Electrochemical regeneration of granular activated carbon saturated with organic compounds

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ABSTRACT

Electrochemical regeneration of granular activated carbon (GAC) saturated with organic compounds was investigated using SnO_2/Ti anodes. Experiments were carried out under a semi-batch electrochemical reactor covering wide range in operating conditions. A kinetic model has been developed to describe the electrochemical regeneration and the performance indicators such as instantaneous current efficiency and regeneration efficiency were estimated. The proposed model has been effectively demonstrated for the regeneration of GAC saturated with organics present in the 4,4′-diamino stilbene-2,2′-disulfonic acid manufacturing wastewater. It has been observed from the present experiments that the applied current density has significant influence on adsorption capacity of regenerated GAC. A good agreement of the model predictions with the experimental observations shows that the proposed kinetic model can be used as a better design method for electrochemical regeneration of spent GAC.

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1. Introduction

The exponential industrial growth and rapid changes in day-to-day activities of human beings resulted in an increase in the volume and complexity of waste (air, water and soil) to the environment. The treatment and reuse of treated wastewater is also increasing among the industries due to increased awareness and imposed stringent environmental regulations. Though various methods have been proposed to treat the water/wastewater contaminated with organic compounds, the use of granular activated carbon (GAC) is a most common and widely preferred method due to its high surface area, low specificity, high removal efficiency and fast adsorption kinetics. The unique adsorption characteristics of GAC depend on the specific surface area, pore structure and surface functional groups. In recent years, the regeneration of spent GAC has gained greater attention among the industries/researches as the spent GAC is subjected for landfill and the discarded GAC leaches into the ground causing serious environmental issues. Thus, regeneration and reuse of spent GAC becomes decisive to ensure adsorption is most economical and environmentally acceptable method for elimination of organic compounds [1].

Conventionally, the spent GAC is regenerated by chemical and thermal methods. These conventional methods have serious drawback such as high energy consumption, material loss, loss of adsorption capacity, etc. The process of thermal regeneration is widely used with high efficiency, involves a pyrolytic and an oxidative stage, where the spent GAC is exposed to temperatures up to 800 °C. However this technique has a drawback of carbon loss due to oxidation, attrition and high energy consumption [2]. While the chemical methods such as solvent extraction and wet oxidation associated are high cost and loss of adsorption capacity due to residual solvent and catalyst. Furthermore, the regeneration efficiency in chemical methods depend on the type of organic pollutants [3,4].

Electrochemical technology continues to make many contributions to organic synthesis, energy production, material recycling, environmental treatment/monitoring, etc. The uniqueness of electrochemical method includes environmental compatibility, versatility, energy efficiency, safety, selectivity, amenability to automation, and cost effectiveness [5]. Therefore, the electrochemical regeneration of spent GAC has gained greater attention in recent years due to its unique features such as low temperature operation, no addition of chemicals and in situ cracking of organics deposited on the carbon without damaging the structural properties and characteristics of carbon [6]. Good amount of literature has been reported on electrochemical regeneration of spent GAC. Ban et al. reported the potential of electro-adsorption/desorption on GAC for wastewater treatment of industrial effluent [7]. Narbaitz and Cen studied electrochemical regeneration of spent GAC used for phenol adsorption and obtained more than 95% regeneration efficiency with no apparent carbon loss [8]. Zhang studied the electrochemical regeneration of spent GAC enriched with phenol, and reported that over 80% regeneration efficiency was achieved [9].
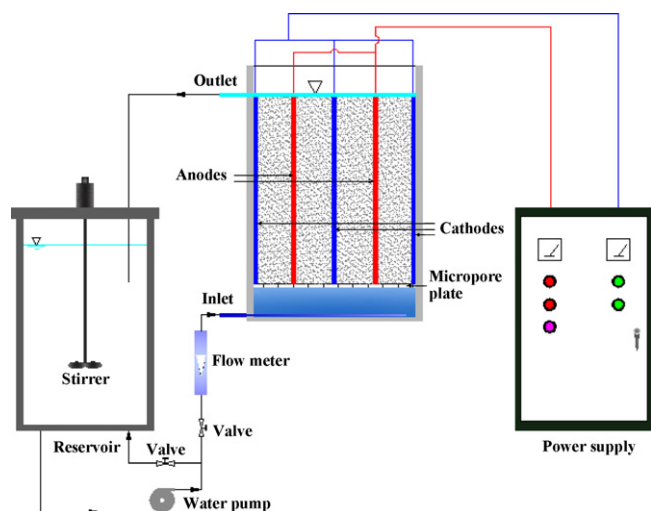
Fig. 1. Schematic representation of experimental set-up.

The author further declared that desorption and destruction of phenol adsorbed on GAC could be greatly enhanced by electrochemical regeneration. Though some studies reported on electrochemical regeneration of spent GAC, the work related to the mechanism of electrochemical regeneration, adsorption capacity of regenerated GAC and optimization is scarce. Thus, it is attempted to study the electrochemical regeneration of GAC in the present study. A kinetic model to describe electrochemical regeneration has been proposed.

2. Experimental

The experimental set-up consists of a reservoir with an effective volume of 30 L, a rectangular electrode chamber made of polymethyl methacrylate with 0.2 m × 0.2 m × 0.3 m in size and a power supply. Fig. 1 shows the schematic representation of present experimental set-up. A micropore plate is fitted at the bottom of the electrode chamber for even distribution of electrolyte inside the electrode chamber. SnO_2/Ti anode and stainless steel (SS) cathode with an active area of $0.06\,m^2$ ($0.2\,m \times 0.3\,m$) were used as anode and cathode, respectively. The electrodes were fixed in the electrode chamber with an inter-electrode distance of 50 mm and the electrode gaps were filled with spent GAC. Constant flow of electrolyte was provided with a metering pump connected to the reservoir. The power supply for the electro-regeneration was drawn from an electric motor (model: KZD1000/12) having capacity of 0–1000 A/0–12 V. Commercially available GAC of 2.5 mm size with a specific surface of $632\,m^2\,g^{-1}$ and a specific weight of $0.50\,kg\,L^{-1}$ was purchased from Huaxin Company, China, used for the experimentation. The carbon was washed with deionised water several times and dried in an oven at 105 °C for 2 days to a constant weight prior to the experimentation.

Two distinct experiments were carried out in the present investigation: adsorption studies using fresh and recycled GAC, electrochemical regeneration of spent GAC.

2.1. Equilibrium sorption studies

Langmuir isotherms were used to evaluate the adsorption capacity of GAC. Isotherms experiments were conducted with both fresh GAC and recycled (electrochemically regenerated) GAC at room temperature. A known amount of GAC was soaked with 150 mL of 4,4′-diamino stilbene-2,2′-disulfonic (DSD) acid wastewater (COD (chemical oxygen demand) value of 3000 mg L^{-1}) in a series of 250 mL flask and the flasks were sealed and agitated for 24 h at 200 rpm in the thermostatic shaker bath until equilibrium

was reached. At time $t = 0$ and equilibrium, measured COD were used to calculate the adsorption capacity of the adsorbent. The relation between adsorption capacity q_e and equilibrium concentration C_e can be represented by the expression [10]:

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

where q_m is the theoretical maximum amount of organics adsorbed on fresh/regenerated GAC and K_L the Langmuir isotherm constant.

2.2. Electrochemical regeneration

The spent GAC (subjected for equilibrium studies) was filtered and taken for electrochemical regeneration studies. The spent GAC was dried at 105 °C for 12 h, cooled to atmospheric temperature and packed between the electrodes in the electrode chamber. The amount required for filling into the electrode gap was worked out to be 6283.2 g. Electrolysis for GAC regeneration was performed at different current densities with 5% (w/w) Na_2SO_4 supporting electrolyte. Samples of the electrolytic solution were periodically collected from the outlet and immediately analyzed to avoid further reaction. The progress of the destruction of the organic pollutant has been monitored by COD removal estimation. The potentials required for oxidation of organic pollutants are generally high and the production of oxygen from the electrolysis of water molecules may determine the reaction yield. The current efficiency (CE) of the electrolysis can be calculated based on which is defined as follows [11]:

$$CE = \frac{(\Delta COD)V}{32 \times It/4F} \times 100\% \tag{2}$$

The instantaneous current efficiency (ICE) may be computed from (instantaneous) difference between two values of COD of the solution as follows:

$$ICE = \frac{(COD_t - COD_{t+\Delta t})FV}{8 \times I\Delta t} \times 100\% \tag{3}$$

where COD_t and $COD_{t+\Delta t}$ are the values of the chemical oxygen demand at times t and $t + \Delta t$, respectively, V the volume of the effluent in liters, I the current (A) and F the Faraday constant.

If the parameter, ICE is plotted as function of time, the area under the curve integrated, the result divided by the total time (τ) elapsed up to the point when ICE = 0. The above equation can be modified for the present case, i.e. in the electrode chamber packed with GAC, the anodic oxidation synchronously occur on both planner anode and particle electrodes where the applied current is utilized by both the electrodes and fillings:

$$ICE = \frac{(COD_t - COD_{t+\Delta t})FV}{16 \times I\Delta t} \times 100\% \tag{4}$$

3. Kinetic modeling of electrochemical regeneration

It is attempted in the section to develop kinetic model for electro-generation of spent GAC. In electrochemical process, the reaction is controlled by reactions (kinetics) or by diffusion (mass transfer) or some time both depend on the nature of the process. In the present case, the electrochemical chamber is charged with spent organics enriched GAC. The supporting electrolyte (5% Na_2SO_4) was allowed to flow across the electrode chamber filled with spent GAC. When charge is supplied to bed, bipolarity is induced in the GAC particles, which are separated from the adjacent ones, by virtue of the difference of Ohmic drop between the liquid phase and the solid surface, causing Faradaic reaction occurrence on each microelectrodes and electro-oxidation of organics adsorbed on the surface of GAC proceeds [12–15]. Fig. 2 shows a typical current mode for the electrochemical regeneration of spent
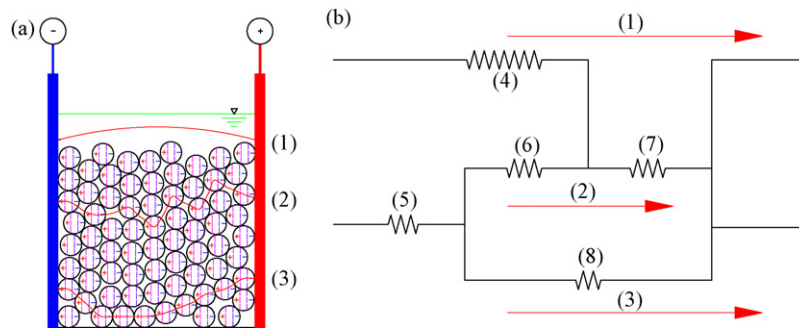
Fig. 2. Current mode for spent GAC regeneration in electrochemical reactor (a) and equivalent circuit of the system (b). (1) Bypass current; (2) effective current; (3) short-circuit current and (4) contact resistance; (5) inter-particle resistance; (6) reaction resistance; (7) inner-particle resistance; (8) bypass resistance.

GAC. It is known fact that the applied current density plays an important role in anodic oxidation of organics, while the bypass and short-circuit current decrease electrical yield. If the reactor is under galvanostatic condition, there are two applied current density associated with the specific surface area of anodes and microelectrodes. Furthermore, the different operation regimes (reaction control and mass transfer control) can be distinguished by the relative values of applied current density, which governs the space–time yield reactant under diffusion control, and limiting current density [16]. The limiting current density per unit of electrode surface can be give as

$$i_{\text{lim}}(t) = nFk_{\text{m}}c(t) \tag{5}$$

where n is the number of electrons transferred, k_{m} the mass transfer coefficient, c the concentration of organics present in the solution.

Eq. (5) can be represented by maximum organic loading at the initial limiting current density:

$$i_{\text{lim}}^0 = nFk_{\text{m}}c_0 \tag{6}$$

The ratio of anodic current density, i_{a}, to initial limiting current density can be written as

$$\alpha = \frac{i_{\text{a}}}{i_{\text{lim}}^0} \quad \text{for } 0 < \alpha < 1 \tag{7}$$

Similarly, the ratio of microelectrode surface area, A_{me} to anodic surface area, A can be given as

$$K = \frac{A_{\text{me}}}{A} \tag{8}$$

The pollutant removal rate, r, can be related to anodic current density as

$$r = \frac{i_{\text{a}}}{nF} \tag{9}$$

The specific area of microelectrode is determined through the particle size of GAC. Therefore, under galvanic conditions, depending on the current density, it is possible to identify three different operating regimes: reaction control ($i_{\text{a}} \leq i_{\text{lim}}$ and $i_{\text{me}} \leq i_{\text{lim}}$), multiphase control ($i_{\text{a}} > i_{\text{lim}}$ but $i_{\text{me}} \leq i_{\text{lim}}$) and mass transfer control ($i_{\text{a}} > i_{\text{lim}}$ and $i_{\text{me}} > i_{\text{lim}}$). It is attempted to analyze the reaction performance under all the three regimes:

(i) Reaction control ($i_{\text{a}} \leq i_{\text{lim}}$ and $i_{\text{me}} \leq i_{\text{lim}}$):
In this case, it is assumed that the reaction is fast (ICE = 1) and the rate is controlled by the kinetics. The material balance for organics in the reactor can be written as

$$\varepsilon V \frac{\text{d}c}{\text{d}t} = -\left(\frac{i_{\text{a}}}{nF}A + \frac{i_{\text{me}}}{nF}A_{\text{me}} \right) \tag{10}$$

where ε refers the void fraction of packed bed.

Substituting Eqs. (7)–(9) into Eq. (10) and integrating with the boundary condition of $c(t) = c_0$ at $t = 0$, results a linear relation between $c(t)$ and t, i.e.

$$c(t) = c_0 \left(1 - \frac{2\alpha Ak_{\text{m}}}{\varepsilon V}t \right) \tag{11}$$

The evolution of the COD in electrolyte will always follow the linear behavior until $i_{\text{a}} = i_{\text{lim}}$ at time t_{F1}. At this stage, the concentration can be calculated by [16]:

$$c(t)_{F1} = \alpha c_0 \tag{12}$$

Substitution of Eq. (12) into Eq. (11) yields t_{F1}, i.e.:

$$t_{F1} = \frac{1-\alpha}{2\alpha} \frac{\varepsilon V}{Ak_{\text{m}}} \tag{13}$$

(ii) Multiphase control ($i_{\text{a}} > i_{\text{lim}}$ and $i_{\text{me}} \leq i_{\text{lim}}$):
In this case, the oxidation on microelectrode is assumed to be under reaction control (ICE = 1), while the electrolysis on planner anode is under mass transfer control. The removal rate can be determined from i_{lim}. The material balance for the reactor can be written as

$$\frac{\text{d}c}{\text{d}t} = -(c + \alpha c_0)\frac{Ak_{\text{m}}}{\varepsilon V} \tag{14}$$

Integration of Eq. (14) with boundary condition of $t = t_{F1}$, and $c = c(t)_{F1}$ results:

$$c(t) = \alpha c_0 \left[2\exp\left(-\frac{Ak_{\text{m}}}{\varepsilon V}t + \frac{1-\alpha}{2\alpha} \right) - 1 \right] \tag{15}$$

Eq. (15) can be used for the estimation of the change of COD in electrolyte from αc_0 to $c(t)_{F2}$ and

$$c(t)_{F2} = \frac{\alpha}{K}c_0 \tag{16}$$

where t_{F2} can be given as

$$t_{F2} = \left(\frac{1-\alpha}{2\alpha} - \ln\frac{1+K}{2K} \right) \frac{\varepsilon V}{Ak_{\text{m}}} \tag{17}$$

The ICE for the present condition is

$$\text{ICE} = 2\exp\left(-\frac{Ak_{\text{m}}}{\varepsilon V}t + \frac{1-\alpha}{2\alpha} \right) - 1 \tag{18}$$

(iii) Mass transfer control ($i_{\text{a}} > i_{\text{lim}}$ and $i_{\text{me}} > i_{\text{lim}}$):
The entire system (reaction at the planner electrode and the microelectrode) is assumed to be under mass transfer control. The rate equation can be written as

$$\frac{\text{d}c}{\text{d}t} = -\frac{2Ak_{\text{m}}c}{\varepsilon V} \tag{19}$$

Table 1
Isotherm constants for the adsorption of organics in DSD acid wastewater onto fresh and regenerated GAC at room temperature.

Regeneration cycle	q_m (mg g^{-1})	K_L (L mg^{-1})	R^2
0 (fresh GAC)	53.48	0.0017	0.9986
1	53.05	0.0013	0.9994
3	52.30	0.0009	0.9987
5	51.25	0.0008	0.9950
7	50.38	0.0007	0.9953

This equation is subject to the following boundary conditions:

$$\begin{cases} t_{F2} = \left(\dfrac{1-\alpha}{2\alpha} - \ln \dfrac{1+K}{2K} \right) \dfrac{\varepsilon V}{Ak_m} \\ c(t)_{F2} = \dfrac{\alpha}{K} c_0 \end{cases} \text{ and } \begin{cases} t = t \\ c(t) = c(t) \end{cases} \quad (20)$$

Subsequent integration results

$$c(t) = \frac{4K\alpha c_0}{(1+K)^2} \exp\left(-\frac{2Ak_m}{\varepsilon V} t + \frac{1-\alpha}{\alpha} \right) \quad (21)$$

$$ICE = \frac{4K}{(1+K)^2} \exp\left(-\frac{2Ak_m}{\varepsilon V} t + \frac{1-\alpha}{\alpha} \right) \quad (22)$$

The organic pollutant concentration in supporting electrolyte (referred in terms of COD) can be estimated using Eqs. (11), (15) and (21) under three different regimes, while Eqs. (18) and (22) give the ICE of GAC regeneration. Theoretical predictions of both COD and ICE were performed for the conditions used in the experimentation and compared with the experimental values.

4. Results and discussion

4.1. Langmuir isotherm

Experiments were carried out covering wide range in operating conditions and the observations are presented in the forms of table and figures. As stated earlier, Langmuir isotherm studies were carried out to get the characteristics of fresh and spent GAC. The linearization of Eq. (1) result:

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{q_m K_L} \quad (23)$$

The binding constant (K_L) and the sorbent capacity (q_m) are estimated by plotting C_e/q_e against C_e and the values obtained from the experiments are given in Table 1. The isotherm studies carried with regenerated GAC by electrochemical regeneration is also given in Fig. 3 for comparison of adsorption capacity of fresh and reused activated carbon. It can be noticed from the figure that the adsorption capacity is in par with fresh activated carbon even after five times of recycling, which is a very interesting observation. It clearly indicates that the electrochemical regeneration does not affect the adsorption capacity of GAC under proper applied current density.

4.2. Evolution of COD and ICE

Fig. 4 shows the COD removal during electrochemical regeneration. It can be observed from the figure that the COD values reduce with increase in the regeneration time and the rate of reduction of COD is sharp at the beginning. Further it can be noticed that the COD reduction increased with an increase in the applied current density. For example, when the current density increased from 200 to 675 A m^{-2} the requirement time for complete degradation decreased from 140 to 34 min. The observation is similar to Zhang [9], Zhou and Lei [17] and Mohan et al. [11]. This can be explained
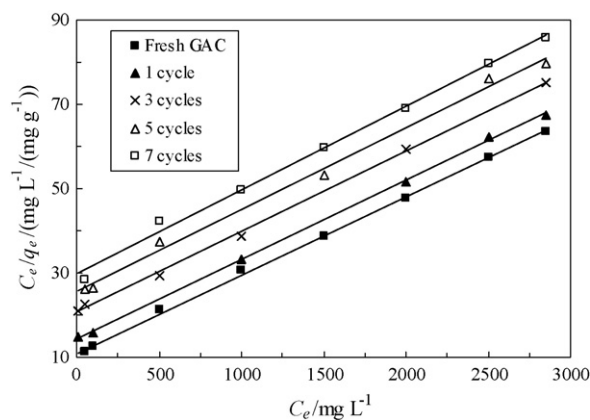

Fig. 3. Comparison of adsorptive capacity of fresh and reuse GAC with different regeneration cycles using linear plots of Langmuir isotherm at room temperature.

that the rate of generation of ·OH ions increases with current density, which eventually enhances the pollutant degradation. At the same time, another phenomenon of the increase of COD during the beginning of the regeneration is observed due to the oxidation velocity is far less than that of desorption one affected by concentration gradient, resulting in the accumulation of organic matters in the electrolyte. Moreover, when the dynamic equilibrium between oxidation velocity and desorption one is achieved, the experimental data could be well represented by the model with extreme accuracy (the correlation coefficients R^2 under 200, 675 and 2533 A m^{-2} are 0.9992, 0.9873 and 0.9253, respectively).

Fig. 5 also shows the variation of ICE with different operating current density estimated using Eqs. (4), (18) and (22). It can be ascertained from the figure that the ICEs keep constant regardless of the values of applied current density initially and decreases beyond certain current density values. The curves in this figure indicate that the proposed model is in good agreement with the experimental results with R^2 of 0.9614, 0.9308 and 0.8924 under 200, 675 and 2533 A m^{-2}, respectively.

4.3. Electrochemical regeneration efficiency

The electrochemical regeneration efficiency (RE) of GAC can be calculated from the values of adsorption capacities for isotherm
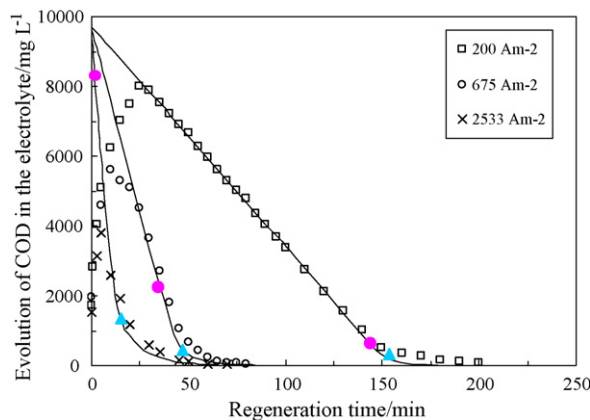

Fig. 4. Comparison between experimental and theoretical values of the evolution of COD with different applied current density in 5% Na$_2$SO$_4$ solution under circulated operation. The continuous lines represent the theoretical approach, while the symbols are the values obtained experimentally. ● and ▲ display t_{F1} and t_{F2} calculated by Eqs. (13) and (17), respectively.
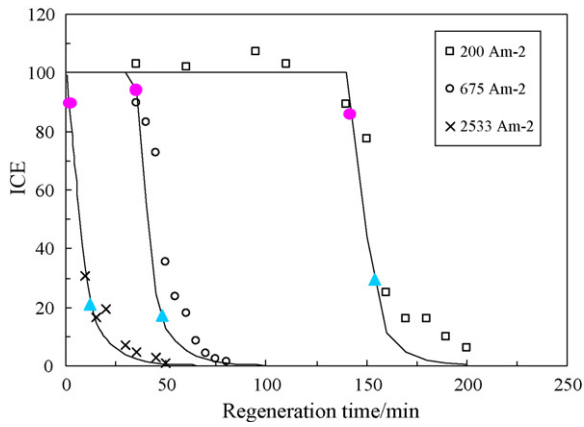
Fig. 5. Comparison between experimental and theoretical values ICE for different applied current density in 5% Na$_2$SO$_4$ solution under circulated operation. Notes of the lines and symbols as in Fig. 4.

studies with fresh GAC and electrochemically regenerated GAC, i.e.:

$$RE = \frac{q_{mr}}{q_{mi}} \times 100\% \tag{24}$$

where q_{mr} and q_{mi} are the adsorption capacities of the regenerated and fresh GAC of the same sample under identical adsorption conditions.

On the other hand, RE is also calculated by the values of COD in electrolyte predicted using the proposed model with the expression of

$$RE = \frac{c_0 - c(t)}{c_0} \times 100\% \tag{25}$$

Fig. 6 shows the RE with respect to regeneration time. It can be seen from the figure that the RE increased with an increase in the regeneration time and approaches its maximum value at proper regeneration time. It can also be noticed that the RE increased with applied current density. In addition, RE of 100% has been achieved for all the process conditions except for 2533 A m^{-2}. Furthermore, higher current density causes the rapid desorption/oxidation behaviors, shortening the time reaching its maximal value of RE. However, when an increasing current is employed, perhaps GAC is also oxidized leading to the destruction of the micropore, which is not favorable for the adsorption of organics as for the decease of its surface area [18].
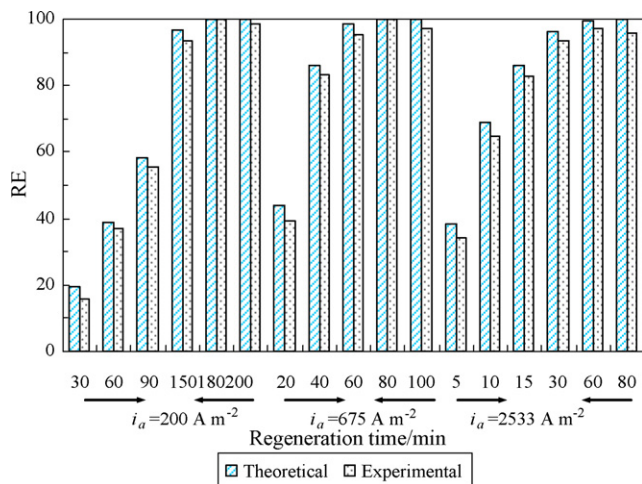

Fig. 6. Comparison between experimental and theoretical data of the RE at different regeneration time with different applied current density in 5% Na$_2$SO$_4$ solution.

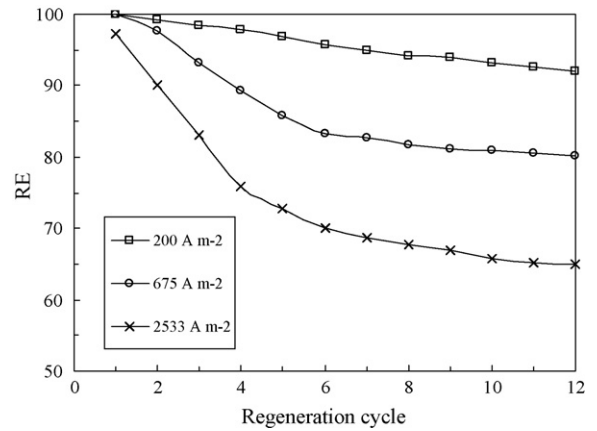**Fig. 7.** Evolution of the RE with regeneration cycles under different current density with regeneration time of 180, 80 and 60 min in 5% Na$_2$SO$_4$ solutions, respectively.

The theoretical RE can be estimated using Eq. (25). For comparison purpose, the model prediction on RE also plotted in Fig. 6. It can be observed that the model predictions match satisfactorily with the experimental data under all operating conditions. This indicates that the evolution of COD in electrolyte could be used for the evaluation of recovery degree of spent GAC.

4.4. Regeneration cycles

It is also attempted to study the influence of regeneration cycle on RE which has significant commercial impact. The repeated regeneration of GAC was conducted under the same operating conditions. It can be ascertained from Fig. 7 that the rate of decrease in the RE is marginal for lower current density and significant at higher current densities. For example, the RE is more than 90% even after regeneration cycle of 10 at current density of 200 A m^{-2} while the RE is reduced to 78% within regeneration cycle of 4 when operated at current density of 2533 A m^{-2}. This can be explained that at higher current densities the micropores of GAC may be easily destroyed by strong electric field. The RE remains constant with regeneration cycles at low current densities, which shows the similar trend to our earlier observation with three-dimensional electrode for phenolic wastewater treatment [19]. The results clearly demonstrate that electrochemical regeneration is a better choice for recovery of the adsorption capacity of spent GAC.

5. Conclusions

Experiments were carried out to study the feasibility of regeneration of spent GAC using a novel technique of electrochemical regeneration in a semi-batch reactor filled with a 5% Na$_2$SO$_4$ solution as supporting electrolyte. The following conclusions can be made from the present investigations:

- The spent GAC can be effectively regenerated using electrochemical method and a complete 100% regeneration is possible obtained under optimum conditions.
- The adsorption capacity of GAC is not obviously affected during electrochemical regeneration if operated under proper applied current density. The Langmuir isotherm studies for recycled GAC shows that the recycled GAC perform in par with fresh GAC even after several cycles of regeneration.
- A kinetic model has been developed for electrochemical regeneration of spent GAC and the proposed model simulations match satisfactorily with the experimental conditions. That is to say, the electrochemical performance indicator, ICE and RE estimated by

the suggested model was in a good agreement with the experimental results.

Acknowledgement

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