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Kinetics of Adsorption of Lead onto Low Cost Adsorbents: Comparison of Linear and Non-Linear Regression Methods

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ABSTRACT: The purpose of this paper is to utilize common solid wastes (carbon rods from used batteries, egg shells and corn cobs) as adsorbents for lead removal from aqueous solution. For this purpose, linear and non-linear regression methods of widely used kinetics models were compared. Solid wastes (specifically, egg shells, corn cobs and used dry batteries) were collected from selected location in Nigeria. The materials collected were washed with distilled water, air-dried, ground into powder and classified using British Standard sieve. The mineral contents of these adsorbents were determined. Adsorption capacities of powdered adsorbents were examined on synthetic lead solution prepared by using standard method. The results of adsorption capacities analysed using linear and non-linear techniques were evaluated statistically. The present study reflected that these materials are good adsorbents based on the micrograph and adsorption capacities. In the result of the statistical evaluations, non-linear regression method is the best method to be used for: analysis adsorption kinetics model(intraparticle diffusion and pseudo second order) based on high MSC (2.54, 2.34 and 2.57; 4.45, 4.15 and 4.22) and CD (0.960, 0.951 and 0.961; 0.994, 0.992, 0.992); low error (0.375, 0.325 and 0.213; 2.994, 2.604 and 2.909) and Chi squared value (0.030, 0.030 and 0.020; 0.180, 0.190 and 0.200) and determination of engineering parameters to prevent failure of adsorption reactors. Cost analysis showed that these adsorbents are cheaper compared to the cost of producing other adsorbents such as pen con shell based activated carbon (2.72 USD kg^{-1}) and sugar cane based granular activated carbon by steam (3.12 USD kg-^{1}).

Keywords: Pb^{2+} removal, PCC, aqueous solution, regression methods, environmental pollution control

Kurşunun Düşük Maliyetli Absorbent ile Absorbsiyonunun Kinetiği: Doğrusal ve Doğrusal Olmayan Regresyon Modellerinin Karşılaştırılması

ÖZET: Bu makalenin amacı, sıvı çözeltiden kurşun giderimini sağlamak amacıyla absorbentler olarak sıradan katı atıklardan (atık pillerin karbon çubukları, yumurta kabukları ve mısır koçanları) yararlanmaktır. Bu amaç için, kinetik modellerde yaygın olarak kullanılan doğrusal ve doğrusal olmayan modellerin karşılaştırılmıştır. Bu katı atıklar (atık kuru pillerin karbon çubukları, yumurta kabukları ve mısır koçanlar) Nijerya'nın belli bölgelerinden toplanmıştır. Toplanan materyallar, distile su ile yıkanıldıktan sonra hava ile kurutulmuş ve toz haline getirildikten İngiliz elek standartları ile sınıflandırılmıştır. Toz haline getirilen absorbentlerin absorbsiyon kapasiteleri, standart metotlar kullanarak hazırlanan sentetik kurşun çözeltisi ile incelenmiştir. Absorbsiyon kapasiteleri, doğrusal ve doğrusal olmayan analiz teknikleri ile değerlendirilmiş, elde edilen sonuçlar istatistiksel olarak değerlendirilmiştir. Çalışma sonuçları, mikrograf ve absorbsiyon kapasitelerine bağlı olarak incelenen materyallerin, iyi absorbentler olduğunu göstermiştir. Yapılan istatistiksel değerlendirmeler sonucunda, doğrusal olmayan modellerin, yüksek MSC (2.54, 2.34 ve 2.57; 4.45, 4.15 and 4.22) and CD (0.960, 0.951 ve 0.961; 0.994, 0.992, 0.992); düşük hata (0.375, 0.325 and 0.213; 2.994, 2.604 and 2.909) and ki-kare değerleri (0.030, 0.030 ve 0.020; 0.180, 0.190 ve 0.200) temelinde absorbsiyon kinetik modelleri (intra-partikül difüzyon ve pseudo ikinci mertebe) için kullanılan ve absorbsiyon reaktörlerinin başarısızlığını önleyen en iyi metot olduğu saptanmıştır. Maaliyet analizi, bu absorbentlerin diğer absorbentlerden (aktive edilmiş karbona dayalı atıklar (2.72 USD kg^{-1}) ve buharlı taneli aktif karbona dayalı seker kamışı (3.12 USD kg-^{1})) daha ucuz olduğunu göstermiştir.

Anahtar Kelimeler: Çevre kirliliği, Pb^{2+} giderimi, regresyon modeller, sıvı çözelti, Toz haline getirilmiş mısır koçanı (THGMK).

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INTRODUCTION

Population growth and higher living standards will cause ever increasing demands for good quality municipal and industrial water, and ever increasing sewage flows. At the same time, more and more potable water will be needed to meet increasing demands for food and for growing populations. Also, more and more water will be required for environmental concerns such as aquatic life, wildlife refuges, recreation and scenic values. Thus, increased competition for quality water and effective wastes management are expected. This will require intensive management and international cooperation. Since almost all liquid fresh water on the planet occurs underground, groundwater will be used more and more and, hence, must be protected against depletion and contamination, especially from various sources like intensive agriculture and solid waste disposal (Bouwer, 2003).

Water resources management should be flexible so as to be able to cope with changes in availability and demands for water. This calls for integrated water management where all pertinent factors are considered in the decision making process. Such a holistic approach requires not only supply management, but also demand management (e.g., water conservation and transfer of water to uses with higher economic returns), water quality management, recycling and reuse of water, economics, conflict resolution, public involvement, public health, environmental and ecological aspects, socio-cultural aspects, water storage (including long-term storage or water "banking"), conjunctive use of surface water and groundwater, water pollution control, flexibility, regional approaches, weather modification and sustainability. Proper solid wastes and water management increasingly must be integrated with other wastes management and environmental objectives such as water and wastewater treatments.

Wastewater and water treatment processes are pH adjustment, chemical treatment, adsorption, electrochemical, ion exchange, precipitation, evaporative recovery and membrane processes. Out of all these treatment processes, adsorption has been found economical and applicable at all levels (Sa and Kutsa, 1995; Bhide et al., 1996; Ozer et al., 1997). Khashimova et al (2008) reported that the use of adsorption for separation of pollutants from mixtures has been increasing continuously and that the main advantages of adsorption are its high selectivity compared with other separation techniques and relatively high capacity of the adsorbents for the pollutants even at low concentrations. Ho (2007) reported that about 9058 articles have been published on adsorption of materials**.** It is well known that adsorption dynamics relationship between sorbents and sorbates are described by sorption kinetics models. Table 1 presents adsorption kinetics models. Adsorption kinetics models give the capacity of a sorbent for a sorbate. Kinetics can be obtained by examining batch reactions at fixed operational conditions. Linear regression methods are frequently used to determine the best-fitting isotherm and kinetics. The linear least-squares method with linearly transformed kinetics equations has also been widely applied to confirm experimental data (kinetics) using coefficients of determination.

In recent years, literature has highlighted the use of non-linear regression methods in solving environmental engineering problems (Oke and Akindahunsi, 2005; Ho, 2006; Oke et al., 2006; Yeh and Chen, 2009; Oke, 2009). These call for statistical evaluation of non-linear and linear regression methods to ascertain their accuracy, reliability, good fitting and predictability to prevent engineering failure of adsorption reactor, which is a common phenomenon in both developed and developing countries. There are several error analysis methods, such as the coefficient of determination (CD), the sum of the errors squared (SES), the Squared Euclidean Distance, average relative standard error, average relative error, sum of the absolute errors, sum of the squares of the errors, a hybrid error function (HEF), Marquardt's percent standard deviation, the average relative error (ARE), the sum of the absolute errors (SAE), model of selection criterion (MSC) and Chi-square have been used to determine the best-fitting isotherm equation (Monte et al., 2003; Subhash, 1996; Han et al., 2009; Kundu and Gupta; 2006; Han et al., 2007; Allen et al., 2003; Ho, 2006; 2002). In this study, the linear least-squares and non-linear regression methods of widely adsorption kinetics models were compared in an experiment examining lead sorption onto low cost adsorbents (specifically, powdered corn cobs, powdered egg shell and powdered carbon rods). A software (SPSS 15) was used for both linear least-squares and non-linear regression methods..

MATERIALS AND METHODS

Selected solid wastes (specifically, egg shells, corn cobs and used dry batteries) were collected from selected location in Nigeria. They (the materials collected) were washed with distilled water, air-dried, ground into powder and classified using British Standard (BS) sieve. These adsorbents were selected on the basis of their availability (Figure 1.) and their lower initial cost of production. Powdered adsorbents with sieve sizes of 300-425 μm, 212-300 μm, 75-150 μm and 150-212 μm were separated and stored in different desiccators. The mineral contents of these adsorbents were determined by using AAS after acid digestion of 2 g samples of the adsorbents (APHA, 1998) . Structures (Micrograph) of the adsorbent were examined to ascertain its nature and porosity. Adsorption capacities of powdered adsorbents were examined on synthetic lead solution prepared by dissolving a known mass of lead salt in distilled water (APHA, 1998). Specifically, a known mass (1.599g) of lead nitrate ($Pb(NO_3)_2$) was dissolved in 200ml of distilled water and 10 ml of concentrated HNO_3 was added. The mixture was diluted to 1000ml mark using distilled water and working solutions were prepared from the stock solution. In the determination of adsorption capacities 300ml of the lead solutions containing 150.00 mg L $-^1$of Pb^{2+} each were taken into different beakers and known masses of the adsorbent were added at a known initial pH. The mixtures were stirred at 60 revolutions per minute (rpm) for 3 minutes and allowed to stand for 24 hours. The supernatants were filtered through a filter paper Number 40 (Whatman) to remove suspended solids and lead concentration in the filtrate was determined (Concentration of Pb^{2+} in the solution was determine hourly). The laboratory determination of lead concentrations in synthetic solutions were conducted by using procedures as specified in APHA (1998) using the Alpha 4 Atomic Absorption Spectrophotometer (AAS) (Chem Techn Analytical) at the Central Science Laboratory, Obafemi Awolowo University, Ile-Ife, Nigeria. The final concentrations of Pb^{2+} were determined using standard curves (not presented). The amount of solute removed (adsorbed) was calculated using equation (1). The percentage of lead ion removed (R_R %) from the solution was calculated using equation (2).

$$q_t = \frac{\left(C_0 - C_t\right)}{M} V \qquad (1)$$

$$R_R = 100 \frac{\left(C_0 - C_t\right)}{C_0} \qquad (2)$$

All the data presented are the average of the replicates. The adsorption capacities of the adsorbent through adsorption kinetics models were analyzed using Levenberg Marquardt algorithm (1963) through linear least-squares and non-linear regression methods. The statistical evaluations of these adsorption kinetics models (Pseudo second order and intraparticle diffusion) were expressed as coefficient of determination (CD), sum of the squares of the errors (SSE), model of selection criterion (MSC) and Chi-squared. Cost analysis of producing these adsorbents was conducted. The lower the value of total error the higher the accuracy, validity and good fitness of the methods. Total error (Err^2) can be computed using equation (3):

$$Err^2 = \sum_{i=1}^{n} \left(Y_{obsi} - Y_{cali} \right)^2 \qquad (3)$$

The coefficient of determination *(CD)* can be interpreted as the proportion of expected data variation that can be explained by the obtained data. Higher values of CD indicate higher accuracy, validity and good fitness of the device. CD can be expressed as follows:

$$CD = \frac{\sum_{i=1}^{n} \left(Y_{obsi} - \overline{Y_{cali}} \right)^2 - \sum_{i=1}^{n} \left(Y_{obsi} - Y_{cali} \right)^2}{\sum_{i=1}^{n} \left(Y_{obsi} - \overline{Y_{cali}} \right)^2} \qquad (4)$$

The model selection criterion (MSC) is interpreted as the proportion of expected data variation that can be explained by the obtained data. Like, CD the higher the value of MSC, the higher the accuracy, validity and the good fitness of the device. MSC can be computed using equation (5) as follows:

$$MSC = ln \frac{\sum_{i=1}^{n} \left(Y_{obsi} - Y_{obs} \right)^2}{\sum_{i=1}^{n} \left(Y_{obsi} - Y_{cali} \right)^2} - \frac{2p}{n} \qquad (5)$$

Chi squared, which is the sum of the squares of the relative errors between the obtained adsorption capacity and the expected adsorption capacity can be interpreted as a measure of variation in the values expected left unexplained by the values obtained. The lower the value of Chi squared the higher the accuracy, validity and good fitness of the device. Chi squared (χ^2) can be computed as follows (Oke et al., 2008; Ismail et al., 2009):

$$c^2 = \sum_{i=1}^{n} \left(\frac{\left(Y_{obsi} - Y_{cali} \right)^2}{Y_{obsi}} \right) \qquad (6)$$

RESULTS AND DISCUSSION

Results of this study will be presented and discussed in the following ways: the adsorbents and their properties, adsorption of lead, kinetics models and statistical evaluation of the kinetics models.

The Adsorbents: Figure 2 presents the adsorbents. From the figure it can be seen that these adsorbents are available as either agricultural waste, household (domestic) waste or both, which indicates that utilization of the wastes would be a method of reducing environmental problem often caused by rapid urbanization and industrialization.

Properties of these adsorbents: the study revealed that powdered egg shells; powdered corn cobs and powdered carbon rods were made of pores, carbon, nitrogen, hydrogen, aluminum, iron and calcium, without cadmium, chromium and lead. These compositions make these materials good adsorbents. Figure 3 shows some of the micrograph structures of these important adsorbents. From these micrographs it can be seen that there are pores on the adsorbents, which are common features of adsorbents on the wastes materials. These indicate that these wastes materials can be used as adsorbents for pollutants in water and wastewater purification. More on structures of the adsorbents can be found in literature such as Oke et al (2008) ; Ismail et al (2009) ; Oke (2007) .

Adsorption of Lead onto these adsorbents: Adsorption kinetics is an important ingredient in environmental pollution control. Figure 4 (a and b) presents adsorption kinetics of lead onto these adsorbents.

In order to investigate in detail the mechanism of adsorption rate for the adsorption of Pb^{2+} onto the adsorbents, the rate constants were determined by applying the equations of Lagergren (pseudo first order), pseudo second order, Elovich and intraparticle diffusion mechanisms. Literature reported (Alam et al., 2007; Erhan et al., 2004; Yasmin et al., 2009; Olarinoye et al., 2012) that pseudo second-order adsorption kinetic rate equation is expressed as shown in equation (7):

$$\frac{dq}{dt} = k_2 \, q_e\left(-q_t\right)^2 \tag{7}$$

Yasmin et al (2009) reported that integrating equation (8) and rearrangement gives equation (8)

$$q_t = \frac{k_2 (q_e)^2 t}{1 + k_2 (q_e) t} \tag{8}$$

Equation (9) can be lineralized as:

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 (q_e)^2} + \frac{1}{q_e} t \tag{9}$$

The plot of (t/qt) and t of equation (9) should give a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot respectively. Table 1 shows more about the model. The values of k_2 and q_e *in* pseudo second-order range from 0.200 to 0.315g mg^{-1} . h and 4.928 to 6.330 mg g $^{-1}$respectively.

The intraparticle diffusion model is expressed as equation (10)

$$R = k_{id}\left(t^{a}\right) \tag{10}$$

Erhan et al (2004) and Oke et al (2008) reported that " a" depicts the adsorption mechanism and k_{id} may be taken as a rate factor (per cent pollutant adsorbed per unit time). Higher values of k_{id} illustrate an enhancement in the rate of adsorption, whereas larger k_{id} values (> 1.00) illustrate a better adsorption mechanism, which is related to an improved bonding between pollutant and the adsorbent particles. Table 1 presents more on this dynamic model. These two parameters (a and k_{id}) were found to be in the range of 0.551 to 0.595 %/ h and 17.469 to 21.434 l/h respectively. These indicate that better adsorption mechanisms and improved bonding occurred between Pb^{2+} and the adsorbents.

Statistical Evaluation of the models: The total errors for the methods were from 0.293 to 2.994 and 0.309 to 3.142 (Table 2)for non-linear and linear regression respectively. The coefficient of determination (CD) for the methods were from 0.941 to 0.991 and 0.951 to 0.992 (Table 2) for linear and non-linear regression methods respectively. Model selection criterion (MSC) for the methods ranges from 2.17 to 4.25 and 2.34 to 4.45 (Table 2) for linear and non-linear regression methods respectively. Chi squared for the methods were from 0.020 to 0.200 and 0.020 to 0.200 for linear and non-linear regression methods respectively. In summary, from the table it can be seen that in all case non-linear regression method has the least error, the highest MSC and CD, but the same range of Chi squared. This indicates that non-linear regression method provides higher accuracy, best fitting and higher validity than linear regression method. The same Chi squared indicates that there is no significant difference between the methods

Estimated Cost of Producing these adsorbents: Costs of producing these adsorbents were based on 95% yield from every dry cells collected, assuming that 320 days per year, 100 kilogram of these adsorbents were produced per day and 6 men per a shift of 8 hours. Tables 3 and 4 show the breakdown of the estimated cost. From the table average cost of producing a kilogram of these adsorbents was found to 1.404USD and 1.804 USD using public and commercial electricity sources respectively. It can then be said that the study has identified the estimated cost of 1.404 or 1.804 USD kg^{-1} of these adsorbents. The cost is dearer compared to the cost of producing empty fruit bunches (0.50USD kg^{-1}, Alam et al., 2007), but cheaper than the cost of producing pencon shell based activated carbon (2.72 USD kg^{-1}) and sugar cane based granular activated carbon by steam (3.12 USD kg^{-1}).

CONCLUSION

This study examined adsorption of Pb^{2+} onto three agricultural wastes as adsorbent. Adsorption capacities were evaluated. Kinetics of the adsorption of the adsorbate unto these three adsorbents were examined as well. In addition, techniques (linear and non- linear) for adsorption kinetics analysis were evaluated statistically. It was concluded that:

- Powdered corn cobs, Powdered egg shells and Powdered carbon rods can be used to remove lead from aqueous solutions.
- Utilization of these wastes as adsorbents will reduce solid waste disposal problems encountered in Nigeria's cities.
- Based on high MSC and CD, low total error and Chi squared, non-linear regression method is the best method to be used in the determination adsorption kinetics In the design of adsorption rectors parameters obtained by using non-linear regression method should be used to prevent failure of the reactor, although it might be argued that solutions to non-linear regression are complex, but availability of software such as SPSS, Microsoft excel, Probstat for engineers and others have minimized this limitation.

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NOMENCLATURES and SYMBOLS

χ^2	Chi squared
MSC	model selection criterion;
n	number of data points
p	number of parameters
CD	coefficient of determination;
$\underline{Y}_{cali}$-	expected values of each fitting procedure
Y_{cali}	average of obtained (experimental) values

$\underline{Y_{obsi}}$	obtained (experimental) values
Y_{obsi}	average of obtained (experimental) values
Err^2	squared total error ;
V	volume of solution (0.3L)
q_t	the adsorption capacity at time t (mg g^{-1}), the per cent pollutant adsorbed (%)
M	adsorbent mass (mg)
C_0	initial liquid-phase concentration of sorbate
C_t	experimental concentration in the solution at time t (mg l-^{1})
t	the contact time (h)
k_2	the rate constant of pseudo second-order adsorption.
k_{id}	the intraparticle diffusion rate constant (/h)
a	constant for intraparticle diffusion model

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