3) and upon simplification, equations 4 and 5 were obtained.

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_t} + k_2 t \quad (3)$$

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t) \quad (4)$$

$$\frac{1}{q_t} = \frac{1}{h} + \frac{1}{q_e}(t) \quad (5)$$

The implication of equation 5 is that a plot of $1/q_t$ versus t should be linear with slope and intercept equal to q_e and $\frac{1}{h}$ (h = $k_2 q_e^2$) respectively. Fig. 2 shows a pseudo-second-order kinetic plots for the adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by unmodified and modified kolanut pod husk respectively. Adsorption parameters obtained from the plots are presented in TABLE 3. From the results obtained, it can be seen that values of R^2 are very close to unity indicating the application of a pseudo-second-order model to the adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by unmodified and modified kolanut pod husk. It is also evident from the results that values of k_2 calculated from the plots are relatively low. It was observed that h and k_2 values for the adsorption of Cd^{2+} and Pb^{2+} by kolanut pod husk were found to be higher for the modified kolanut pod husk. According to [32], a pseudo-first and -second order kinetics cannot reveal the mechanism of adsorption therefore, a model of Elovich was also used to test for the adsorption mechanism of the studied adsorbates. Elovich adsorption model can be expressed as follows [33, 34]**:**

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t) \qquad (6)$$

Whereα is the initial adsorption rate ($mgg^{-1}min^{-1}$) and β is the desorption constant (gmg^{-1}) during any one experiment. [33] had proposed that $\alpha\beta t >> t$ and if the boundary conditions are applied (i.e t=0 and $q_t = q_t$ at t = t), the Elovich equation becomes:

$$q_t = \frac{1}{\beta} ln(\alpha\beta) + \frac{1}{\beta} \ln(t) \qquad (7)$$

The implication of equation 7 is that a plot of q_t versus ln(t) should be linear with slope and intercept equal to $\frac{1}{\beta}$ and $\frac{1}{\beta} ln(\alpha\beta)$ respectively. Fig. 3 shows Elovich equation for the adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by unmodified and modified kolanut pod husk. Values of Elovich constants deduced from the plots are presented in TABLE 4. From the results obtained, it can be seen that the adsorption of some heavy metal ions by unmodified and modified kolanut pod husk fitted the Elovich adsorption model excellently. However, some of them did not fit the model.

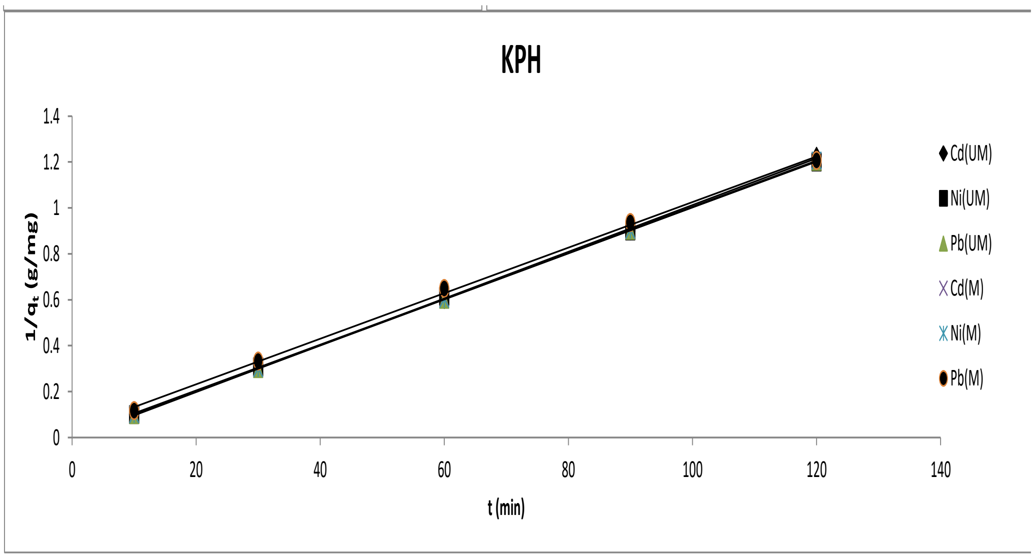

Fig. 2: Variation of $1/q_t$ with t (Pseudo-Second-Order Kinetic) for the Adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by Unmodified and Modified Kolanut Pod Husk.

Table 3: Pseudo-Second-Order Adsorption Parameters for the Adsorption of Cd^{2+}, $Ni^{2+}$and Pb^{2+} by Unmodified and Modified Kolanut Pod Husk.

System	Ions	Slope	q_e	h	K_2	R_2
KNPH	Cd(UM)	0.0102	-0.006	-1.66E+02	-1.73E-02	0.9997
	Ni(UM)	0.0100	0.0062	1.61E+02	1.61E-02	0.9998
	Pb(UM)	0.0100	0.0006	1.67E+03	1.67E-01	1.0000
	Cd(M)	0.0100	0.0008	1.25E+03	1.25E-01	1.0000
	Ni(M)	0.0100	0.0001	1.00E+04	1.00E+00	1.0000
	Pb(M)	0.0099	0.0331	3.02E+01	2.96E-03	0.9987

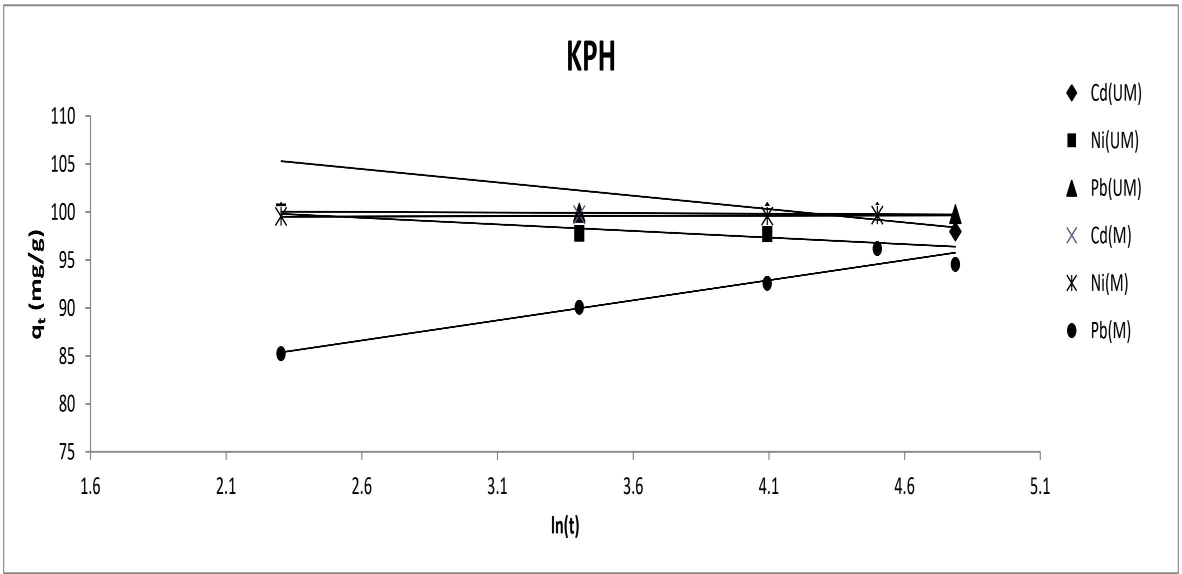

Fig. 3: Variation of q_t with ln(t) (Elovich Adsorption Model) for the Adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by Unmodified and Modified Kolanut Pod Husk.

Table 4: Elovich Parameters for the Adsorption of Cd^{2+}, Ni^{2+} and Pb^{2+} by Unmodified and Modified Kolanut Pod Husk.

System	Metal ion	Slope	Intercept (I)	β	Iβ	α	R^2
KNPH	Cd(UM)	-2.7855	111.71	-0.3590	-40.10	-1.0E-17	0.6722
	Ni(UM)	-1.3761	102.96	-0.7267	-74.82	-4.4E-33	0.8717
	Pb(UM)	-0.1112	100.27	-8.9928	-901.71	0	0.9772
	Cd(M)	0.0774	99.33	12.9199	1283.32	-	0.6376
	Ni(M)	0.0547	99.37	18.2815	1816.64	-	0.6526
	Pb(M)	4.1882	75.70	0.2388	18.07	2.96E+08	0.9414

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