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Batch kinetics Studies on the Removal of Uranium and some Lanthanides from Aqueous Solutions onto Sonicated Emulsion Polymer

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Abstract: Several kinetic models were accustomed to check the experimental rate data and to examine the controlling mechanism of the sorption process for elimination of uranium and some lanthanides from aqueous solutions using synthesized sonicated emulsion polymer. The results showed thatpseudo-second-order and Elovich kinetic models provided a best-fit description and best correlate the experimental rate data for the sorption of U^{4+}, Gd^{3+}, Nd^{3+}, Eu^{3+}, Ce^{3+}, and La^{3+} ions because of their high correlation coefficients and low residual error relative to other used kinetic models. The intra-particle-diffusion (IPD) model reveals that the pore diffusion is not the sole rate-controlling. Using the pseudo-first order (PFO) and the pseudo-second order (PSO) rate constants combined with previously-established Langmuir sorption models, the kinetic sorption (k_a) and desorption (k_d) Langmuir rate constants were theoretically calculated. The pseudo-second order Langmuir model better fits the data during the early part of the experiments and it observed that sorption during later times occurred more slowly than the first part, which supports the hypothesis that IPD becomes the rate-limiting process during the course of the experiment.

Keywords: Sonicated emulsion polymer; Sorption kinetics, Uranium, Lanthanides.

1 Introduction

The nuclear industry can provide large quantities of radwaste comprising uranium and lanthanides. The radioactive and chemical toxicity of these radionuclides has the potential to threaten the global environment and human health in the long term if not properly addressed [1]. Thus, the development of new materials is of great importance for effective elimination of radionuclides from radwaste [2]. Sorption processes are mostly more interesting technology for the elimination of such hazardous ions owing to its low cost, and convenient operations [3-8]. The most serious point for sorption process is the design of proficient sorbents. Presently, numerous types of sorbents have been designed for extortion of radionuclide. Zeng et al. [4] synthesized phosphonate-functionalized polystyrene microspheres for proficient elimination of uranium(VI) from aqueous solution; Zhang et al. [9] synthesized amidoxime-functionalized macroporous fibrous polymeric sorbent by irradiation-induced graft polymerization for retention of uranium; and Zhang et al. [10] reported highly efficient removal of uranium(VI) from aqueous solutions using poly(acrylic acid)-functionalized microspheres. Lately Sun et al. reported a new polymeric nanofibers [11] for the proficient enrichment of radionuclides comprising uranium(VI), europium(III), and etc. Up till now, only a few studies were stated on the exact sorption mechanisms for numerous ions existent in radwaste. To optimize contaminant elimination capacities at nuclear treatment facility (NTF) by Sonicated emulsion polymer, it is important to understand the rate controlling processes of mass transfer at the sorbent-sorbate interface [12, 13]. This comprises but is not limited to, scrutinizing the required contact time amid the contaminants in solution and the sorbent to reach apparent or true equilibrium conditions [13]. For example, when the sorption rate is fast, sorption equilibrium is attained within a short time period, thus, relatively; a short

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hydraulic retention time is desired allowing NTFs to treat large influent radwaste volumes. This is very important as numerous NTFs need to manage with large and seasonally-fluctuating volumes of radwaste. Thus, kinetic sorption desires to be scrutinized and mathematical kinetic models of sorption need to be developed so that they can be used in combination with mass-transport models to sufficiently predict and manage the treatment of radwaste within a sorption medium.

The pseudo-first-order (PFO), the pseudo-second order (PSO), Elovich and the intra-particle diffusion (IPD) kinetic sorption models are some of the most commonly-used models to quantify the elimination of numerous contaminants from solution as a function of time [9]. The main benefit of the abovementioned four models is their ease and simplicity with which they can be fitted to the observed data. Additionally, the IPD model has the ability to provide insight concerning the processes underlying sorption. Nevertheless, distinguishing between sorption mechanisms based on the model fitting is difficult as the differences in the measures of fit are often minimal [13]. Furthermore, these models lack a true predictive capacity in that the fitted parameter values are often powerfully reliant on the initial experimental conditions [13]. All sorption rate coefficients, kinetic or equilibrium, can be reliant on other exterior factors such as temperature, solution pH and ionic strength. Besides, the PFO rate constant, and the PSO rate constant, can be powerfully reliant on the employed initial ion concentration. The reliance could be quantified and employed to make estimates of sorption times in a non-dynamic situation, but this reliance reduces the degree to which the PFO and PSO models can be employed to make estimates in a transport setting where the concentrations entering different sections of a sorbent would be constantly varying. To increase the predict ability of these sorption investigations, we have employed these derivations to determine the Langmuir kinetic sorption rate constants from the PFO and PSO model fits. All of the four used kinetic sorption models were used, and based on these models; a predictive kinetic Langmuir sorption model was derived.

In our previous work [6], it was reported how to successfully synthesize and characterize sonicated emulsion nanopolymer, and after that, adsorption isotherm, thermodynamic and fixed bed column and desorption experiments were successfully performed. The aim of the present work is to study the time-dependent models and also the solute sorption that determines the residence time required for completion of the sorption reaction. Concerning this target, many mathematical models have been planned to describe sorption data [8].

2 Experimental

2.1 Chemicals and Reagents.

All the reagents used in this work were of AR grade chemicals and were used without further purification. N, N'-methylene diacrylamide (DAM) (Riedel-De Haen AG) was used as a cross linker and Nitriliotriacetic acid (NTA) (Merck) ,that is a polyamino carboxylic acid, was used as a filler. All of the employed ions were BDH products.

2.2 Synthesis of Sonicated Emulsion Polymer

The synthesis of emulsion polymer of carboxy methyl cellulose as a spine with grafting of both acrylic acid and methyl acrylate has been investigated using ultrasonic irradiation and Tween 80, used as a surfactant to enhance interfacial tension between outer sphere water molecule and polymer and to prevent the aggregation of the components inside the polymer which, simplify the grafting process,. The resultant product was obtained after using water bath for about 3 hours at 70^{o}C to complete the reaction. The inorganic amorphous product was collected and washed with both acetone and distilled water for elimination of unreacted species and then the product was partially dried [7, 9, 10]. The full characterization of the material was presented elsewhere [7].

2.3 Sorption Study

2.3.1 Effect of Ph

The effect of pH on the sorption of U^{4+} and trivalent lanthanides from aqueous solution using synthesized sonicated emulsion polymer was investigated at the pH range from 1.0 to 6.0. The value of solution's pH was adjusted using dilute HCl and NaOH solutions. In this respect, 10 ml of 200 mg/l U^{4+} or studied lanthanides solutions was agitated with 0.05 g synthesized polymer; centrifuged to separate the solid and liquid phases.

2.3.2. Effect of Time

In order to investigate the effect of time on the elimination efficacy and to determine the reaction kinetic parameters, a series of batch kinetic studies were executed and shown in Fig. (1). The kinetics of the sorption is an important factor which controls the efficacy of the process [13]. The studies were conducted at a constant solution volume to solid mass ratio (V/m) of 200 ml/g, fixed pH, and initial metal ion concentration of 200 mg/l. The solution was stirred in a thermostat shaker and a fixed volume (1 ml) of the aliquot was withdrawn as a function of time. The withdrawn solution was centrifuged to separate the sorbent and a fixed volume of the clear solution was pipetted out for the determination of the amount of unsorbed metal ion still present in solution. The measurements were executed using spectrophotometer (Buck scientific model Sentra 101). The amount of metal ion eliminated on the synthesized material, q_t, (mg/g) was calculated using:

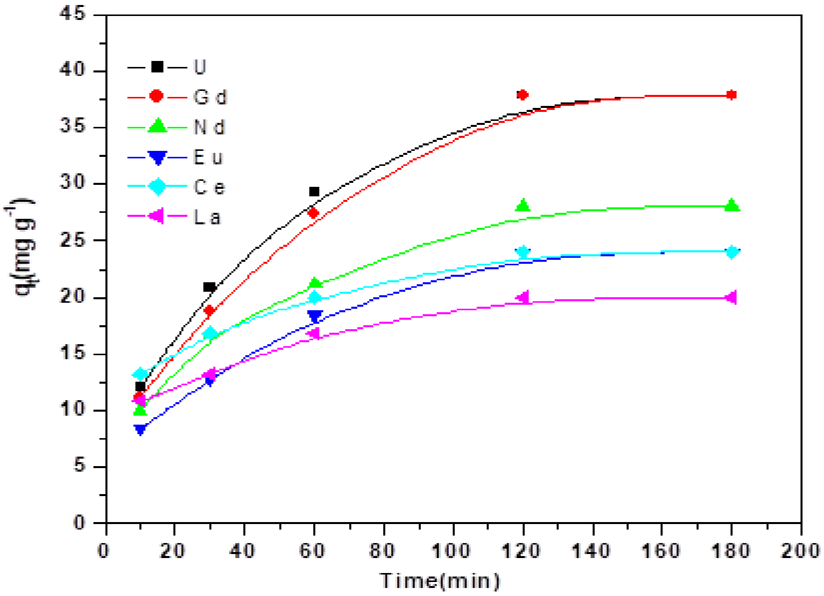


Fig.1: Effect of contact time on the amount sorbed of U^{4+}, Gd^{3+}, Nd^{3+}, Eu^{3+}, Ce^{3+} and La^{3+} ions from aqueous solutions onto sonicated emulsion polymer.

$$q_t = (C_o - C_t).\frac{V}{m} \tag{1}$$

where, C_0 and C_t are the initial and transient concentrations (mg l^{-1}) of metal ion in solution, V is the solution volume (l) and m is the sorbent weight (g).

2.3.3 Kinetic Modeling

It is well recognized that the characteristic of a sorbent surface is a critical factor that affects the sorption rate parameters and that the diffusion resistance plays a significant role in the overall transport of the solute. To describe the changes in the sorption of studied ions with time, numerous kinetic models were tested. The rate constant of each metal ions elimination from the solution onto sonicated emulsion polymer was determined via the use of pseudo-first-order (PFO) and pseudo-second-order rate (PSO) models. The Lagergren first-order rate expression [15] is written as:

$$q_t = q_e(1 - e^{-K_1.t}) \tag{2}$$

where, q_e and q_t (m g^{-1}) are the amounts of sorbed ions onto sonicated polymer at equilibrium and at time t and K_1 is the pseudo-first order rate constant (min^{-1}) [16]. The kinetic plots of q_t versus t for studied ions sorption are shown in Fig. (2 A).

Pseudo second-order (PSO) chemisorption kinetic rate equation is derived on the basis of the sorption capacity of the solid phase. It assumes that the rate of sorption is directly proportional to the number of active surface sites and that the rate limiting step may be a chemical sorption involving valence forces through sharing or exchange of electrons amid the sorbent and the sorbate. The pseudo-second-order model can be expressed as [17]:

$$q_t = \frac{(K_2 q_e^2 .t)}{(1 + K_2 q_e .t)} \tag{3}$$

where, K_2 is the rate constant of pseudo-second-order equation (mg^{-1} min^{-1}). The kinetic plots of q_t versus t for studied ions sorption are shown in Fig. (2 B).

The Elovich model has been extensively applied to chemisorption kinetics data and is often valid for systems in which the adsorbing surface is heterogeneous. This model can be expressed as follows [20]:

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

where, φ is the initial sorption rate (mg g^{-1} min^{-1}) that is related to the activation energy for chemisorption and degree of surface coverage. α is the Elovich model constant (g mg^{-1}). The kinetic plots of q_t versus t for studied ions sorption are shown in Fig. (2. C).

The intra-particle diffusion (IPD) kinetic sorption model (Eq. (5)) clarifies a multi-step kinetic sorption process that includes the external sorbate diffusion from the bulk solution to the sorbate-sorbent interface and thru the boundary layer surrounding the individual particles, sorption on the particles external surfaces, then sorption of sorbate by active sorption sites in the sorbent interior pore spaces and finally intra-particle sorbate diffusion onto the sorbent pores. However, the external diffusion can be neglected when there is a high agitation degree. Kinetic sorption via IPD is modeled by linearization of the equation developed by Weber and Morris gave as shown in equation (5).

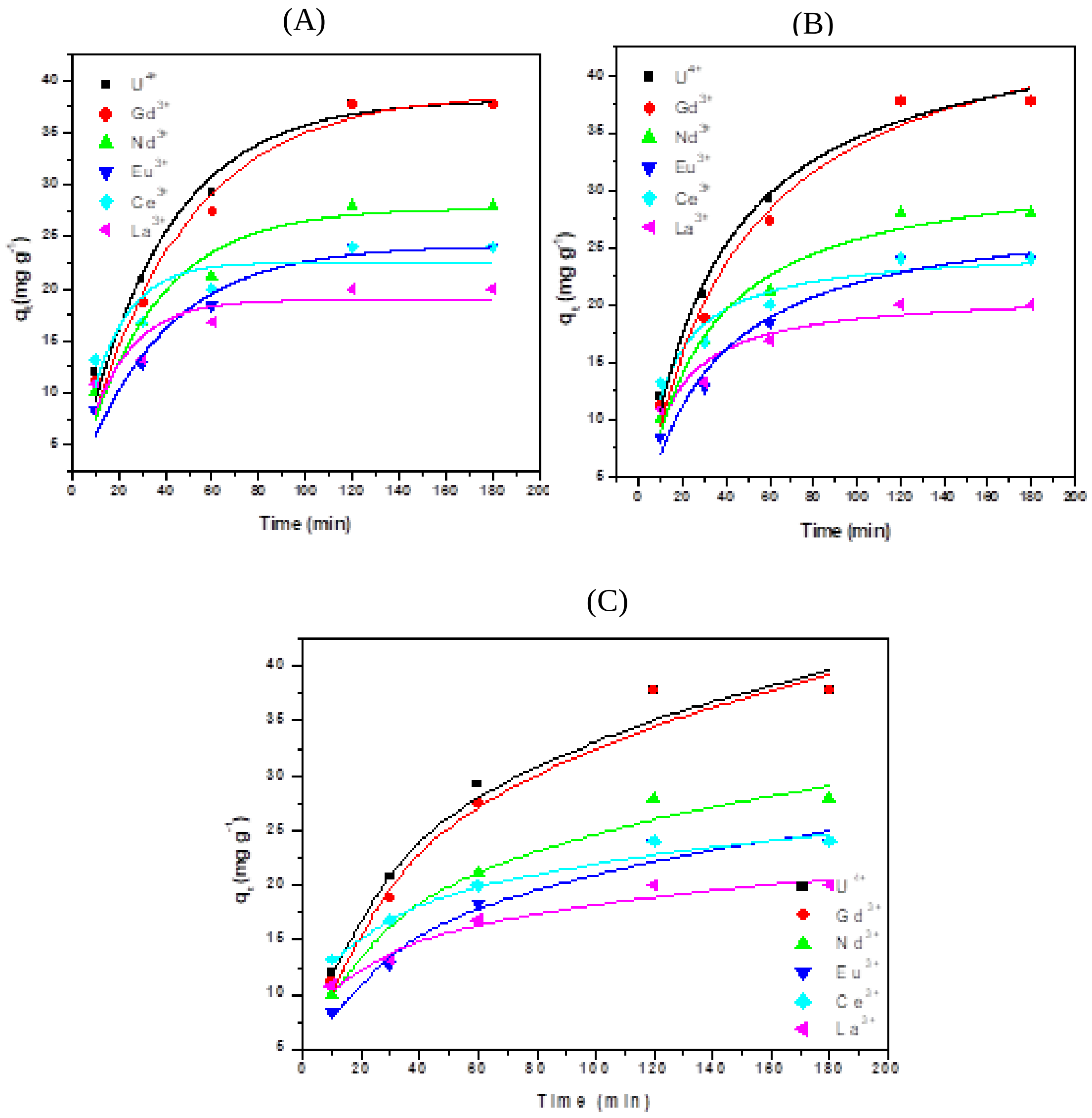


Fig.2: (A) PFO; (B) PSO and (C) Elovich kinetic plots for the sorption of U^{4+} ,Gd^{3+}, Nd^{3}, Eu^{3+}, Ce^{3+} and La^{3+} ions onto sonicated emulsion polymer.

$$q_t = \mathrm{K_p} t^{\frac{1}{2}} + \mathrm{C} \tag{5}$$

where K_p is the intra-particle diffusion rate constant (mg $g^{-1}min^{-1/2}$). C is intra-particle diffusion constant (intercept of the line) (mg g^{-1}) and is directly proportional to the boundary layer thickness. If the regression line passed thru the origin, then IPD is the rate-determing factor of the kinetic sorption process [13, 23-26]. Thus, the kinetic sorption models depicted above are commonly considered to be flexible mathematically to depict the numerous physical kinetic sorption processes comprising surface interactions and IPD. However, the interpretations and the theoretical applications of the models should be constrained by a given sorption experiment setup conditions or particular sorption system [13].

None of the aforementioned models depicted above has a true predictive capacity excluding under controlled conditions as the obtained parameter rate constants are reliant in the experimental conditions. It is needed to use a kinetic model whose parameters are not changing with time, e.g., the Langmuir kinetic based model. This model presumes that adsorption is a chemical process taking place on the solid sorbent surface, the sorbent has a finite sorption sites number and the adsorption rate is partially controlled by the filling degree of those sites and can be represented as:

$$\frac{dq}{dt} = k_a C(q_m - q) - k_d q \tag{6}$$

where, q_m is the maximum sorption capacity (mg g^{-1}) , C is the concentration (mg l^{-1}), k_a is the adsorption rate constant (L mg^{-1} min^{-1}) and k_d is the equilibrium desorption rate constant (min^{-1}). The solution of equation 6 relating PFO and PSO explained in details elsewhere [13].

3 Results and Discussion

It was observed that each metal ion sorption process is dependent on the initial pH of the solution and the amount of metal ion sorbed increased with increment in the pH value [12]. At lower values, the metal ion uptake was repressed in acidic medium that can attribute to the existence of H^+ particles that contend with the U^{4+} and trivalent lanthanides ions in the sorption sites. The optimum sorption capacity of the synthesized polymer was found to be 39.2, 39.2, 28, 24, 24, 20 mg/g at pH 5.0 for the studied metal ions U^{4+},Gd^{3+}, Nd^{3+}, Eu^{3+}, Ce^{3+} and La^{3+}, respectively. This can be attributed to the existence of a large number of carboxyl groups in the structure of the polymer, which, can dissociate to form carboxylate ion at higher pH. However, at pH values higher than 5.0, the metal hydrolysis will be occur.

The kinetic results (Fig. 1) at a constant initial ion concentration of 200 mg/g indicated that the sorption rate of metal-ion increases swiftly till reaches to steady state at about 3 h. This finding could be explained as the sorption sites firstly are highly accessible and the metal ions can interact effortlessly with the prevailing pores on the sorbent surface, thus high sorption rate is attained. Moreover, the driving force for the sorption is the concentration gradient amid the bulk solution and the solid–liquid interface that is high in the first period, which results in a high sorption rate. A slow sorption at the interior surface was then observed because of pore diffusion of metal solution into the matrix. The maximum sorption capacity of the synthesized polymer towards the concerned ions was found to be (20 mg/g) for La^{3+}, (24 mg/g) for Ce^{3+}, (24 mg/g) for Eu^{3+}, (28 mg/g) for Nd^{3+}, (37.8 mg/g) for U^{4+} and (37.8 mg/g) for Gd^{3+}, respectively.

The time-dependent data was modeled consistent with the PFO model. The PFO rate constant (K_1), equilibrium capacity (q_e) and correlation coefficients (R^2) are illustrated in Table (1). The analysis of the experimental data using pseudo-first-order kinetic depicts that the sorption rate relied in the sorption capacity and assumes that the reaction rate is limited by only one process or mechanism on a single class of sorbing sites and that all sites are of the time dependent type [16]. The visual examination of the results, Fig. (2 A), suggest the applicability of pseudo-first-order kinetic equation to fit the experimental data. By comparing the values of theoretically calculated equilibrium sorption capacities, q_e, with the apparent experimental sorption capacities, a considerable deviation between these values is noticed as shown from Table (1) and Fig. (2 A). This deviation indicates that it is not appropriate to use PFO equation to represent the sorption of the concerned ions onto the polymer for the entire sorption period. Moreover, it was observed that the obtained residual error (Fig. 3A) is high enough to be excluded from representing the experimental data.

The kinetic data of the studied ions were fitted to the PSO model. The acquired kinetic model parameters are summarized in Table (2). It was found that the PSO kinetic model had higher R^2 values at 0.908 to 0.985, as compared with that acquired from PFO model. Moreover, the computed sorption capacities from the PSO kinetic model were nearer to the experimental sorption capacities at equilibrium. Residual error analysis patterns (Fig. 3B)

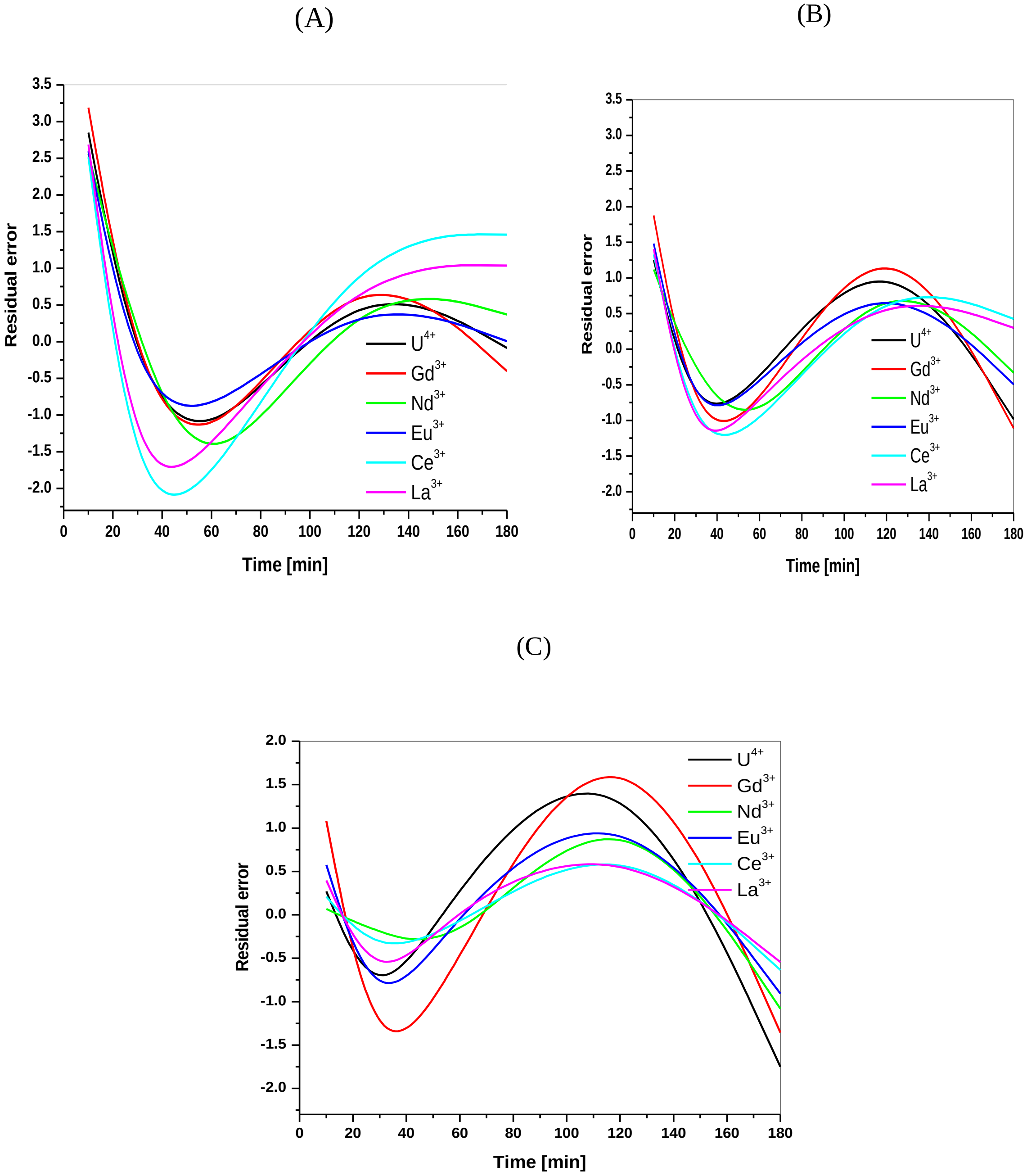


Fig.3: Residual error analysis for non-linear regression of (A) PFO model ; (B) PSO model, and (C) Elovich model.

were scrutinized and found that the residual errors values from PSO model is smaller than that of the PFO model. This indicates that a chemical reaction occurs amid sorbent surface and the sorbate via covalent forces or sharing electrons, till active sites onto sorbent surface are fully employed with ions.

Fig. (2 C) showed that the results follow an Elovich equation which assured that the rate-determining step is diffusion in nature and that this model should apply at conditions that desorption rate may be neglected. The acquired kinetic curve demonstrated good fitting with a high correlation coefficient values ($R^2 > 0.96$) and lower residual errors (Fig.3C). The acquired parameters are estimated using ORIGIN 6.0 software and the obtained values were tabulated in the Table (3).

Fig (4) demonstrates that the plot show the diffusion of ions through the solution to the external surface of sorbent. However, the intercept of the line fails to pass through the origin for all the studied ions which may be owing to the difference in the rate of mass transfer in the initial and final stages of sorption [13]. Additionally, such deviation of the straight lines from the origin reveals that the pore diffusion is not the sole rate-controlling step. The results indicate that the mechanism of ions sorption onto the sorbent is complex and both the film diffusion as well as intra-particle diffusion contributes to the actual sorption process. The IPD rate constants values are given in Table 4.

According to the PFO- and PSO- based Langmuir model, it is concluded that the data fit much better for the PSO-based Langmuir model than for the simulated PFO- based Langmuir model. For the PSO- based Langmuir model, the early-time data fit better than the later-time data (Fig. 5A, B).

The acquired results are consistent with the assumption that the sorption occur in more than one step, i.e., film diffusion dominating at early times and IPD becoming a rate-limiting process at later times. Additionally, PSO-based Langmuir model demonstrated that the sorption is chemisorption kinetics. Furthermore, this model is essential for designing experimental work, and the model variables are illustrated in Table (5).

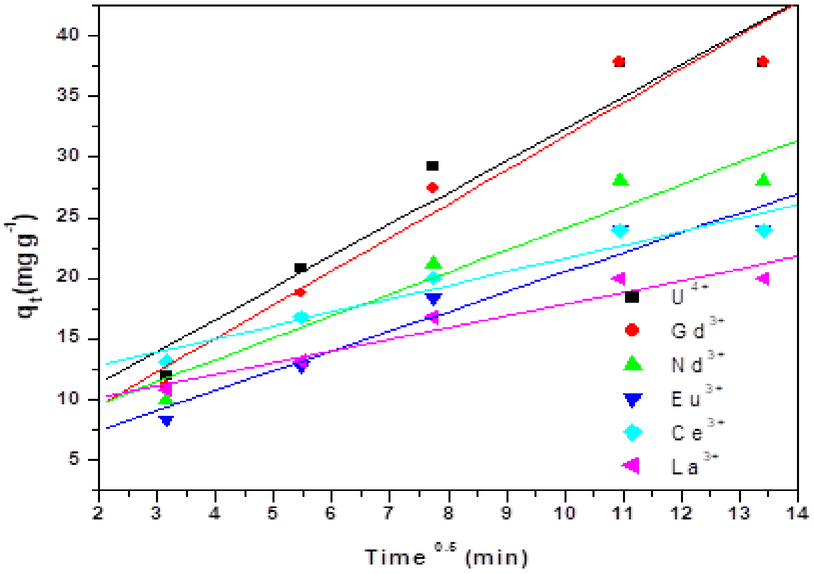
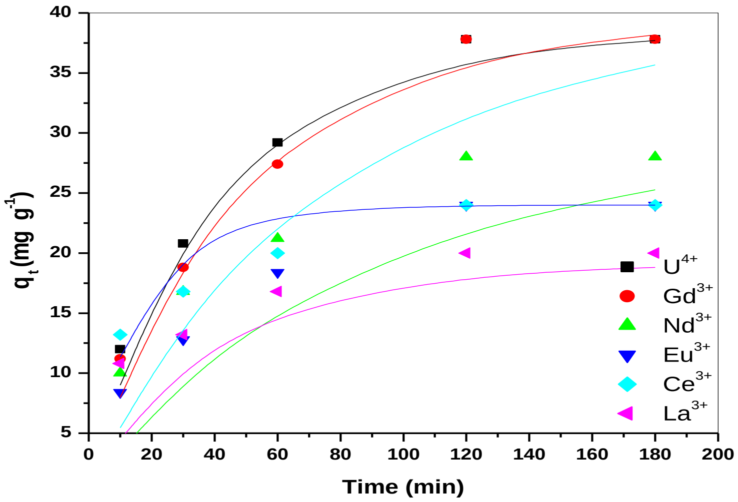


Fig.4: IPD kinetic plots for the sorption of U^{4+} ,Gd^{3+}, Nd^{3+}, Eu^{3+}, Ce^{3+} and La^{3+} ions onto sonicated emulsion polymer.



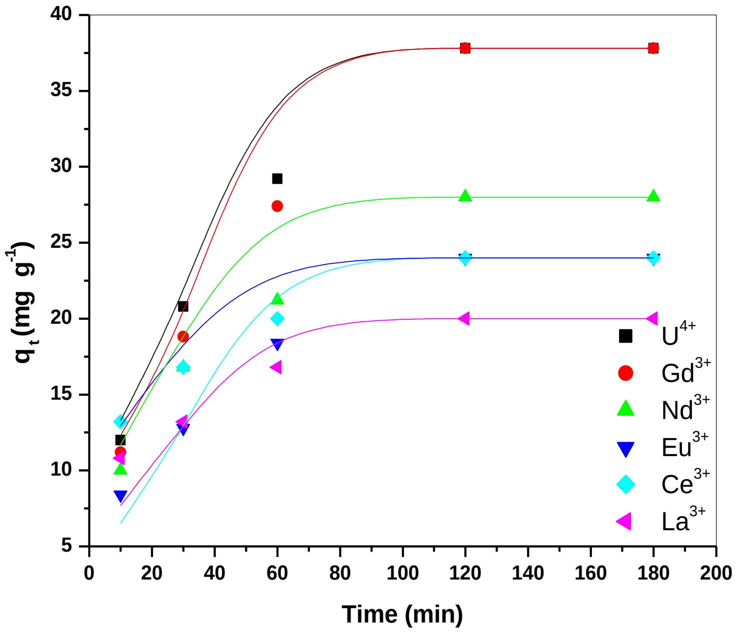


Fig.5: Langmuir based (A) PFO and (B) PSO kinetic plots for the sorption of U^{4+} ,Gd^{3+}, Nd^{3+}, Eu^{3+}, Ce^{3+} and La^{3+} ions onto sonicated emulsion polymer.

Table 1: Kinetic parameters of PFO for the metal sorption.

Ion	$q_{e,\ exp.}$ (mg g^{-1})	K_1(min^{-1})	$q_{e,cal.}$(mg g^{-1})	**R^2**
U	**37.9**	0.027	38.16	0.975
Gd	**37.9**	0.023	38.80	0.972
Nd	**28.0**	0.031	27.73	0.946
Eu	**24.0**	0.027	24.16	0.952
Ce	**24.0**	0.064	22.54	0.761
La	**20.0**	0.055	18.96	0.754

Table 2: Kinetic parameters of PSO for the metal sorption.

Ion	$q_{e,\ exp.}$ (mg g^{-1})	K_2 (g mg^{-1}min^{-1})	$q_{e,cal.}$(mg g^{-1})	**R^2**
U	**37.9**	0.031	45.81	0.985
Gd	**37.9**	0.024	47.84	0.977
Nd	**28.0**	0.037	32.52	0.977
Eu	**24.0**	0.032	28.80	0.971
Ce	**24.0**	0.090	25.03	0.925
La	**20.0**	0.081	21.05	0.908

Table 3: Kinetic parameters of the Elovich model for the sorption of studied ions.

Ion	$q_{e,\,exp.}$ **(mg g^{-1})**	α **(g mg^{-1})**	φ **(mg g^{-1} min^{-1})**	**R^2**
U	**37.9**	-0.1007	120.0537	0.980
Gd	**37.9**	-0.09653	890.96	0.972
Nd	**28.0**	-0.14626	44.16702	0.982
Eu	**24.0**	-0.1642	41.412	0.972
Ce	**24.0**	-0.24096	-12.533	0.979
La	**20.0**	-0.27624	-6.2264	0.966

Table 4: Kinetic parameters of the IPD model for the sorption of studied ions.

Ion	$q_{e,\,exp.}$ **(mg g^{-1})**	**Intra-particle diffusion equation**		
		K$_p$ (mg g^{-1}min $^{-1/2}$)	**C (mg g^{-1})**	**R^2**
U	**37.9**	2.62	6.15	0.964
Gd	**37.9**	2.76	4.02	0.972
Nd	**28.0**	1.80	6.06	0.967
Eu	**24.0**	1.62	4.28	0.969
Ce	**24.0**	1.10	10.62	0.967
La	**20.0**	0.964	8.29	0.967

Table 5: Kinetic parameters of the PFO- and PSO- based Langmuir model for the sorption of studied ions.

Ion	K_a (L mg^{-1} min^{-1})		K_d (min^{-1})		q_m
	PFO	PSO	PFO	PSO	
U	0.000135	0.00051	6.75E-07	0.0042	0.016
Gd	0.000115	0.00126	5.75E-07	0.0089	0.034
Nd	7.75E-05	0.00024	5.75E-07	0.0025	0.0155
Eu	6.75E-05	0.000126	3.38E-07	0.0015	0.0135
Ce	0.00032	0.00047	1.6E-06	0.0048	0.032
La	0.000135	0.00025	6.75E-07	0.0031	0.0275

Conclusion

The obtained results showed that the synthesized sonicated emulsion polymer is an efficient sorbent for the elimination of U^{4+}, Gd^{3+}, Nd^{3}, Eu^{3+}, Ce^{3+} and La^{3+} ions from aqueous solutions. Kinetic sorption experiments were systematically conducted for sorption of U^{4+}, Gd^{3+}, Nd^{3}, Eu^{3+}, Ce^{3+} and La^{3+} ions using a synthesized sonicated emulsion polymer as a sorbent. Different kinetic sorption models were compared to fit kinetic sorption experimental data. Generally, the PSO and the Elovich models outperformed the PFO model. The PFO, PSO model rate constant and the equilibrium model parameter values were used to calculate the Langmuir kinetic rate constants that could be used in a predictive capacity in a model of ion transport. The Langmuir constants did a good job predicting the early sorbed concentrations from the kinetic experiments but over-predicted the rates of sorption at later times, consistent with IPD and film diffusion becoming rate-limiting steps in the experiments.

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