

Research Article

Development of Activated Carbon from Cotton Fibre Waste as Potential Mercury Adsorbent: Kinetic and Equilibrium Studies

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The study attempted to develop the activated carbon of cotton fibre (ACCF) from cotton waste as a high Hg^{2+} adsorbent media and characterize physicochemical properties using scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) and Hg^{2+} adsorption kinetic by batch adsorption study with the function of contact time, solution pH, dosages of ACCF, and Hg concentration. The SEM-EDS study revealed that ACCF is composed of carbon (95.1%) and phosphorus pentoxide (4.9%). Obtained results of adsorption kinetics showed that 15 min of contact time is required to achieve the equilibrium state and wide range of pH (4.08–7) is favourable for maximum Hg adsorption. The Hg^{2+} adsorption capacity showed a decreasing trend with increasing dose of ACCF, whereas a reverse response of adsorption capacity was pronounced with increasing Hg concentration. The data was well described by Freundlich isotherm model and determined the high Hg^{2+} adsorption capacity of ACCF (169.2 mg/g). To our knowledge, the application of ACCF in removing Hg^{2+} is the first study. High Hg^{2+} adsorption capacity, economic feasibility, availability of cotton fibre waste, and simple preparation method concluded that it could be used as a novel low-cost and environmentally sound adsorbent media for removing high rate of Hg^{2+} from aqueous phase.

1. Introduction

Mercury (Hg) is one of the top ten most toxic and hazardous pollutants in the Priority List of Hazardous Substances [1] for causing serious human and environmental health risks by bioconcentration, bioaccumulation, and biomagnification phenomena. Hg and its derivatives can be adsorbed through the gastrointestinal tract, skin, and lungs and hence it is recognized as dangerous and insidious poisons [2]. The monomethylmercury (MeHg^+) or dimethylmercury (Me_2Hg) can penetrate through cell membranes within seconds and cross the blood-brain barrier, which results in the induction of serious carcinogenic, mutagenic, teratogenic, tyrosinemia, paralysis, serious neural, intestinal, and renal disorders in the organ systems. The toxic, nonmetabolic function, conversion of lower to higher toxic forms, bioaccumulation, and serious health disorders on animal systems are the significant criteria of Hg [2–5]. The severity of Hg toxicity was recognized in the late 1950s and 1960s because

of an environmental tragedy in Minamata Bay, Japan, where hundreds of individuals suffered from mercury poisoning (Minamata disease) [6]. The US Environmental Protection Agency (EPA) estimated 630,000 newborns in America are at risk for unsafe levels of Hg exposure [7].

The major sources of Hg contamination in the environment are geogenic, anthropogenic, and reemitted Hg [8]. The geogenic sources are volcanic activity and weathering of rocks and the major industrial sources are coal burning, electronic, paper, pharmaceutical industries [9, 10] chlor-alkali, paint, pulp and paper, oil refinery, electrical, rubber processing, and fertilizer industries [11, 12]. In particular, the World Health Organization (WHO) reported that between 25,000 and 125,000 tons of Hg were released into the environment in 1976 [13]. To discharge heavy metals into the environment, the WHO set up various permissible limits. According to USA's Environmental Protection Agency (EPA) permissible limit of Hg in wastewater is nil; according to the WHO [14] the limit for mercury in drinking water is 0.001 mg/L and

according to the Bureau of Indian Standards it is 0.01 mg/L for wastewater.

Due to indiscriminate discharge, environmental hazardous, and severe harmful impacts on life, it is necessary to remove Hg from wastewaters before they are discharged into the environment. In order to remove the Hg from water, the adopted common methods include sulfide precipitation, ion exchange, alum and iron coagulation, electrodialysis, ultrafiltration, activated carbon adsorption, and various biological processes. Activated carbon (AC) has been widely used as an important adsorbent to remove various pollutants especially Hg from wastewaters due to having their well-developed porous structure, large active surface area, and good mechanical properties [15–19]. The AC can be prepared from a variety of materials. Commercially available AC in the market derived from peat, lignite, and wood are expensive. Though varieties of AC have been developed recently from apricot stones, nut shells, grape seeds maize cobs, rice husk, coconut husk, wood almond, hazelnut shells, pine timber, pine scrap, and jute sticks to adsorb Hg from water, but many of them are costly and/or inaccessible and/or ineffective in the practical application. In this regard, therefore, the low-cost Hg adsorbent materials with easy application methods still have demand. So there is a need to produce low-cost AC that can be prepared following the simple process and applied to pollution control [20]. Some studies prepared AC from cheaper and readily available materials for Hg removal [12, 20–24].

Activated carbon fibres are a comparatively modern form of porous carbon material with a number of significant advantages over the more traditional powder or granular forms. These include high surface area and adsorption capacity, as well as very high rates of adsorption from the gas or liquid phase. Nabais et al. developed AC fibres by microwave heating from acrylic textile fibres [25]. The cotton stalk can be used to prepare AC by the processes of zinc chloride [26] and phosphoric acid [16] activation. Kawasaki et al. proposed that the low-cost fibrous AC can be produced, since cotton fiber retains its fibrous form under carbonization [27]. Though the study concerning the preparation of AC from the cotton fibre waste has been performed, its application as adsorbent media for removing the pollutants from water has not been executed so far. Besides, the disposal and incineration of cotton waste derived from the various modern anthropogenic activities is one of the troublesome problems in the environment by generating huge waste as well as green house gas CO₂. From this point of view, the properly collection keeping in mind the environmental aspect of waste management and conversion of cotton fibre waste into high surface area containing adsorbent media with high adsorption capacity of various environmental pollutants may be a novel and emerging solution in this respect. Therefore, the purposes of the present study were to develop the AC of cotton fibre (ACCF) as a novel and potential Hg²⁺ adsorbent media from waste of cotton fibre and characterize the Hg²⁺ adsorption kinetic by batch adsorption study for application in removing high rate of Hg²⁺ from the aqueous phase.

2. Materials and Methods

2.1. Preparation of Activated Carbon of Cotton Fibre (ACCF). The AC of cotton fibre (ACCF) was prepared by the process of microwave assisted pyrolysis. Microwaves can be used to pyrolyze the carbon source [25, 26, 28]. Waste cotton (used for microbiological work) of the laboratory was collected, extensively washed under tap water to remove any particulate, and finally sprayed with distilled water. This washed cotton was dried in sunlight. To pyrolyze, the cotton fibre (50 g) was placed within a lidded porcelain cup which in turn was placed inside the multimode microwave and heated at >500°C for 10 min. The temperature of the carbon bed during microwave treatment was measured using an infrared optical pyrometer. Activated carbon manufacturing consists of a charring or carbonization step in which most of the noncarbon material (and much of the carbon) is volatilized by pyrolysis (usually between 500 and 750°C) and usually weight (60 to 70%) is lost [29–31]. The basic microstructure of the char with microporosity is formed around 500°C [31]. After cooling, the ACCF was crushed into powder and stored in a sealed glass container for using in different studies.

2.2. Physicochemical Properties of ACCF Adsorbent. The physical and chemical characterizations were carried out by capturing images of surface morphology and analyzing the chemical composition of the ACCF using scanning electron microscopy (SEM) with a jeol model equipment coupled with energy dispersive spectroscopy (EDS) facility (JSM-6500F, JEOL) in the Center for Advanced Marine Core Research, Kochi University, Japan, following the standard process [32].

2.3. Batch Adsorption Experiment. The effect of process parameters (contact time, pH, dosage of ACCF adsorbent, and initial concentration of Hg) on the Hg²⁺ adsorption property of ACCF were performed in 100 mL capped glass bottles following the batch operation mode. All experiments used the known volume (50 mL) of Hg solution with known concentration as adsorbate and known weight of ACCF as adsorbent. The Hg solution containing capped glass bottles were shaken by mechanical shaker at the rate of 150 excursion/min within a temperature controlled incubator at 25°C.

The effects of contact times were determined maintaining the 0.15 mg/L initial concentration of Hg solution with pH 4.08 and 0.05 g/L dosage of ACCF. The influences of different pH values (4.08, 5.08, 6.08, 7.0, 8.12, 10.04, and 11.98) of Hg solution were examined keeping 0.15 mg/L initial concentration of Hg solution and 0.05 g/L dosage of ACCF. The effects of initial Hg concentrations were evaluated using 0.05, 0.1, 0.15, 0.2, 0.3, and 0.4 mg/L Hg solutions with pH 4.08 and 0.05 mg/L dosage of ACCF, whereas the effects of ACCF dosages were determined using 0.0125, 0.025, 0.05, 0.1, 0.2, and 0.5 g/L of ACCF maintaining the 0.1 mg/L initial concentration of Hg solution with pH 4.08. Each experiment was performed at least twice following the identical conditions using controls of only Hg solution and adsorbent without Hg in solution.

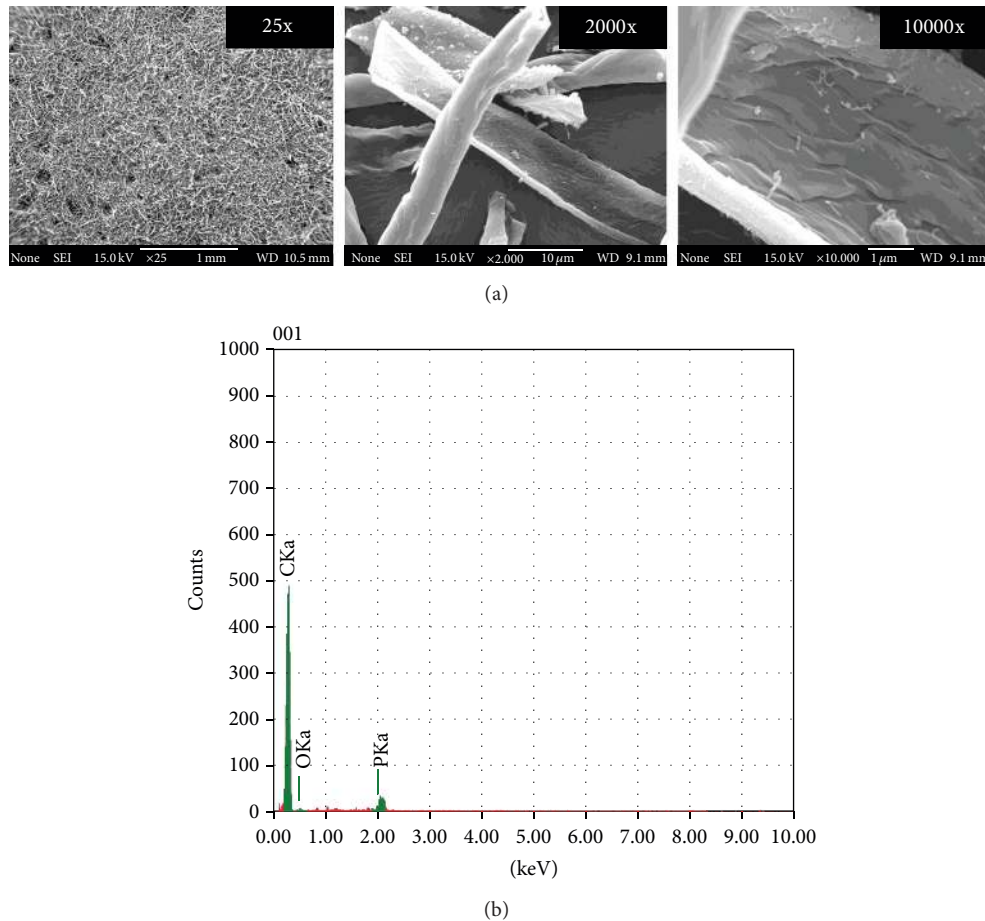


FIGURE 1: SEM macro- and microimages in three magnifying stages (25x, 2000x, and 10000x) (a) and EDS spectrum (b) of ACCF employed in the experiment.

The Hg^{2+} adsorption capacity (q_e , mg/g) of ACCF at the equilibrium condition of the different experiments performed above was calculated following the mass balance relationship as

$$q_e \text{ (mg/g)} = \left[\frac{C_i - C_e}{C_i} \right] \times V, \quad (1)$$

where C_i (mg/L) and C_e (mg/L) are water-phase concentrations of Hg at the initial and the equilibrium states, respectively, V (L) is the volume of the Hg solution, and M (g) is the mass of ACCF.

The percentage of Hg removal from water phase was calculated using

$$R_e \text{ (%) } = \left[\frac{C_i - C_e}{C_i} \right] \times 100, \quad (2)$$

where R_e is the percentage (%) of Hg adsorbed and C_i and C_e are the initial and equilibrium concentrations of Hg (mg/L) in the solution, respectively.

2.4. Hg Analysis. The water samples were collected and centrifuged and supernatant was used for analysis using

the RA-3 Mercury Analyzer (Nippon Instruments Corporation, Japan). Hg removed from water was calculated by quantifying the residual Hg content in water phase.

3. Results and Discussion

3.1. Physicochemical Properties of ACCF. Figure 1(a) shows the SEM images of the developed ACCF adsorbent. These images displayed the morphological properties and megascopic and microscopic textures of the ACCF in three magnifying stages (25x, 2000x, and 10000x). Megascopic images (25x and 2000x) give the shape and size of the ACCF, whereas microscopic images (10000x) clearly showed the elaborated surface structure of the ACCF.

Figure 1(b) represents the EDS analysis for chemical composition of ACCF. The EDS data also exhibited that ACCF is composed of carbon (95.1%) and phosphorus pentoxide (4.9%). The large percentage of carbon constituent of ACCF indicating the availability of large surface area developed by microwave heating process, which is the important quality of an excellent adsorbent of AC. Generally, AC possesses an extraordinarily large surface area and pore volume that gives it a unique adsorption capacity [33].

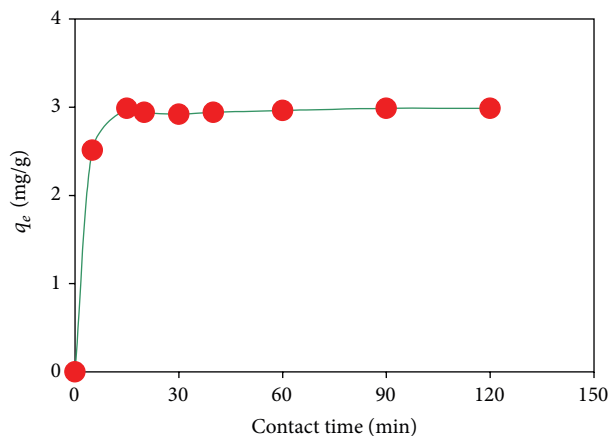


FIGURE 2: Effect of contact time on Hg^{2+} adsorption of ACCF. (Initial concentration 0.15 mg/L, ACCF dosage 0.0025 g/L, pH 4.08, and contact time 120 min).

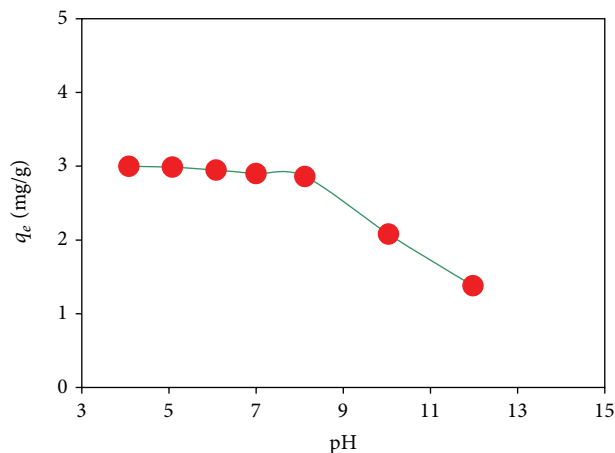


FIGURE 3: Effect of pH on the of Hg^{2+} adsorption of ACCF. (Initial concentration 0.15 mg/L, contact time 15 min, and ACCF dosage 0.0025 g/L).

3.2. Hg Adsorption Kinetics of ACCF

3.2.1. *Effect of Contact Time (t).* The effects of contact times on the Hg^{2+} adsorption of ACCF from water phase were depicted in Figure 2. The ACCF showed a rapid adsorption rate of Hg^{2+} and equilibrium state of adsorption was achieved at 15 min contact period. The percentage of Hg^{2+} adsorption (0–96.3%) increased with time and maximum percentage of adsorption was found at 15 min. The equilibrium contact times of most of the adsorbent media are much higher than that of the ACCF. Therefore, it is pertinent to mention here that the ACCF achieved the equilibrium state within a narrow time period, which indicated its significantly rapid rate of Hg^{2+} adsorption properties that is helpful for treating the massive amount of Hg contaminated water within a short time span.

3.2.2. *Effect of pH.* Figure 3 elucidates the effects of initial pH of solution on the Hg^{2+} adsorption process of ACCF. The adsorption capacities (q_e , 2.9–2.98 mg/g) exhibited a steady state from pH 4.08 to 7 and sharply decreased (from 2.9 to 1.38 mg/L) thereafter with increasing pH. The percentage of Hg^{2+} adsorption varied from 44 to 96.7% and maximum adsorptions were recorded between pH 4.08 and 7. It indicated that the pH plays an important role in the Hg^{2+} adsorption mechanism of ACCF and slightly acidic or neutral initial pH of solution (i.e., from 4.08 to 7) is optimum for adsorbing the maximum Hg^{2+} from water phase. It has been shown that pH 7 is the optimum for Hg^{2+} adsorption [17] and highest arsenic adsorption was also reported at pH 7.5 [34] by AC and ceramic. The results also clearly signified that the developed ACCF can work efficiently in wide range of pH. Activated carbon is also equally absorbent for both ionic and neutral species and is the most universal absorbent for complex mixtures of chemicals structures [35, 36]. The strengths of AC as a poison antidote and water purification aid are its great absorption capacity, and its ability to absorb many chemicals with different structures [35]. Ion exchange

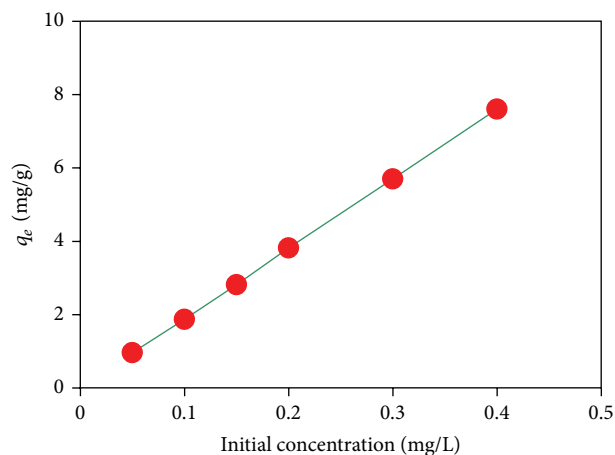


FIGURE 4: Effect of initial Hg concentration on the of Hg^{2+} adsorption of ACCF. (ACCF dosage 0.0025 g/L, pH 4.08, and contact time 15 min).

resins exist that are better absorbers than AC for either acidic or basic chemical species but not for neutral chemical species.

3.2.3. *Effect of Hg Concentration (C_i).* The effect of initial Hg^{2+} concentrations on the adsorption process of ACCF was shown in Figure 4. The Hg^{2+} adsorption (q_e , 0.9–7.6 mg/g) increased with increasing the initial Hg^{2+} concentration of solution. The maximum Hg adsorption percentage (96%) was observed in the highest initial concentration (0.4 mg/L). The obtained results implied that the saturation rate of Hg^{2+} binding onto the functional groups of ACCF was greater in the higher concentrations by means of increasing electrostatic interactions which resulted in the increasing rate of adsorption.

3.2.4. *Effect of Adsorbent Dosage (M).* Figure 5 represents the effects of adsorbent dosages on the adsorption of Hg^{2+} onto

the ACCF. The total amount of Hg^{2+} adsorption increased with increasing the dosages of ACCF, whereas the adsorption capacity (q_e , 0.19–6.64 mg/g) of ACCF showed a reverse response with dosages. The percentage of adsorption varied from 83 to 99% in all dosages employed. The adsorption capacity and percentage were maximal at the dosages 0.0125 and 0.5 mg/L, respectively. It has been proposed that the electrostatic interactions, interference between binding sites, and reduced mixing in higher densities of higher adsorbent doses are responsible for decreasing the adsorption capacity (q_e , mg/g) with increasing adsorbent dosages [37, 38]. The obtained results are supported by the results of sequential adsorbent treatment using solid adsorbents (such as sand, silica, coal, and alumina) proposed by Yabe and de Oliveira [37].

3.3. Adsorption Isotherms. The adsorption phenomenon is usually studied through adsorption isotherm, which indicates the relationship of the amounts of adsorbate in the liquid phase adsorbed onto the surface of the adsorbent at the equilibrium condition at constant temperature. Adsorption isotherms are most commonly used to select the adsorbent or even the adsorption process as a unit operation for the adsorptive separation by evaluating the efficiency of an adsorbent. Thus, the adsorption isotherms are useful to design and optimize the adsorption systems [38, 39]. Generally, the Langmuir and the Freundlich equations are commonly used to evaluate such isotherm models. Therefore, the Langmuir isotherm and the Freundlich isotherm models have been studied to describe the data of adsorption experiments of present study, which show the different adsorption mechanisms. The present study considered these two isotherms using (3)-(4) [40] to describe the data obtained.

Equation (3) of the Langmuir adsorption isotherm is as below:

$$q_e = \frac{q_m b C_e}{1 + b C_e}, \quad (3)$$

$$\frac{C_e}{q_e} = \frac{1}{b q_m} + \frac{C_e}{q_m},$$

where q_e is the mass of the contaminant adsorbed per unit weight of the adsorbent (mg/g), C_e is the equilibrium concentration of the Hg in solution (mg/L), q_m is the monolayer adsorption capacity (mg/g), and b is the Langmuir constant related to the free energy of adsorption (L/mg). The Langmuir parameters and correlation coefficient were obtained by fitting the experimental data to the linearized equation derived from (3), which are represented in Table 1. The Langmuir adsorption isotherm assumes that adsorption takes place at definite points of attachment on the surface of the adsorbent and has found successful application too many sorption process of monolayer adsorption.

Equation (4) of the Freundlich isotherm is as follows:

$$q_e = K_f C_e^{1/n}, \quad (4)$$

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

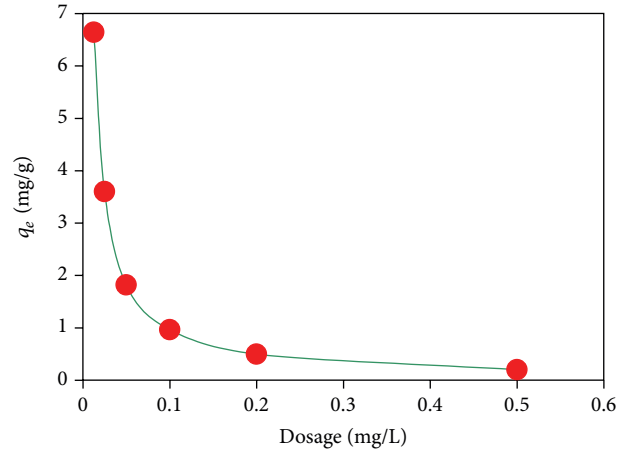


FIGURE 5: Effect of adsorbent dosage on the of Hg^{2+} adsorption of ACCF. (Initial concentration 0.15 mg/L, pH 4.08, and contact time 15 min).

TABLE 1: Freundlich and Langmuir parameters for the Hg^{2+} adsorption of ACCF.

Adsorbent	Freundlich			Langmuir		
	K_f (mg/g)	$1/n$	R^2	q_m (mg/g)	b (L/mg)	R^2
ACCF	169.2	0.837	0.974	22.27	21.41	0.287

where q_e is the mass of the contaminant adsorbed per unit weight of the adsorbent (mg/g), C_e is the equilibrium concentration of the cadmium in solution (mg/L), K_f is the adsorption capacity of the Freundlich constant (mg/g), and n is the adsorption intensity of Freundlich adsorption isotherm constant. The Freundlich adsorption parameters are shown in Table 1.

The Hg^{2+} adsorption isotherm of the ACCF is represented in Figure 6. The correlation coefficients of the Freundlich and the Langmuir models are 0.974 and 0.287, respectively, which clearly demonstrated that the Freundlich isotherm is well fitted with the experimental data rather than the Langmuir isotherm and it cannot be mentioned about the monolayer coverage of Hg^{2+} ions on ACCF. The adsorption capacity (K_f) is 169.2 mg/g which indicated that ACCF has high Hg^{2+} adsorption capacity. It is generally stated that the values of n in the range of 1 to 10 represent good adsorption [41]. In this study, the Freundlich constant n was 1.19, which also implied the favorable adsorption. This high adsorption capacity is probably due to having high active surface area and well as unique electrical double layer of AC [42].

3.4. Management of Spent AC. The AC carbon does not itself appear on any of the lists of hazardous substances [43]. Activated carbon used to treat hazardous waste could be considered to be hazardous waste itself [44]. Though it was not considered here, the Hg loaded spent AC can be recycled, reactivated, or regenerated using thermal or hydrothermal methods to extract/recover Hg [45] from ACCF in order to avoid the further hazardous environmental problems.

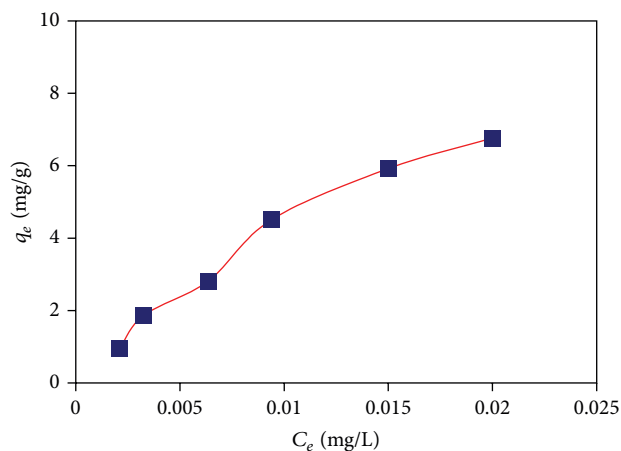


FIGURE 6: Equilibrium curve for the adsorption of Hg^{2+} on ACCF.

4. Conclusions

This study developed an AC of high Hg adsorbing capacity (169.2 mg/g) from cotton fibre waste using the microwave assisted heating process. The batch adsorption experiments clearly revealed the significant influence of various environmental process parameters on the Hg adsorption capacity of ACCF. The study proposed that contact time 15 min and wide range of pH (4.08–7) are to be optimal for maximum Hg adsorption. The results also showed an increasing Hg adsorption capacity of ACCF with increasing initial concentration of Hg, whereas a reverse response of adsorption capacity was found in the effect of ACCF dosages. The adsorption isotherm determined that the resultant data of adsorption kinetic is well fitted with the Freundlich isotherm model which largely indicates its efficiency in practical field of application. The high surface area of ACCF is the major driving factor for adsorbing high amount of Hg from water phase. In spite of above properties, the economical feasibility, availability of waste cotton and simple preparation method concluded that it could be used as a novel low-cost adsorbent for removing Hg from water by converting the cotton waste into environmentally sound adsorbent media of AC with high Hg adsorbing capacity.

Conflict of Interests

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

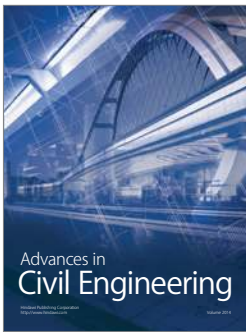
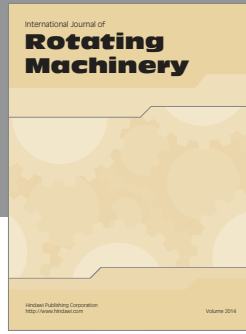
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