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Adsorptive Removal of Acetic Acid by Walnut Shell as Low-Cost Adsorbent

Kavita Kulkarni*, Rahul Kulkarni, Yogesh Chendake, Arghya Garg and Pragati Srivastava

Department of Chemical Engineering, Bharati Vidyapeeth (Deemed to be University), College of Engineering, Pune-411043, India

*Corresponding author: Fax: +91 20 24372998; Tel: +91 20 24107390; E-mail: kskulkarni@bvucoep.edu.in; kavitashreya@gmail.com

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Walnut shell was used as an adsorbent for adsorption of acetic acid. Batch experiments were conducted for adsorption equilibrium studies and kinetics by using walnut shell. Optimization of parameters like temperature, rotation, adsorbent dose and contact time were studied. Amount of acetic acid adsorbed was analyzed by titration. Equilibrium condition was investigated. Adsorption kinetics were tested for different models and found best suitable for intraparticle diffusion model. The batch experimental data was fitted to Langmuir, Freundlich, Temkin, Sips and Redlich-Peterson isotherms. The best fitted isotherm was Langmuir isotherm. Regeneration was tried by heating the adsorbent in microwave at 200 ºC. Up to three cycles adsorbent gave good efficiency for adsorption of acetic acid.

Keywords: Acetic acid, Kinetics, Walnut shell, Isotherms, Adsorption.

INTRODUCTION

Hazardous effects and pollution caused by the various acids from effluent stream has resulted in imposing various stringent norms for their disposal. These norms are in terms of pH, acidic content and concentration. It has made the treatment of acids compulsory for its separation or treatment for conversion into non-hazardous product before disposal of acidic effluent. Such treatment would require chemical usage and generate the secondary waste with disposal requirements. It would be intensive due to requirement of chemical consumption [1]. Both these issues can be solved by economic recovery of acidic components from effluent streams.

Various processes have been reported for the separation of acetic acid *viz.* electrodialysis [2-4], solvent extraction [5,6], membrane separation processes like supported liquid membranes [7], pervaporation [8,9], reactive distillation [10-12] and adsorption [13-15]. These processes possess certain limitations, which affects their real life industrial applicability. Electrodialysis works on transport of ion through ionic membranes using electric potential as driving force. The acid transport depends upon electrical potential, membrane properties, solution composition, operational parameters, *etc.* It suffers from the co-transport of other components, which affects the transport properties and purity of recovered acids. This affects its applicability in further processes. Similarly other membrane processes of pervaporation and liquid membranes are affected by fouling, transport and selectivity property limitations along with stability issues. This limits the real life applicability of these processes for industrial effluent treatment.

Stripping of extracted acids is an important issue, which makes the recovery of acids difficult. It would generate the secondary waste along with loss of extractant. Secondary extractants used in reactive extraction has toxicity issues [16,17]. Though distillation based separation are possible there are issues with the present of secondary components into distillate [18,19]. It would affect distillate purity and applicability of recovered acids. Presence of other acids would affect purity and applicability.

Adsorption and ion-exchange can be used for selective separation of acids using adsorbent of ion exchange agents. Ion-exchange works upon selective interaction of materials in solution by combination of ions with similar charges [20,21]. The interacted acids ions are recovered by stripping using suitable stripping agent followed by activation of ion exchange resins. This recovery and reactivation of resins would generate secondary, which would require further treatment or disposal. On the other hand, adsorption works on the selective sorption of materials due to surface properties of adsorbents and the properties of materials to be separated. Sorbed materials are recovered by stripping using suitable eluent. This would reduce the consumption of chemicals while selective interaction would be able to provide desired purity for recycle or further application.

Various researchers are working on the different natural materials and their activated components like, rice husk, coconut shell, fruit peels, bamboo dust, wood, ground nut shell, *etc*. for separation of acids from various process and effluent streams [22-25]. Current work is aimed towards the formation of activated sorbent from walnut shell for the separation of acetic acid. Formed sorbent is analyzed for separation of acid using its synthetic solution and its mathematical analysis for isotherm fitting and parameter analysis. Further, the isotherm and equilibrium based investigations are used to define the mass transfer and diffusion models.

EXPERIMENTAL

All the chemicals used in this investigation were of analytical grade. Acetic acid (80%) was purchased from Loba Chemie Pvt. Ltd. and used as such. The sodium hydroxide flakes were purchased from Thomas Baker, U.K. The walnut shells were procured from local market (Pune, India).

Adsorbent preparation: Walnut shells being waste are available easily in large quantity easily. They were procured from the local market (Pune, India). Shells were cleaned for removal of surface impurities and crushed for size reduction. They were meshed through 850 μm sieve to remove unwanted coarse particles. Obtained walnut powder was cleaned with ample of distilled water to remove dust and external surface impurity present. It is dried in the microwave at 100 ºC for 48 h. Powder was characterized for microporous structure by scanning electron microscopy COMP JEOL model JSM, 6360A at 100 X magnification.

Adsorption studies: Batch experiments were conducted for the process of adsorption of acetic acid by using walnut shells. Five Erlenmeyer flasks (250 mL) containing 100 mL of acetic acid and with known quantity of adsorbent were placed in thermostatic shaker. Experiments were conducted for various adsorbent dosages for optimizing the weight of adsorbent. After regular interval the samples were drawn and analyzed for acetic acid concentration using titrimetric analysis. The experiment is continued to analyze acetic acid sorbed per unit quantity of adsorbent based on the difference between initial and final concentration till equilibrium stage. Initial investigations were carried out to determine effect of temperature, time, stirrer speed (rpm) and concentration on equilibrium adsorption conditions. They were repeated at equilibrium condition to obtain the amount of acid adsorbed at these conditions to avoid errors due to material loss during analysis and used in further analysis. The investigations were carried out at different acetic acid solution concentrations of 2, 4, 6, 8 and 10% prepared in distilled water. Reproducibility of the results of batch experimentation was checked thrice and for all calculation average values of the results were used. For optimization of all the other parameters was done at 10% acetic acid. Obtained equilibrium data was analyzed by fitting with Langmuir, Friendlich, Temkin, Sips and Redlich-Peterson isotherms. Further, it was analyzed for the thermodynamic models of sorption to determine the mass transfer and sorption efficiency properties.

RESULTS AND DISCUSSION

Selection of walnut shells: Formation of shells for sorption experiments goes through the processes of grinding dried shells to powder form to pass through 850 μm mesh. This smaller size particles and crushing would be important to enhance material surface area for the sorption analysis. Further, it would enhance exposure of active sites for interaction and sorption. They would also beneficial during mixing with solution, improve the mass transfer and sorption properties. Formed shells were porous in nature with rock like structure as observed from SEM analysis presented in Fig. 1. Such porous structure would provide higher surface area for sorption.

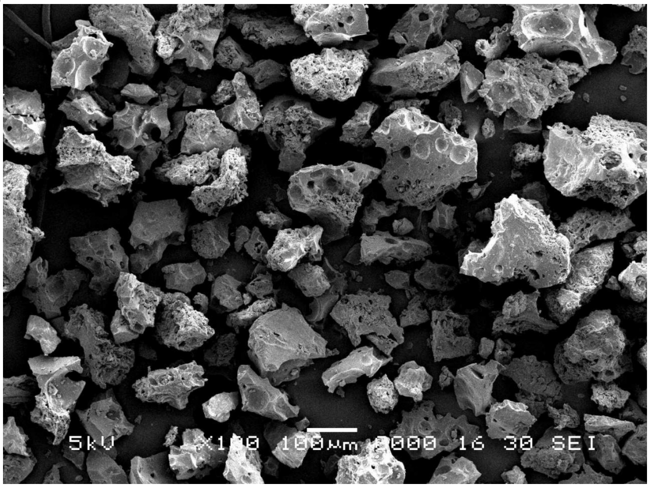

Fig. 1. Scanning electron microscopy for walnut shell powder

Further these shells are known for their excellent chemical resistance, which are known to stable in acidic and alkaline conditions [26]. These properties of higher surface area, presence of interactive groups, excellent chemical resistance, organic origin and biodegradability makes walnut shell a potential material for the sorption based separation of acetic acid.

Effect of concentration: Adsorption in is dependent upon surface properties of adsorbent, its interactions with adsorbate, thermodynamics and process kinetics. The investigations initiated with exploring effect of feed acid (2-10%) content on equilibrium sorption with 5 g of adsorbent. This would provide the acid uptake capacity and saturation limit for the adsorbent. Initially, the experiment was carried by drawing samples at regular time interval to evaluate the time required to reach the equilibrium condition. This was followed by repeating the experiment at equilibrium time conditions. It would avoid the issues of variation in solution concentration due to the loss during samples drawn for analysis.

The result of the adsorption process was studied and the acetic acid adsorbed is given in number of moles adsorbed per gram of adsorbent at equilibrium as per eqns. 1 and 2:

$$q_t = \frac{(C_o - C_t)V}{X} : \text{At anytime} \quad (1)$$

$$q_e = \frac{(C_o - C_e)V}{X} \text{ : At equilibrium} \tag{2}$$

where, C_o and C_e: concentration of acetic acid at any time and at equilibrium; q_t and q_e: number of moles of acetic acid adsorbed at any time and at equilibrium; V: volume of the acetic acid; X: weight of adsorbent.

As observed from Table-1, amount of acid sorbed per unit of sorbate in reaction media increases with increase in solution concentration. The acid removal increase was low and linear initially till the concentration upto 8% of acetic acid in water. Further the sorption of acid per unit amount of walnut shell showed an exponential increase with increase in feed acid concentration as shown in Fig. 2. This could be attributed to availability of higher number of moles of acetic acid in solution. The presence of interactive sites and limited availability of acetic acid molecules at lower feed concentration would lead to strong interactions. This would lead to initial quick interactive sorption on surface. Further, the variation in nature of sorption conditions are observed with increase in solution concentration from 8 to 10%. This can be attributed to higher interactions and initiation of multilayered sorption when the concentration goes beyond this limit. It is expected that at 8% the surface sorption is near saturation level. While the concentration beyond 8% would trigger higher or multilayer interactions and higher sorption on the sorbent. Similar higher sorption of acid at higher solution concentration has been reported earlier [27].

TABLE-1
EFFECT OF INITIAL CONCENTRATION

Initial concentration (%)	Acetic acid removal (%)	Acid adsorbed on walnut shell (mol/g)
2	17.14	0.54
4	17.33	1.30
6	18.28	1.80
8	19.23	2.40
10	30.69	4.60

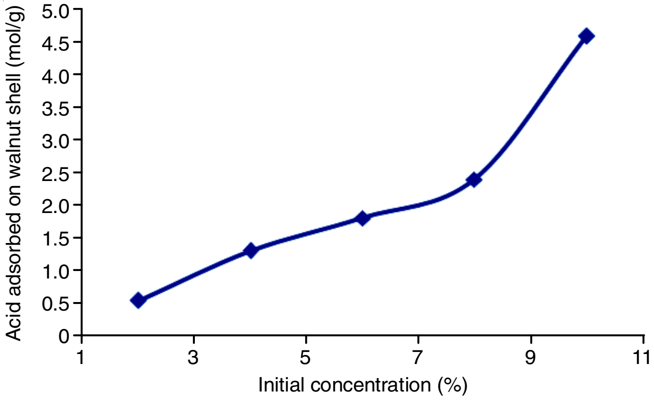

Fig. 2. Acetic acid adsorbed for various initial concentration

Here the higher acid removal capacity at lower concentration is highly important for the acid sorption and recovery point of view. In case of industrial effluent or other process streams containing lower concentration of acids would trigger higher and preferable removal by sorption at lower concentration resulting in higher acid recovery. Again the secondary sorption is predominant at higher acid concentrations. This would assist further for acid recovery at these concentrations. Thus current walnut shell based sorbent has benefits as sorbent at both the conditions of lower and higher concentrations.

Time dependency: Time is the major factor in the adsorption process. It is dependent upon mixing between sorbate and sorbent, its interaction kinetics. Additionally it is also affected by the surface properties and pore size, porosity and pore structure of adsorbent. Hence time required for saturation and optimum sorption should be analyzed to define the flow pattern, column contact pattern and residential time. The batch investigations were carried out at contact time varying in the range of 20 to 100 min to determine optimum contact time.

The amount of acid sorbed was increased by 68.75% parabolically from 20 to 60 min, while it reaches to the saturation level beyond this time. Initially the sorption of 1.12 mol/g was observed within 20 min (Table-2). This could be attributed to availability free interaction sites. It would result into quicker and higher interactive sorption. Further with passage of time the sorption sites would get saturated which would limit the interactions and reduce the rate of interaction. It resulted into parabolic increase in sorption properties with time, which achieve the saturation or plateau like situation after 60 min. A smaller decrease observed at 100 min could be due to manual error during concentration analysis. Similar parabolic increase in sorption with chemical interactions has been reported earlier [28].

TABLE-2
EFFECT OF TIME ON ACETIC ACID ADSORPTION

Time (min)	Acetic acid removal (%)	Acid adsorbed on walnut shell (mol/g)
20	7.76	1.12
40	9.70	1.40
60	13.10	1.89
80	13.10	1.89
100	12.62	1.82

Optimization of temperature: It was observed from Table-3 that the equilibrium acid uptake per gm of adsorbent was decreased with increase in temperature. The maximum adsor-ption was observed at 30 ºC for the investigations carried out in the rage of 30 to 50 ºC with 30 ºC being room temperature. This indicated that the adsorption process was exothermic, which is a sign of chemical sorption.

TABLE-3
TEMPERATURE EFFECT ON ADSORPTION OF ACETIC ACID

Temperature (°C)	Acetic acid removal (%)	Acid adsorbed on walnut shell (mol/g)
30	13.10	1.89
35	11.21	1.61
40	9.75	1.40
45	8.29	1.19
50	8.09	1.19

Optimization of agitation speed: The adsorption process is also affected by contact of adsorbent and adsorbate. Hence the contact pattern and mixing system needs to be analyzed for optimum sorption based recovery of acids. In present work, effect of agitation speed investigated in the rpm range of 90 to 130. It was observed that agitation speed has direct impact on adsorption efficiency (Table-4). Adsorption based acid removal and amount of acid sorbed per unit amount of adsorbate increased with the increase in agitation speed. This could be attributed to the increased contact efficiency and between adsorbent and adsorbate. It would reduce the mass transfer resistance, which would result in enhanced interaction and sorption of acids. A sudden increase in sorption rate and removal efficiency was seen at 130 rpm. It could be due to overcoming the mass transfer boundary layer and enhancement in contacts with the interaction sites in pores of porous adsorbent. Further no increase in the adsorption capacity was observed after 130 rpm. An increase in the adsorption of acetic acid with increase in agitation speed on porous adsorbent has also been reported [29].

TABLE-4
OPTIMIZATION OF AGITATION SPEED

Agitation speed (rpm)	Acetic acid removal (%)	Acid adsorbed on walnut shell (mol/g)
90	7.84	1.05
100	12.25	1.75
110	12.74	1.82
120	14.70	2.10
130	29.30	4.41

Effect of adsorbent dose: An increase in amount of acid adsorbed and its removal efficiency was observed with increase in adsorbent dosing as shown in Table-5. However, after 5 g of adsorbent dosing a saturation point was obtained. The constant acid removal and saturation point beyond the loading of 5 g could be attributed to maximized separation of acids and reduction in resultant acid concentration in solution. It would limit the availability of acid molecules for sorption based separation. Further higher loading would lead to overlapping and aggregation of adsorbent and limiting the removal efficiency.

TABLE-5
EFFECT OF ADSORBENT DOSE

Adsorbent dose (g)	Acetic acid removal (%)	Acid adsorbed on walnut shell (mol/g)
2	9.13	1.26
3	10.15	1.4
4	11.67	1.61
5	16.6	2.29
6	16.6	2.29

Adsorption studies

Langmuir isotherm: Langmuir isotherm gives details of surface covered by adsorption in the process, defines the interaction intensity and adsorption capacity [30]. The Langmuir isotherm is represented by the following equation:

$$\frac{C_e}{q_e} = \frac{1}{q_m k_e} + \frac{C_e}{q_m} \tag{3}$$

where, q_m is maximum adsorption capacity, k_e (1/g) is Langmuir constant and C_e represents adsorbate concentration at equilibrium.

The Langmuir constant (k_e, 1/g) correlates to the concentration and availability of adsorbed sites. The data analysis and fitting of curve provided values of kinetic constants of Langmuir isotherm. Further the feasibility of Langmuir isotherm is given by separation factor R_L [31], given by eqn. 4:

$$R_L = \frac{1}{1 + k_d C_o} \tag{4}$$

where, C_o is the initial concentration and k_d is the rate constant. Separation factor R_L for adsorption of acetic acid on walnut shell was 0.6784, which signifies the favourability of the Langmuir isotherm. The kinetic data for Langmuir isotherm is represented in Table-6.

TABLE-6
KINETIC PARAMETERS DATA OF DIFFERENT ISOTHERMS

Isotherm	Parameters	Values	Correlation coefficient
Langmuir	Q_0 (mol/g)	5.9312	0.9849
	K_L (mL/mol)	0.315	
	R_L	0.6784	
Freundlich	n (g/mL)	0.7432	0.9053
	k_f ((mL/mol)$^{1/n}$)	2.9603	
Temkin	B_1 (mol/g)	2.2459	0.653
	A (mL/mol)	3.9515	
Sips	β_s	1.3455	0.9053
	a_s/K_s	0.3378	
Redlich Peterson	β	-0.3473	0.3913
	A	2.9659	

Freundlich isotherm: Freundlich isotherm is another way of analysis of adsorption. It provides adsorption analysis towards sorption of sorbate on heterogeneous surfaces as sorbent [32]. Freundlich constants K_F and 1/n presents the capacity of adsorption and intensity of adsorption.

$$\log q_e = \log K_F + \frac{1}{n} C_e \tag{5}$$

Data analyzed for the fitting of Freundlich isotherm and the resulting correlation constants are given in Table-6, which shows Freundlich isotherm favourably defines the adsorption of acetic acid on walnut shell.

Temkin isotherm: This isotherm is valid for intermediate concentration values. It shows reduction in heat of transfer with increase in surface coverage. The linear form Temkin isotherm is represented by eqn. 6:

$$q_e = \frac{R_t}{b} \ln K_T + \frac{RT}{b} \ln C_e \tag{6}$$

It can be observed from plot and related constants that Temkin isotherm is not suitable for the process. Though the sorption is by interaction between molecules, it is not well defined by Temkin isotherm.

Sips isotherm: Sips isotherm is another way to define the sorption process by combining Langmuir and Freundlich isotherms [33]. This isotherm follows Freundlich isotherm at low adsorbate concentration and Langmuir isotherm at high concentration of adsorbate. The isotherm, sorption progress and Sips parameters are affected by concentration, temperature and pH. The linearized form of Sips isotherm is given by eqn. 7 [34].

$$\beta_s \ln C_e = -\ln\left(\frac{K_S}{q_e}\right) + \ln(\alpha_s) \tag{7}$$

Redlich-Peterson isotherm: Similar to Sips isotherm, Redlich-Peterson isotherm is also a combination of Freundlich and Langmuir isotherms [35]. It has advantages that it approaches the Henry region at any dilution [36]. This isotherm is applicable for multilayer adsorption, while monolayer adsorption is not followed by it. The linearized form of this isotherm is given by eqn. 8 [37]:

$$\ln\frac{C_e}{q_e} = \beta \ln C_e - \ln A \tag{8}$$

The nature of plot and correlation coefficient obtained showed that Redlich-Peterson isotherm does not follow the adsorption process from this work.

Further the kinetic data (Table-6) for isotherms and values of correlation constants shows that Freundlich and Langmuir isotherms are best fit and suitable for current adsorption process. At lower concentration, it follows Langmuir region of increase in adsorption with increase in adsorbent concentration till the availability or abundance of surface area on adsorbate. Further, at higher adsorbent (beyond certain critical concentration) shows sudden increase in adsorbent uptake resulting in multilayer adsorption as suggested by Langmuir adsorption.

Kinetic models

Intraparticle diffusion model: Intraparticle diffusion model in the linearized form as presented by eqn. 9:

$$q_t = k_p t^{0.5} \tag{9}$$

where k_p is rate constant for diffusion (mol/g min$^{0.5}$), q_t is acetic acid adsorbed and t is time of contact.

Intraparticle diffusion is rate controlling when the plot of q_t and $t^{½}$ (Webber Morris) is straight line [38]. The plot (Fig. 3) is nearly linear after some period initially at lower concentration. This can be attributed to the initial diffusion resistance of surrounding at low concentration followed by intraparticle diffusion. The sharper lines for higher acetic acid concentration can be attributed to intraparticle diffusion as major controlling resistance for adsorption. The correlating parameters are expressed in Table-7.

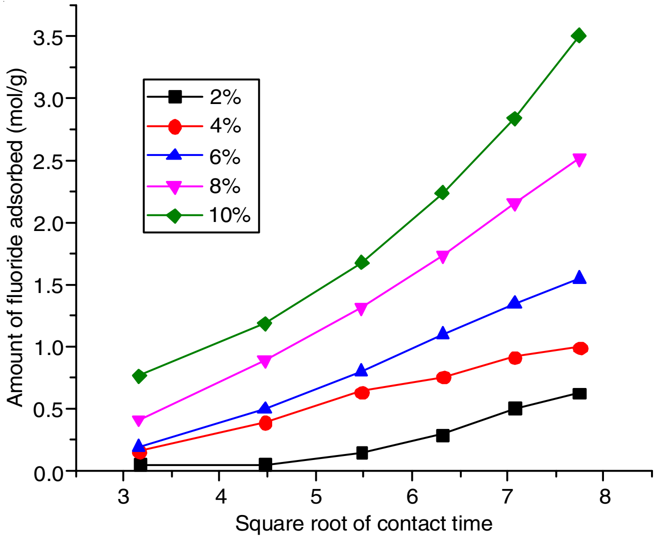

Fig. 3. Intraparticle diffusion model for the sorption of acetic acid on walnut shell

Pseudo-first order and pseudo-second order model: Linearized form of the pseudo first order model is given by eqn. 10 in which q and q_e is amount of acetic acid adsorbed at time 't' and at equilibrium, respectively. For pseudo second order the linear form is given by eqn. 11:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{10}$$

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{11}$$

where k_1 and k_2 are rate constants for pseudo-first order and pseudo-second order, respectively. The plot of ln (q_e-q_t) *vs.* t (Fig. 4a) was not-linear which showed deviation in pseudo first order model. Similarly, the plot of t/q_t *vs.* t (Fig. 4b) showed deviation from linearity in turn pseudo-second order model.

The kinetic model parameters compiled and analyzed from Table-7 indicated that intraparticle diffusion showed best fit for the process, although with small deviation at lower concentration. All the coefficient of correlation is in the range of 0.864-0.994. The rate constant values for the intra particle diffusion model were increased with increase in concentration, showing stronger dependency on adsorbate concentration for the same. This is characteristics of intraparticle diffusion model, where the diffusion resistance in boundary layer decreased with increase in concentration [39,40].

TABLE-7
KINETIC PARAMETERS FOR INTRAPARTICLE DIFFUSION MODEL, PSEUDO FIRST ORDER MODEL AND PSEUDO SECOND ORDER MODEL

Initial concentration of acetic acid (%)	Intraparticle diffusion model		Pseudo-first-order kinetic model		Pseudo-second-order kinetic model	
	Rate constant (mol/g min$^{0.5}$)	R^2	Rate constant (min^{-1})	R^2	Rate constant (g/mol min)	R^2
2	0.1315	0.8642	0.0366	0.7801	3.6670	0.4593
4	0.1874	0.9945	0.0547	0.9127	0.0006	0.0032
6	0.3017	0.9943	0.0466	0.9459	0.0774	0.4860
8	0.4607	0.9923	0.0425	0.9454	0.0003	0.0683
10	0.5920	0.9599	0.0344	0.9305	0.0305	0.4386

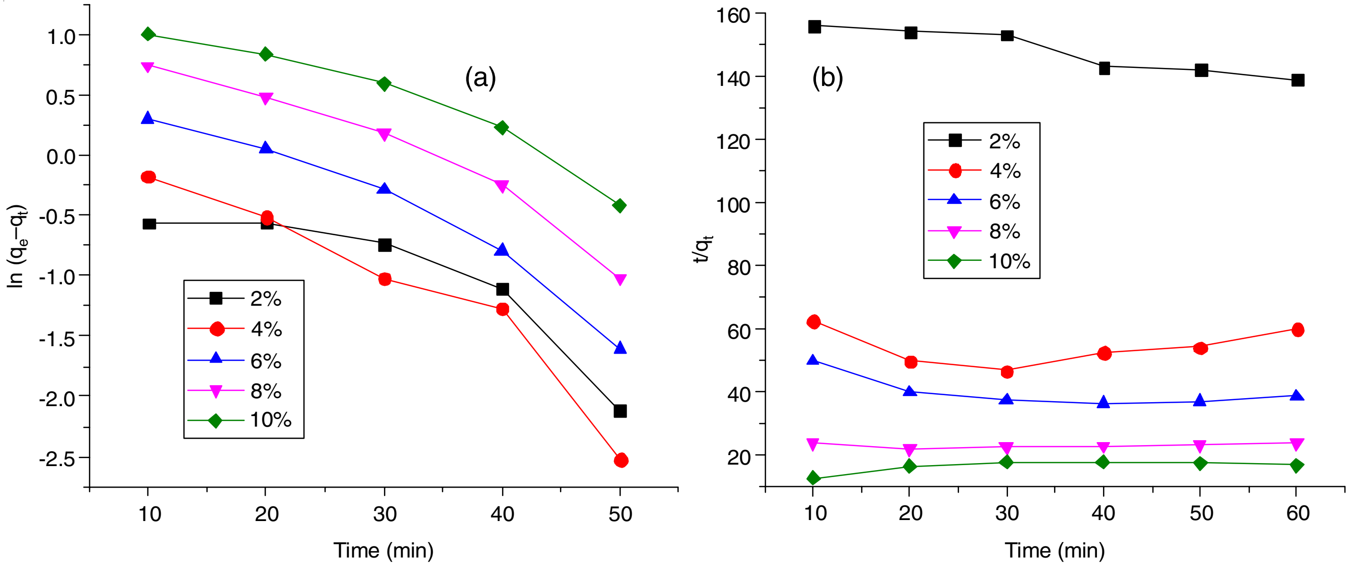

Fig. 4. (a) Pseudo first order model, (b) Pseudo second order model

Thermodynamics studies: At 30 ºC, adsorption of acetic acid was maximum. At this temperature change in Gibb's free energy for the adsorption process was calculated by the use of Langmuir constant (eqns. 12 and 13).

$$\Delta G^o = -RT \ln K \quad (12)$$

$$\Delta G^o - RT \ln (55.5 K) \quad (13)$$

where k is the equilibrium constant and the value of activity of water is 55.5 K found for solution. At 30 ºC, the value of ΔGº was calculated and found to be -7.2077 kJ/mol. The value of ΔGº signified the type of adsorption was spontaneous

Regeneration of adsorbent: In current process, the regeneration was simple by washing the adsorbent with distilled water followed by drying in microwave at 100 ºC for 12 h. Dried adsorbent was used for the second cycle of the batch operation. Similar procedure was repeated for second and third cycle. After third cycle, the adsorbent gave very less efficiency for the adsorption of acetic acid. This needs to be improved to enhance reusability to avoid repeated loading and cleaning issues along with its waste generation.

Conclusion

In current study, walnut shell was used as low cost adsorbent for the removal of acetic acid. The optimum time for maximum removal was 60 min for 10% acetic acid concentration. Langmuir isotherm was best fitted to the kinetic data. The Freundlich and Sips isotherm was also fitted with correlation factor of 0.9. Maximum removal of acetic acid was 32% at optimized parameters. For the adsorption process, intraparticle diffusion was applicable. Adsorption of acetic acid was impulsive by using walnut shell. Some further modifications in the adsorbent will improve the recovery of acetic acid.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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