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Adsorption Kinetics of Pb^{2+} Removal from Aqueous Solutions Using Spent Batteries

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ABSTRACT: Lead poison and lead in drinking water sources are common reports nowadays. This paper presents a report on removal of lead ion from aqueous solution using a household waste (used batteries). Spent batteries were collected, sectioned; carbon rods were removed and ground into powder. Powdered Carbon Rods (*PCR*) were sieved into different particle sizes. Adsorption properties of the adsorbent were studied using synthetic lead solutions and typical wastewaters with a particular attention to kinetics models and cost analysis. The study revealed that kinetic models can be grouped into two based on the values of correlation coefficient (R^2) as adsorption kinetics models with R^2 less than 0.96 and adsorption kinetics models with R^2 greater than 0.96. The adsorption kinetic of Pb^{2+} onto *PCR* at different pH were 0.250 ($l\ h^{-1}$), 0.375 ($mg\ g^{-1}$) with $R^2 = 0.855$ and 0.095($l\ h^{-1}$), 0.169($mg\ g^{-1}$) with R^2= 0.992 at initial pH of 7.2 and 3.2 respectively for Pseudo first-order. Applicability of the process to typical wastewaters and raw water samples showed that *PCR* can be used as an adsorbent for Pb^{2+} removal from aqueous solutions. The study concluded that the mechanism of adsorption of Pb^{2+} onto PCR as an adsorbent followed two-steps intra-particle and pore diffusion transport. The average costs of producing a kilogram of powdered carbon rods were found to be 0.875USD and 1.018 USD for public and generator electricity sources respectively. The cost producing PCR is dearer compared to the cost of producing empty fruit bunches (0.50USD kg^{-1}), 0.068 USD kg^{-1} of powdered corn cob, cheaper compared to 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}).

Keywords: Used batteries, PCR, adsorption properties, lead removal, adsorption kinetics

Atık Pilleri Kullanarak Sıvı Çözeltilerden Kaldırılan Pb^{2+}'nin Adsorbsiyon Kinetiği

ÖZET: İçme su kaynaklarındaki kurşun ve kurşun zehri günümüzde yaygın olarak ifade edilmektedir. Bu çalışma evsel bir atık (kullanılmış pil) kullanarak, sıvı çözeltiden kurşun iyonlarının kaldırılması üzerine bir bilgi sunmaktadır. Kullanılmış piller toplanılıp kesitlerine ayrıldıktan sonra, karbon çubukları çıkarıldı ve öğütüldü. Adsorbentlerin adsorpsiyon özellikleri kinetic modeller ve maliyet anazlileri dikkate alınarak, tipik kirleticiler ve sentetik kurşun çözücüler kullanılarak çalışıldı. Korelasyon katsayısı değerlerine göre mevcut çalışma kinetic modellerin iki şekilde gruplandırılabileceğini ortaya koydu. Farklı pH değerlerinde (7.2 and 3.2) PCR üzerine Pb^{2}'nin adsorpsiyon kinetiği, sırasıyla R^2 = 0.855 de 0.250 (l/h), 0.375 (mg/g) ve R^2 = 0.992 de 0.095(l/h), 0.169(mg/g) idi. Tipik kirleticiler ve ham su örnekleri, PCR'nin sulu çözeltilerden Pb^{2}+'nin kaldırılması için bir adsorbent olarak kullanılabileceğini gösterdi. Maliyet analizleri ortlama 1 kg karbon çubukları üretmenin ortak ve ortak olmayan elektrik kaynakları için sırasıyla 0.875USD ve 1018 USD mal olacağını gösterdi. Çalışma, bir adsorbent olarak PCR üzerine Pb^{2}+ adsorbsiyon mekanizmasının iki adımda gerçekleştiği ortaya çıktı.

Anahtar kelimeler: Atık piller, PCR, adsorbsiyon özellikleri, kurşun kaldırmı, adsorbsiyon kinetikleri

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INTRODUCTION

Pollutants such as heavy metals, volatile organic compounds and dissolved solids are found in wastewaters. They are removed on adsorbents such as activated carbon, clay and sediments in riverbeds and in suspension. Over the years, the role of adsorption in wastewater and water treatment had been critically investigated. Adsorption of heavy metals onto suspended particles had been studied as a model of transportation of metals in rivers and sea. The effects of chemical composition and particle size on adsorption by suspended particles had also been studied. Adsorption of phenol, parabromophenol and benzenesulphonate by carbon had been examined by Sheindorf et al*,* (1982)*.* Adsorption of colloid by activated sludge and adsorption of bovine serum albumin by activated sludge had been investigated (Crombie-Quilty and McLoughin, 1983). Johannes and Johan (2002) reported the performance of granular activated carbon at three South African water treatment plants while Michele and Johannes (2004) gave practical experiences with granular activated carbon at the Rietvel water treatment plant.

Attention had mostly been on carbon as adsorption materials and indeed had documented not less than 37 sources of carbon, which had been studied as adsorbents for the removal of water pollutants. However, three problems associated with the use of carbon for the adsorption of pollutants in a regular volume of waters are its relatively high cost in making renovation of water by this means very expensive; the fragility of present types of activated carbon makes the regeneration and reuse of activated carbon difficult; and activated carbon is virtually limited to the removal of non–polar materials. The literature pertaining to the occurrence of lead in the environment and its relationship to human health is quite extensive and spans a wide variety of disciplines, including the fields of medicine, environmental and occupational health, toxicology, environmental geology, petrology, geochemistry, economic geology, hydrogeology, and soil science. Consequently, any attempt to summarize the state of knowledge concerning lead and environmental health is necessarily limited to a broad overview. Lead in wastewater comes mainly from the discharge of battery manufacturing, printing, dying and other industries. Other major sources of lead in the environment include lead-based paint, household dust and food containers. Lead has been found to be acute toxic to human beings when present in high amounts in water. Studies have shown that young children, infants and pregnant women are particularly susceptible to unsafe lead levels. For adults, increased levels of lead have been linked to high blood pressure and damaged hearing. Drinking, eating, inhaling even at low level of lead can cause other serious health effects. However, most technologies presently employed for lead removal are based on physicochemical processes, such as dilution, adsorption, coagulation and flocculation, chemical precipitation, oxidation, ion-exchange, reverse osmosis and ultra-filtration . Among these processes, adsorption technology is attractive for the lead treatment from the wastewater, due to its easy availability, simplicity of design, high efficiency, ease of operation, and ability to treat dyes in more concentrated form. The importance of adsorption as water purification process has been documented in literature. Ho (2007) reported that about 9058 articles have been published on adsorption of materials.

These indicate that adsorption process is an important process in environmental pollution control, but adsorption of lead onto powdered carbon from used dry cells is rare in literature, which calls for documentation of lead adsorption onto this house hold waste considering effects of lead on human's health and on the environment and availability of dry cells as solid waste in developing countries such as Nigeria (Oke et al*.,* 2007 a and b); Iran (Zand and Abduli, 2008; Almeida et al., 2009). The main aim of this study was to investigate removal of lead from synthetic water using house hold waste of carbon rods from dry cells.

MATERIALS AND METHODS

Spent (Used) dry cells (R_{20S} Size D UM-1) were collected from 2002 to 2009 from dustbins, solid waste dump site and street collections. The batteries (dry cells) were characterized, dismantled (sectioned vertically) and the carbon rod (cathode) were removed, washed with distilled water and air dried. The cathodes removed were characterized and ground into powder. Powdered Carbon Rods *(PCR)* were sieved into different particle sizes. Properties of the powdered carbon rods (moisture content, water solubility, acid solubility, ash, and volatile solid and metallic contents) were determined. Structures (micrograph, presented in another article) of the adsorbent were examined to ascertain its nature and porosity. Adsorption properties of the adsorbent were studied using synthetic lead solution and typical wastewaters (raw water and domestic institutional wastewater) with a particular attention to kinetics models. Cost analysis of PCR processing was conducted based on literature and presented economics. *PCR* was selected based on availability in developing countries as solid wastes.

Moisture content: a well mixed samples of PCR were evaporated in a weighed dishes to a constant weight in an oven at 105°C (APHA, 1998). In detail, the samples were weighed initially, dried in the oven at 105 ^{0}C for 24 hours, cooled in desiccators and reweighed. The decreased in the weight of the PCR represents the moisture content as follows:

$$M_c\,(\%) = 100\left(\frac{W_1 - W_2}{W_1}\right) \tag{1}$$

Volatile solid and ash content: Known masses of dried samples used for moisture content determination were placed in crucible dishes and transferred into a muffle furnace. The furnace was heated to 550°C for 2 hours (APHA, 1998). The samples were cooled in desiccators to balance the temperature and the final weights were measured. Volatile solid and ash content of PCR were calculated as follows (APHA, 1998):

$$VS\,(\%) = 100\left(\frac{W_2 - W_3}{W_2}\right) \tag{2}$$

$$Ash\,(\%) = 100\left(\frac{W_3}{W_1}\right) \tag{3}$$

Water and acid solubilities: Known dried masses of the samples were soaked in 300 ml of distilled water and in 300 ml of 0.25 M of HCl(hydrochloric acid) for 24 hours respectively. The samples were filtered out using pre-dried and weighted filter paper (Number 1). The samples and the filter paper were dried in the oven at 105°C for 24 hours, cooled in desiccators to balance the temperature and the final weights were measured. Water and acid solubilities of PCR were calculated as follows (APHA, 1998):

$$WS(\%) = 100\left(\frac{W_2 - W_4}{W_2}\right) \tag{4}$$

$$AS\,(\%) = 100\left(\frac{W_2 - W_5}{W_2}\right) \tag{5}$$

Metal concentrations: A known mass (1.0g) of the PCR was digested using nitric acid digestion method (APHA, 1998) and chemical components of the adsorbent were determined using standard methods (APHA, 1998). Specifically,1.0 gram of PCR was digested using nitric acid and total metal concentrations were determined using spectrometry method. Metal concentrations were computed as follows:

$$M_c\,(mg/L) = \left(\frac{A * B}{W_1}\right) \tag{6}$$

In the preparations of lead solutions, procedures specified in Standard Methods for the Examination of Water and Wastewater (APHA, 1998) were followed. Specifically a known mass (1.599g) of lead nitrate ($Pb(NO_3)_2$) was dissolved in 200ml of distilled water, 10 ml of concentrated HNO_3 was added, 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 a known concentration (mg/L) of Pb^{2+} were taken into five different beakers and known masses (0.3, 0.5, 0.7, 1.0 and 1.2g) of the adsorbent were added at a known initial pH. The mixture was stirred at 60 revolutions per minute (rpm) for 2 minutes and allowed to settle for 18 hours (when equilibrium concentrations have been reached). The supernatant of the samples taken were filtered through Whatman filter paper number 40 to prevent particulate. Determinations of lead concentrations were conducted using spectrophotometer method (APHA, 1998) and spectrophotometer facilities available at Central Science Laboratory Obafemi Awolowo University, Ile-Ife, Nigeria. Kinetics parameters and percentage lead adsorbed were computed by using equations (7 and 8) as follows:

$$q_t = \frac{(C_0 - C_t)}{M} V \tag{7}$$

$$R_R(\%) = 100\frac{(C_0 - C_t)}{C_0} \tag{8}$$

Effects of pH, initial Pb^{2+} concentration and particle size of the PCR on adsorption kinetic were studied.

RESULTS AND DISCUSSION

The results of this study have been presented and discussed in the following ways: properties of cathode, adsorption properties (kinetics models) of Pb^{2+}onto *PCR* and cost analysis of producing PCR.

Types of Spent Batteries: Figure 1 (a and b) presents various R_{20S} Size D UM-1 batteries collected. Out of about 150, 000 spent batteries collected the orders of their numbers are as follows: Tiger > flash > Xion Jian > Berec (England and Nigeria) > Duracell > others. Figure 1(c_i and c_{ii}) presents vertical section (dismantled) batteries as presented in another paper (Oke et al., 2010).

Characterisation of the cathode: Table 1 presents physical properties and chemical composition of the cathode. From the table it can be seen that it contains no cadmium (Cd), arsenic (As), chromium (Cr) chloride (Cl^-), sulphate (SO_4^{2-}), lead (Pb^{2}+), manganese (Mn) and zinc (Zn). This indicates that utilization of PCR as an adsorbent cannot impose any danger or harm on the environment and human's health rather it will help in cleaning the environment as follows:

- Removal of Pb^{2+} by PCR (as it is known that lead is toxic and harmful to man);
- Removal of spent batteries reduces solid wastes disposal problem as in developing countries there is no law against disposing the waste in the municipal waste. Literature such as Almeida et al (2006) present properties of AA household alkaline batteries (Table 1a).

Adsorption kinetics: Adsorption kinetics information is the most important in understanding adsorption processes. No matter how many components are present in the system, the adsorption kinetics models are the essential ingredients for an understanding of how much of those components can be accommodated by a solid adsorbent. This information can be used to study the adsorption kinetics of a single component, adsorption equilibria of the systems, and then adsorption kinetics of multi-component systems. The adsorption data of Pb^{2+}on the *PCR* were obtained at different particle sizes, pH, initial concentration of Pb^{2+}, stirring speed, contact time and temperatures. The data obtained were replicated and the averages were used for the adsorption kinetics. Oke et al (2008) reported that the nature of the adsorption reaction can be described by relating the adsorption capacity (mass of solute adsorbed per unit mass of adsorbent) to the concentration of the solute remaining in the solution. Such a relation is known as adsorption kinetics (dynamics). There are many basic adsorption kinetics models, which include frequently used kinetics models such as pseudo first and second orders, Elovich and intra particle diffusion models (Alam et al*.,* 2007; Oke et al., 2008; Yasmin et al., 2009); solid diffusion, driving force, Vermeule; and Nakao and Suzuki models (Ryu et al*.,* 2001). Zero order, first order, second order and Fractional power (Yasmin et al., 2009).

Figures 2 - 7 present the relationship between adsorption capacity and contact time under various operational conditions. From the Figures, it can be seen that the relationship between adsorption capacity and time for Pb^{2+} removal by *PCR* was of two parts. The first part is linear and the latter part is logarithmic (curve) for synthetic solutions and typical wastewater (raw water and domestic institutional wastewater). This behavior can be attributed to solute – solute competition, the solute –surface interaction, ionic radii, hydration capacity, pH, electro negativity and availability of sites on the adsorbent (Ulamn et al*.,* 2003). In order to investigate in detail the mechanism of adsorption rate for the adsorption of Pb^{2+} onto *PCR,* the rate constants were determined by applying the equations of Lagergren (pseudo first order), pseudo second order, Elovich and intraparticle diffusion mechanisms. The pseudo first-order equation is generally expressed as follows:

$$\frac{dp}{dt} = k_i\left(q_e - q_t\right) \qquad (9)$$

After integration and applying boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, the integrated form of Equation (9) becomes (Alam et al*.,* 2007; Oke et al., 2008; Yasmin et al., 2009):

$$q_t = q_e\left(1 - \exp^{\frac{k_i}{2.303}t}\right) \qquad (10)$$

$$\log_e\left(q_e - q_t\right) = \log_e\left(q_e\right) - \frac{k_i}{2.303}t \qquad (11)$$

The values of $\log_e (q_e - q_t)$ were linearly correlated with contact time (*t*). The plot of $\log_e (q_e - q_t)$ against contact time should give a linear relationship from which k_i and q_e can be determined from the slope and intercept of the graph, respectively. Tables 2 - 5 and Figure 8 show the pseudo-first order model and their values, respectively. These values range from 0.045 to 0.250 L h^{-1} and 0.149 to 0.375 mg g^{-1} for k_1 and $q_{e,}$

respectively. Literature reported (Alam et al., 2007; Erhan et al., 2004; Oke et al., 2008; Yasmin et al., 2009) that pseudo second-order adsorption kinetic rate equation is expressed as shown in equation. (12):

$$\frac{dp}{dt} = k_2\left(q_e - q_t^{\,2}\right) \qquad (12)$$

Yasmin et al 2009 reported that integrating equation (12) and rearrangement gives equation (13)

$$q_t = \frac{k_2 (q_e)^2 t}{1 + k_2 (q_e) t} \qquad (13)$$

Equation (14) can be lineralized as:

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 (q_e)^2} + \frac{1}{q_e} t \qquad (14)$$

The plot of (*t/qt*) and *t* of Equation (14) should give a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot respectively. Tables 2 - 5 and Figure 9 show more data about this adsorption kinetic model. The values of k_2 and q_e *in* pseudo second-order range from 0.749 to 14.795 g mg^{-1} h and 0.134 to 0.621 mg g^{-1} respectively. The Elovich model equation is generally expressed as indicated in equation (15):

$$\frac{dq_t}{dt} = a \ \exp -\left(b q_t\right) \qquad (15)$$

Oke et al 2008 simplify Elovich equation as shown in equation (16)

$$q_t = \frac{1}{b} \log_e \left(ab\right) + \frac{1}{b} \log_e \left(t\right) \qquad (16)$$

If lead adsorption fits the Elovich model, a plot of q_t against. $\log_e$ (*t*) should yield a linear relationship with a slope of (1/ β) and an intercept of (1/ β) $\log_e$ (αβ). Tables 2-5 and Figure 10 presents more data on this adsorption dynamic model.

For Pb^{2+} solutions the values of β and α were found to be in the range of 7.722 to 10.084 g mg^{-1} and 0.381 to 0.638 mg g^{-1}.h respectively. The intraparticle diffusion model is expressed as Equation (17)

$$R_R = k_{id}\left(t^a\right) \qquad (17)$$

$$F = 1 - \frac{6}{\Pi^2} \exp -\left(B_b t\right) \qquad (18)$$

$$F = \frac{q_t}{q_e} \qquad (19)$$

q_e and q_t represent the amount of Pb^{2+} sorbed (mg g^{-1}) at equilibrium and any time t, respectively. To compute $B_b t$, equation (20) is substituted into Equation (19) and the kinetic expression becomes

$$B_b t = -0.4977 - \mathrm{h}\left(1 - \frac{q_t}{q_e}\right) \qquad (20)$$

CONCLUSIONS

It can be concluded based on the study that:

- PCR can be used to remove Pb^{2+} from aqueous solution, typical raw water and wastewaters
- the mechanism of adsorption of Pb^{2+} onto *PCR* as an adsorbent follows two-steps, namely intra-particle and pore diffusion transport.

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SYMBOLS

M the mass of the adsorbent added (g),

R_R the percentage pollutant adsorbed (%);

V the volume of the sample used (300ml) = 0.3L;

q_e the equilibrium solid-phase concentration of sorbate (mg mg^{-1});

q_t the solid-phase concentration of sorbate at t time (mg mg^{-1});

C_0 the initial concentration of arsenic in the solution (mg l^{-1});

C_e the experimental concentration in the solution at equilibrium (mg l^{-1})

C_t the experimental concentration in the solution at time t (mg l^{-1}).

k_i the pseudo first-order constant,

k_2 the pseudo second -order constant

α and β Elovich constants

k_{id} and a intraparticle diffusion constants ,

t treatment time .

B_b constant

F the fractional attainment of equilibrium at time t

DI domestic institutional wastewater;

RW raw water.

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