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Adsorption of Cd(II), Cu(II) and Pb(II) using recycled waste glass: equilibrium and kinetic studies

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ABSTRACT

Recycled waste glass (RWG), from municipal solid wastes (MSW) sorting operations, may be used as a sorbent for cadmium, copper and lead ions removal from industrial wastewaters. To the purpose, the ability of RWG to adsorb Cd^{2+}, Cu^{2+} and Pb^{2+} from aqueous solutions has been studied at different operating conditions: contact time, adsorbent amount and metal ion concentration. Batch adsorption kinetic experiments revealed that the sorption of Cd^{2+}, Cu^{2+} and Pb^{2+} on RWG was very fast and the equilibrium was practically reached after only 60 min at constant temperature and pH (5.6 ± 0.1). It was found that the adsorption mechanisms follow pseudo-second-order kinetics. The adsorption isotherm studies indicate that the adsorption of Cd^{2+}, Cu^{2+} onto RWG follows Langmuir isotherms, while Pb^{2+} can follow Langmuir and Freundlich models. The maximum adsorption capacity (q_{max}) for Cd^{2+}, Cu^{2+} and Pb^{2+} were 6.29, 6.68 and 11.68 mg/g, respectively. From Dubinin–Kaganer–Radushkevich (DKR), the free energy E-value for Cd^{2+}, Cu^{2+} and Pb^{2+} is 14.067, 18.467 and 19.724 kJ/mol, respectively. The positive values of E indicate that the sorption process is endothermic and the energy values for the studied metals sorption on RWG indicate that the sorption process is physisorption.

Keywords: Adsorption; Heavy metals; Kinetics; Langmuir, Freundlich and Dubinin–Kaganer–Radushkevich isotherm

1. Introduction

Environmental problems have become more frequent and complex in recent decades, as a result of human population growth and increasing industrialization [1]. Industrial activities such as mining and metal processing can lead to heavy metal contamination in surface water, groundwater, or the sea [2,3]. Heavy metals are the main hazardous nondegradable substances, persistence and can be accumulate in the environment elements such as food chain, thus may pose a significant danger to human health [4,5].

There are various methods for removing heavy metals including chemical precipitation, membrane filtration, ion exchange, liquid extraction or electrodialysis [6]. However, these methods are not widely used due to their high cost and low feasibility for small-scale industries [7].

The widespread industrial use of low-cost adsorbents for wastewater treatment is strongly recommended at present, due to their local availability, technical feasibility,

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engineering applicability and cost effectiveness [8–10]. Reuse of large amounts of waste glass from municipal solid wastes (MSW) sorting operations is a problem in most developed countries. Glass is a versatile material for its safe and durable use which can be easily reused in the same and/or in related activities with minor (if any) modifications [11–16]. Nonetheless glass is still dumped into landfills and, due to its essential bio-refractory and chemical inertia, leads to long-term accumulation in the environment. In this context, glass recycling operations contribute to waste minimization and promote interest in the recovery operations according to the current legislation [17]. Recycled waste glass (RWG) may be assumed as ‘‘secondary raw material” due to its easy re-shaping in different forms. Therefore, the objective of this investigation is to study the synergistic effect of initial metals concentration, adsorbent dosage and agitation time on removal of Cd(II), Cu(II) and Pb(II) ions using RWG adsorbent; to study the adsorption kinetics models of Cd(II), Cu(II) and Pb(II) ions examining the change in adsorption rate with time; to use the adsorption isotherms models (Langmuir, Freundlich and Dubinin–Kaganer–Radushkevich (DKR)) in order to correlate the experimental results.

2. Materials and methods

2.1. RWG paterials preparation

RWG was recovered from MSW as well as industrial waste sorting operations. Preliminary, clean and crush of the raw materials. Finally, granulated, sieved and passed through 80 μm sieves.

For preservation, they were kept in plastic stopper bottles (containers) and to minimize contact with humidity all these bottles were preserved in desiccators before the time of use.

2.2. Metal solutions

Salts used in the preparation of the synthetic metal bearing solutions were of Merck -analytical grade: $CdCl_2{\cdot}5/2H_2O$, $CuSO_4 . 5H_2O$ and $Pb(NO_3)_2$. Deionized water was used in all experiments. The synthetic wastewater solutions were then prepared by diluting the stock standards of concentration 1000 mg/L of each.

2.3. Analytical methods

2.3.1. Determination of metals concentration

The concentrations of metals in all samples were determined according to [18] using Atomic Absorption Spectrometer (Varian SpectrAA (220)) with graphite furnace accessory and equipped with deuterium arc background corrector. Precision of the metal measurement was determined by analyzing (in triplicate) the metal concentration of all samples.

2.3.2. Quality control

For each series of measurements absorption calibration curve was constructed composed of a blank and three or more standards from Merck (Germany). Accuracy and precision of the metals measurement were confirmed using external standard reference material 1643e for trace elements in water and quality control sample from National Institute Standards and Technology (NIST).

2.4. Batch sorption studies

Each of the batch sorption studies was carried out by contacting the waste glass (adsorbent) with the metal ions at pH 5.6 ± 0.1 in a glass tube. Studies were conducted at room temperature (25 ± 1.0 ºC) to determine the effects of sorbent dosage, contact time and initial ions concentration on the adsorption of Cd(II), Cu(II) and Pb(II) ions. Each experiment was conducted in a mechanical shaker at 120 rpm. All samples were filtered through Whatman filter paper (No. 41) and the metal ions concentration was determined in the filtrate. To distinguish between possible metal precipitation and actual metal sorption, controls were used without sorbent materials.

All the experiments were carried out in triplicate and the mean of the quantitative results were used for further calculations. For the calculation of mean value, the percent relative standard deviation for results was calculated and if the value of standard deviation for any sample was greater than 5% the data were not used.

2.4.1. Sorption kinetics

The kinetic studies were carried out by conducting batch sorption experiments with initial metals concentration of 50 mg/L. Samples were taken at different time periods. Various models were used to analyze the kinetics of the sorption process [19].

2.4.2. Effect of sorbent dosage

The sorbent dosage was varied from 0.1 to 1.00 g using a fixed volume of 100 ml of 50 mg/L of metal solution at the equilibration time for each metal.

2.4.3. Sorption isotherms

Sorption isotherms were measured by varying the initial metal ion concentrations with keeping all other parameters constant. Different sorption models were used for comparison with experimental data [20].

3. Results and discussion

3.1 Characterization of recycled waste glass

The chemical analysis of the RWG was illustrated in Table 1 which reveals that the main content of RWG is 69.506% SiO_2, 10.69% CaO and minor amount of alumina reaching 0.668% Al_2O_3. On the other hand, RWG was subjected to crushing and severs grinding processes; the size distribution of the ground sample is shown in Fig. 1, where more than 90% of the particle size was 80 μm

The Zeta potentials of RWG as a function of pH (ionic strength 10^{-3} M NaCl) are illustrated in Fig. 2. It is clear that the surface charge of RWG is pH dependent. As is evidenced by the negative value of Zeta potential, the surfaces of RWG are negatively charged in the wide pH range of 2–11, and the negative Zeta potential increases with increasing pH in this range, indicating that the quantity of negatively charged functional groups on the RWG surface increases with the increasing of pH [21–23]. The IEP (iso electric point) for the sample was observed at pH 1.5, Fig. 2.

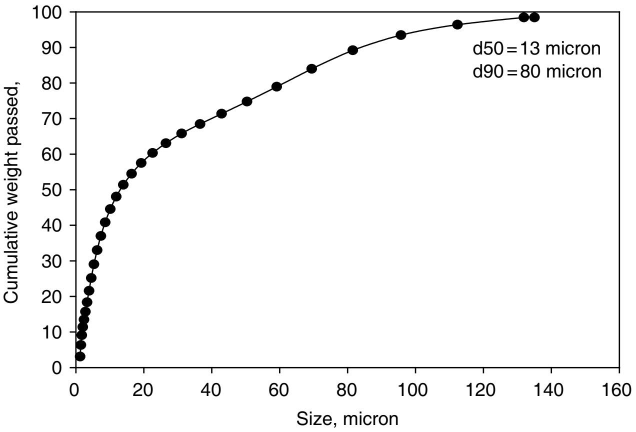

Fig. 1. Size distribution analysis of the ground recycled waste glass.

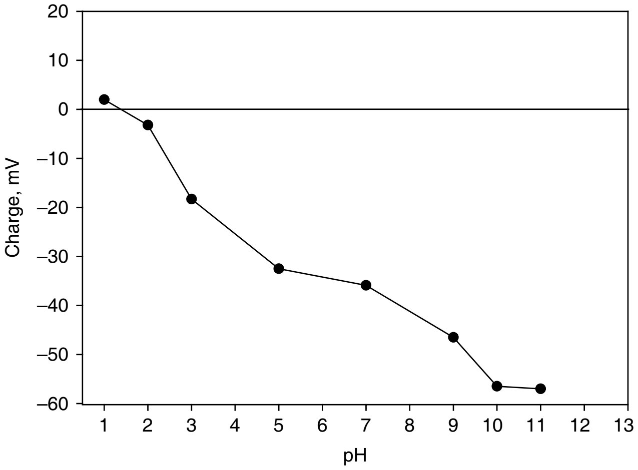

Fig. 2. Electro-kinetic measurement for ground recycled waste glass.

Table 1
Chemical analysis of recycled waste glass

Constituent	Wt. %
Na_2O	13.76
MgO	3.146
Al_2O_3	0.668
SiO_2	69.506
P_2O_5	0.041
SO_3	0.373
K_2O	0.203
CaO	10.69
TiO_2	0.161
Cr_2O_3	0.020
MnO	0.023
Fe_2O_3	0.44
SrO	0.030
Cl	0.127
LOI*	0.8

Note: LOI: loss on ignition.

The studies demonstrated the importance of the oxide surface such as SiO_2, which present the main constituent of RWG. As the interaction between water and oxide surface may result in the development of surface hydroxyl groups show an amphoteric character and surface charges on the oxides/hydroxides are developed according to the mechanisms [24]

$$GOH_2^+ \Leftrightarrow GOH + H^+$$

$$GOH \Leftrightarrow GO^- + H^+$$

where, G is the RWG surface.

3.2. Kinetic modeling – sorption kinetics

As equilibrium time is one of the important parameters for selecting a wastewater treatment system. Where the time consumed for wastewater disposal should be considered. As shown in Fig. 3 the sorption of Cd(II), Cu(II) and Pb(II) ions onto (RWG) was rapid for the first 30 min and equilibrium was reached within 60 min. Therefore, the period of 60 min was considered as the optimum time, where the removal percentages followed the order: Pb (95%) > Cd (94.5%) > Cu (86.3%).

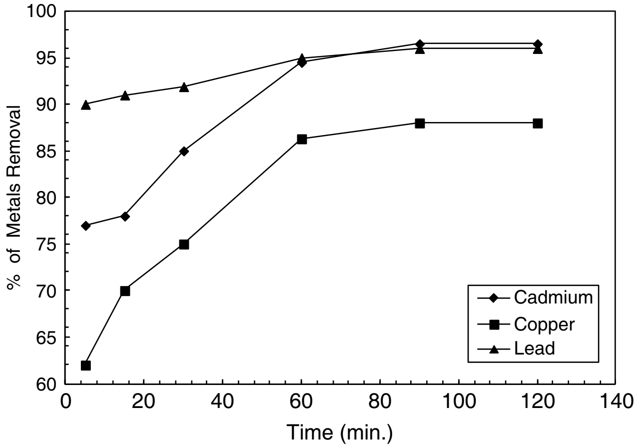

Fig. 3. Effect of contact time on the metal removal by adsorption onto recycled waste glass at an initial concentration 50 mg/L, pH 5.6 ± 0.1 and 10 g/L adsorbent weight.

Kinetic characteristic in a sorbent depends not only on the presence of active metal site but also depends on the accessibility of the metal site without sterical hindrance which is greatly determined by the matrices of the sorbent. This means that, the mechanism of sorption depends on the physical and chemical characteristics of the sorbent as well as on the mass transfer process. Therefore, the rate kinetics of Cd, Cu and Pb ions sorption onto (RWG) were analyzed using pseudo-first-order, pseudo-second order and Elovich kinetic model, which are the most commonly used models [20]. The conformity between experimental data and the model-predicted values was expressed by the correlation coefficients (R^2, values close to 1). The relatively higher value is the more applicable model to the kinetics of Cd, Cu and Pb ions sorption.

3.2.1. Pseudofirst-order kinetic model

The kinetic data were modified with the Lagergren first-order model [25] which is the earliest known one describing the adsorption rate based on the adsorption capacity. The integral form of the pseudo-first-order model generally expressed as follows:

$$log\,(q_e - q_t) = log\,q_e - K_{1,ads} \times t\ 2.303 \qquad (1)$$

where q_e (meq/g) and q_t are the amounts of adsorbed metal ions on the biosorbent at the equilibrium and at any time t, respectively; and $K_{1,ads}$ is the Lagergren rate constant of the first-order sorption (min^{-1}).

The model is based on the assumption that the rate is proportional to the number of free sites. If the pseudo-first-order kinetics is applicable, a plot of log($q_e - q_t$) versus t should provide a linear relationship from which $K_{1,ads}$ and predicted q_e can be determined from the slope and intercept of the plot, respectively (Fig. 4). The variation in rate should be proportional to the first power of concentration for strict surface adsorption. However, the relationship between initial solute concentration and rate of adsorption will not be linear when pore diffusion limits the adsorption process.

It was observed from Fig. 4 and Table 2 that first-order model failed to provide a realistic estimate of q_e of adsorbed Cd, Cu and Pb ions onto (RWG). As the experimental values of q_e (0.074, 0.1265 and 0.04286 meq/g) were higher than the fitted value (0.027, 0.036 and 0.00664 meq/g) for Cd, Cu and Pb ions, respectively. This underestimation of the amount of binding sites is probably due to the fact that qe was determined from

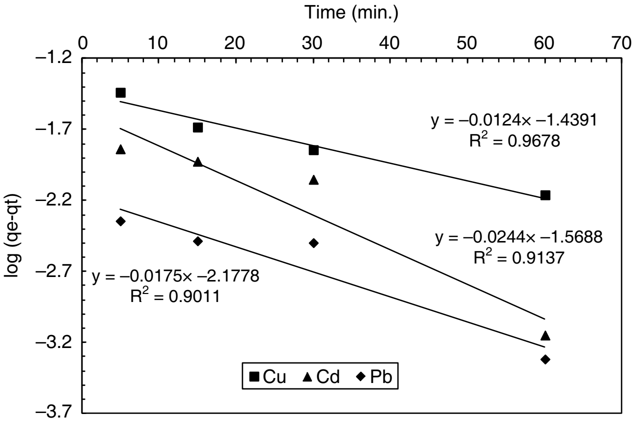

Fig. 4. Pseudo-first order for the sorption of Cd(II), Cu(II) and Pb(II) onto recycled waste glass at 25 °C and pH 5.6.

Table 2
Pseudo-first and -second order rate constants and equilibrium metal uptake for Cd(II), Cu(II) and Pb(II) binding by recycled waste glass

Metals	q_e Exp. (meq/g)	First-order kinetic			Second-order kinetic		
		$K_{1,ads}$	q_e model (meq/g)	R^2	$K_{2,ads}$	q_e Model (meq/g)	R^2
Cd	0.074	0.056	0.027	0.9137	4.523	0.0758	0.999
Cu	0.1265	0.083	0.036	0.9678	2.14	0.13	0.999
Pb	0.04286	0.04	0.00664	0.9011	15.24	0.0434	0.999

the *y*-intercept ($t = 0$). The intercept is most strongly affected by the short-term metal uptake which is usually much lower than the equilibrium uptake. It can be concluded that this is a general disadvantage of using the linearized first-order model. On the other hand, the experimental q_e values do not agree with the calculated ones which obtained from the linear plots even when the correlation coefficient R^2 are relatively high. This showed that the adsorption of Cd, Cu and Pb ions onto RWG is not appropriate to describe the entire process and not a first-order reaction.

3.2.2. Pseudo-second-order model

The kinetics of adsorption process may also be analyzed by pseudo-second order rate equation [26]. The pseudo-second-order model is based on the assumption that sorption follows a second-order mechanism, whereby the rate of sorption is proportional to the square of the number of unoccupied sites. The linearized form of the equation is expressed as:

$$\frac{t}{q_t} = \frac{1}{K_{2,\mathrm{ads}} \times q_\mathrm{e}^2} + \frac{t}{q_\mathrm{e}} \qquad (2)$$

where $K_{2,\mathrm{ads}}$ is the rate constant of second-order sorption (g/meq min).

The linearized second-order plot of t/q against t (Fig. 5) according to Eq. (2) resulted in straight lines for Cd, Cu and Pb ions and led to the determination of the second-order rate constants $K_{2,\mathrm{ads}}$ and q_e from the slope and the *y*-intercept (Table 2). The q_e values were very close to the experimentally determined ones, a first sign of the appropriateness of this model. The R^2 values for the second-order kinetic model were 0.999 for the adsorption of Cd, Cu and Pb ions onto RWG (Table 2). Our results in agreement with [27], who determined that a second-order model fit better than a first-order model for sorption of divalent metals by oxides/hydroxides surface.

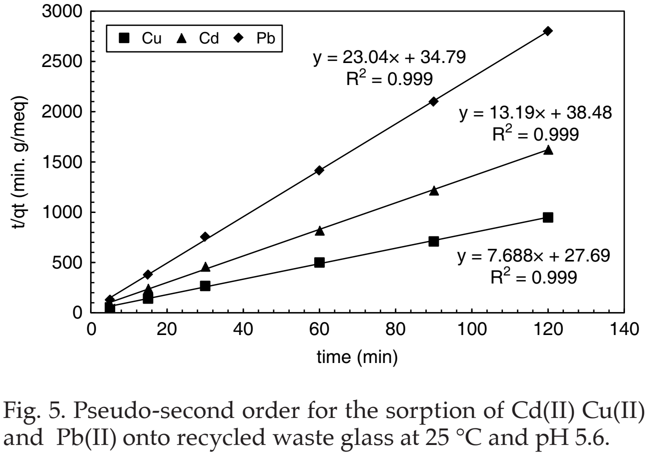

Fig. 5. Pseudo-second order for the sorption of Cd(II) Cu(II) and Pb(II) onto recycled waste glass at 25 °C and pH 5.6.

Based on theoretical considerations, the reaction of a divalent metal ion (M) binding to two free binding sites (B) can be explained by the following expressions:

$$\mathrm{M} + 2\mathrm{B} \leftrightarrow \mathrm{B}_2\mathrm{M} \leftrightarrow r = k\,[\mathrm{M}]\,[\mathrm{B}]^2$$

This means, the sorption rate would be proportional to the metal concentration and the square of the number of free sites, which corresponds to the term $(q_e - q)^2$ in the second-order model. The better fit of the second-order model therefore indicates that a 1:2 binding stoichiometry applies, where one divalent metal binds to two monovalent binding sites.

3.2.3. Elovich kinetic model

Elovich kinetic equation is another rate equation based on the adsorption capacity, which is generally expressed as [28]:

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t) \qquad (3)$$

where α is the initial adsorption rate (mg g^{-1} min^{-1}) and β (gmg^{-1}) is related to the extent of surface coverage and activation energy for chemisorption during any one experiment.

It is simplified by assuming $\alpha\beta t \gg t$ and by applying the boundary conditions $q_t = 0$ at $t = 0$ and $q_t = q_t$ at $t = t$. Eq. (3) becomes form as followed [29]:

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln(t) \qquad (4)$$

If Cd(II), Cu(II) and Pb(II) adsorption by (RWG) fits the Elovich model, a plot of q_t versus $\ln(t)$ should yield a linear relationship with a slope of $(1/\beta)$ and an intercept of $(1/\beta)\times\ln(\beta\alpha)$ (Fig. 6). Thus, the constants can be obtained from the slope and the intercept of the straight line. Correlation coefficients R^2 obtained by Elovich model were higher than that obtained from pseudo-first-order model and comparable to that obtained from pseudo-second-order model. Therefore, it was suggested that diffusion accounted for the Elovich kinetics pattern [30]; conformation to this equation alone might be taken as evidence that the rate-determining step is diffusion in nature [31] and that this equation should apply at conditions where desorption rate can be neglected [32]. The kinetic curve of sorption demonstrated good fitting with the model where R^2 values were 0.989, 0.9249 and 0.911 for the adsorption of Cu, Cd and Pb ions onto RWG which may indicate that the diffusional rate-limiting is

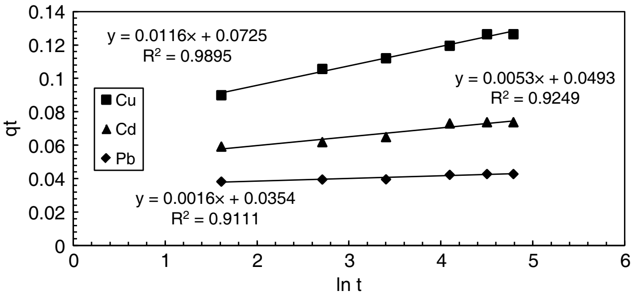

Fig. 6. Elovich model plot for the adsorption of Cd(II), Cu(II) and Pb(II) using recycled waste glass at 25 °C and pH 5.6.

more prominent for the sorption of the studied metals by RWG.

3.3. *Effect of adsorbent dose*

Various amounts of adsorbent (RWG) ranging from 1 to 10 g/L were used (Fig. 7). The percentage removal (*A*%) of Cd(II), Cu(II) and Pb(II) ions varied linearly with the amount of the adsorbent and amount of adsorbate. The increase in *A*% with an increase in adsorbent dosage is due to the availability of larger surface area and more adsorption sites. The optimum dosage of adsorbent was 8 g/L for Pb(II) and 9 g/L for Cu(II) and Cd(II) . Furthermore, it was observed that the incremental of metal removal becomes very low, as the surface of metal ions concentration and the solution of metal ions concentration come to equilibrium with each other [33]. Thus, increased adsorbent dosage did not enhance the removal percent of the studied metal ions.

3.4. *Equilibrium studies and isotherm modeling*

3.4.1. *Langmuir and Freundlich models*

The equilibrium adsorption isotherms are one of the promising data to understand the mechanism of the adsorption. Various isotherm equations are well known and three different isotherms are selected in this study which are the Langmuir, Freundlich, and Dubinin–Radushkevich (DKR) isotherms [34].

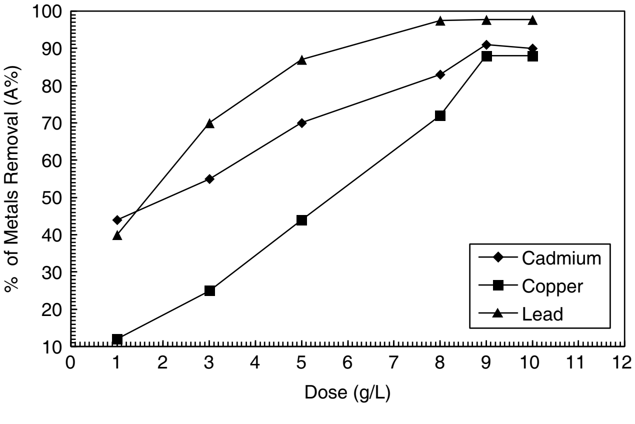

Fig. 7. Effect of adsorbent dose on the removal of Cd(II), Cu(II) and Pb(II) using recycled waste glass at 25 °C and pH 5.6.

The uptake of Cd(II), Cu(II) and Pb(II) onto RWG as a function of their concentrations was studied by varying the metal concentrations from 100 to 400 mg/L while keeping all other parameters constant with respect to optimum dose, time for each metal, temperature and pH. Blank tests without sorbent were performed to confirm that metal precipitation did not interfere with metal sorption.

Due to their simplicity, the Langmuir and Freundlich equations are the most widely used models to describe the relationship between equilibrium metal uptake (q_e) and final concentrations (C_e) at equilibrium.

The Langmuir isotherm relationship is given as

$$q_e = \frac{Kq_{\max}C_e}{(1 + KC_e)} \tag{5}$$

where, *K* (L/g) is the equilibrium adsorption constant which is related to the affinity of the binding sites and $q_{\max}$ (mg/g) is the maximum amount of metal ion per unit mass of biosorbent when all binding sites are occupied.

The Langmuir parameters can be determined from a linearized form of Eq. (5) (by plotting C_e/q_e versus C_e), represented by

$$\frac{C_e}{q} = \frac{1}{Kq_{\max}} + \frac{C_e}{q_{\max}} \tag{6}$$

The Freundlich equation is given by:

$$q_e = K_f C_e^{1/n} \tag{7}$$

where K_f and *n* are the Freundlich constants and are related to the adsorption capacity of the sorbent and the adsorption intensity. To simplify the determination of K_f and $1/n$, Eq. (7) can be linearized in logarithmic form, which allows the determination of the unknown parameters by plotting log qe versus log C_e:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \tag{8}$$

The choice between Langmuir and Freundlich isotherms depends mainly on the nature of equilibrium data [35]. Where, the adsorption phenomena at the solid–liquid interface were commonly described by the adsorption isotherm model, and adsorption isotherms are essential data source for practical design of adsorption systems and understanding of relation with adsorbent and adsorbate [36]. Langmuir and Freundlich isotherm parameters are listed in Table 3. Langmuir isotherm assumes monolayer adsorption, the R^2 values for all the metal ions were very close to 1, which revealed the extremely good applicability of the Langmuir model to these adsorptions, which indicating that Langmuir isotherm model was good for describing the experimental data of metal sorption experiments. The Freundlich model has generally been considered as an empirical equation based on adsorption on a heterogeneous surface and has also been used widely to fit experimental data [37]. Compared with Langmuir isotherm, all the R^2 values of Freundlich model were less than 0.95, except for Pb^{2+} ions where the isotherms of adsorption obtained are in agreement with the models of Langmiur and Freundlich in the whole range of the concentration studied. Similar results were obtained by Tran et al. [38] for his studies of sorption of heavy metals using silica network.

According to the maximum adsorption capacities (Table 3), the selectivity sequence can be given as $Pb^{2+} > Cu^{2+} > Cd^{2+}$. Lead ions have a preferential uptake compared to the other metals. This may be attributed to low tendency of lead ions to form strong complex although it can form hydrated ions [39] (Fig. 8).

3.4.2. Dubinin–Kaganer–Radushkevich isotherms (DKR isotherms)

Langmuir and Freundlich isotherms do not give any idea about sorption mechanism. The DKR isotherm is an analogue of Langmuir type but it is more general because it does not assume a homogeneous surface or constant sorption potential [40]. The Dubinin–Radushkevich [41] isotherm model was used to predict the nature of adsorption processes as physical or chemical.

The linearized DKR isotherm equation can be written as shown:

$$\ln q_{\mathrm{e}} = \ln X_{\mathrm{m}} - \beta \varepsilon^2 \quad (9)$$

where q_e is the number of metal ions adsorbed per unit weight of adsorbent (mol/g), X_m is the maximum

Table 3
Summary of isotherm model parameters for recycled waste glass

Metals	Langmuir model			Ferundlich model		
	K	q_{max} (mg/g)	R^2	K_f	n	R^2
Cd	1.88	6.29	0.971	1.56	4.78	0.845
Cu	1.57	6.68	0.967	2.9	8.66	0.489
Pb	12.71	11.68	0.998	5.58	8.24	0.998

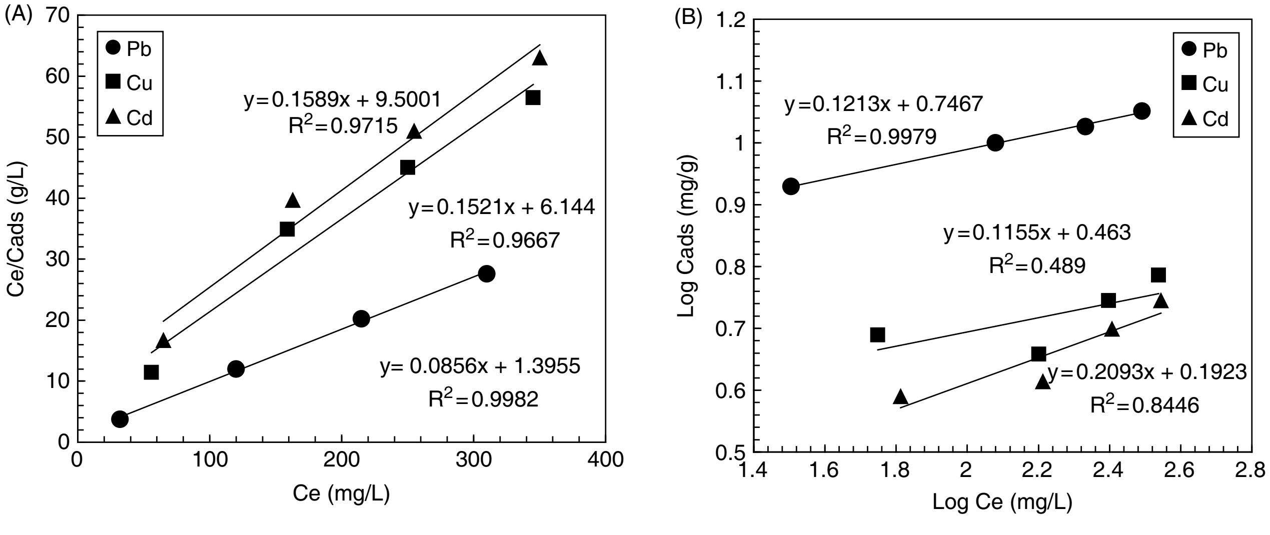

Fig. 8. Langmiur (A), Freundlich (B) adsorption isotherms of metal ions on recycled waste glass.

Table 4
Summary of DKR model parameters for (RWG)

Metals	X_m (mol/g)	β (mol^2/*j*^{2})	Sorption energy (*E*, kJ/mol)
Cadmium	7.859×10^{-5}	-0.2527×10^{-8}	14.067
Copper	1.14×10^{-4}	-0.147×10^{-8}	18.467
Lead	7.5×10^{-5}	-0.128×10^{-8}	19.724

sorption capacity, β is the activity coefficient related to mean sorption energy, and ε is the Polanyi potential, which is equal to:

$$\varepsilon = RT \ln(1 + \frac{1}{C_e}) \tag{10}$$

where, *R* is the gas constant (J/mol K) and *T* is the temperature (K). The saturation limit X_m may represent the total specific micropore volume of the sorbent. The sorption potential is independent of the temperature but varies according to the nature of sorbent and sorbate [42]. The sorption space in the vicinity of a solid surface is characterized by a series of equi-potential surfaces having the same sorption potential. The sorption energy can also be worked out using Eq. (11):

$$E = \frac{1}{\sqrt{-2\beta}} \tag{11}$$

Table 4 shows the *E* values are 14.067 for Cd(II), 18.467 for Cu(II) and 19.724 for Pb(II), kJ/mol on RWG. The positive values of *E* indicate that the sorption process is endothermic and that higher solution temperature will favor the sorption process [43]. The energy values for the studied heavy metals sorption on modified RWG have adsorption energies <40 kJ/mol which indicate that the sorption process is physisorption [44].

4. Conclusion

From this study, it can be concluded that, high-sorption performance and inexpensive of the adsorbent material (RWG). The sorption kinetics studies indicated that the experimental data followed the second-order kinetic reaction. A comparison of different isotherm models revealed that the sorption data perfectly fitted the Langmuir adsorption isotherms models with regression coefficient $R^2 > 0.95$ for all the studied metal ions. except for Pb^{2+} ions where the isotherms of adsorption obtained are in agreement with the models of Langmiur and Freundlich The selectivity sequence according to the maximum adsorption capacities can be given as $Pb^{2+} > Cu^{2+} > Cd^{2+}$. These appropriate features will contribute to the reuse of this novel waste glass as sorbent material in practical application for removal of heavy metals from industrial wastewater.

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