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Investigation on adsorption performance of activated carbon prepared from municipal solid waste for the removal of pollutants from refinery wastewater

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Disposal of municipal solid waste into open landfills is a real challenge, and it causes severe environmental and health issues. Utilization of municipal solid wastes for the production of activated carbon (AC) is a viable alternative to conventional techniques as it supports the concept of circular economy. The most commonly employed solid waste disposal methods are incineration followed by landfill, which has adverse effect on environment by releasing greenhouse gases and ash into environment. Therefore, this study is aimed to produce activated carbon from municipal solid waste (MSW) using pyrolysis process in the absence of oxygen by exposure to high temperature at 550 ℃ for 2 h followed by thermal activation at 600 ℃. The resulting activated carbon has been employed in the removal of pollutants from refinery wastewater by investigating the influence of effluent pH, agitation time, quantity of activated carbon and agitation speed on adsorption efficiency. The characterization techniques employed are Fourier Transform infrared spectroscopy, X- Ray diffraction and scanning electron microscopy. The treatment efficiency has been analyzed for total dissolved solids, total suspended solids, chemical oxygen demand, and dissolved oxygen and the optimum processing conditions are decided. The optimized processing parameters obtained are pH 6.0, 80 min stirring time, 75 RPM agitation speed with an activated carbon dosage of 2.0 g. The study suggests that municipal solid waste is a prospective raw material for activated carbon production and an effective adsorbent for the effective removal of different types of pollutants from refinery wastewater. The conversion of municipal solid waste into AC is an adaptable and economically viable source of carbon and protecting the environment and human health.

Keywords: Activated carbon, Chemical oxygen demand, Municipal solid waste, Pyrolysis, Refinery effluent, Turbidity

Introduction

The worldwide generation of municipal solid waste (MSW) surpasses 2 billion tons a year; and it is expected to increase to 3.5 billion tons by 2050. Around 350 landfills or dumpsites are currently available in Oman, which are managed by different municipalities. In the year 2011, the first engineered landfill in the Sultanate of Oman was commissioned for managing the MSW in a well-ordered and eco-friendly approach[1]. The disposal of solid waste into landfill affects the entire ecosystem due to its high toxicity, mutagenic and carcinogenic nature. The release of obnoxious gases during waste disposal stage is strongly relying on the variety of waste materials and the process parameters. Advancement of sustainable technologies and economically viable materials that are eco-friendly and satisfying the stringent environmental regulations is a trend in the scientific community. Effective solid waste management system utilizing bio wastes has been a hot topic of research in recent times.

MSW composed of food wastes, heterogeneous mixture of residential, commercial, and industrial wastes, wood, textiles, metals, and plastics to biomedical wastes[2]. Urbanization, industrial activities, population growth and agricultural activities are the main sources of municipal solid waste. Majority of these solid wastes are improperly managed and are dumped into landfills or incinerated and causes environmental pollution[3]. The accumulated solid waste results in uncontrolled release of greenhouse gases, shortage of waste disposal sites and builds non sustainable waste management[4]. Incineration of solid wastes leads to greenhouse gas emission and hence contributes global warming. Majority of the countries largely rely on landfill due to economical and easier disposal options. MSW from landfills contain heterogeneous materials with hazardous and non-hazardous nature which has an adverse negative effect on environment[5]. Sultanate of Oman generates more than 5000 tons of MSW per day and management of such solid waste is a serious concern. Municipal solid

waste management is a hot topic of interest to the scientific community. Despite the fact that some of the wastes are recycled, remaining are disposed at neighbouring landfills within the country[6]. The dump sites normally release organic pollutants, volatile organic compounds, toxic leachate, and greenhouse gases to the surroundings[7]. Organic wastes present in these landfills produce undesirable odors, volatile organic compounds (VOC's), methane and carbon dioxide which are known for their effects as greenhouse gases. Biochar composed of carbonaceous substances that are applied as soil fertilizer for pollutant removal through its carbon capture and storage capability[8].

Pyrolysis is employed as a thermal treatment of a substance at high temperatures (300 ℃- 800 ℃) in the absence of oxygen[9]. Biochar produced from pyrolysis process exhibits improved surface area, high porosity and high carbon content and those properties solely depend on the type of feed material, characteristics of the municipal waste in terms of composition of plastic/organic waste materials, and the pyrolysis temperature [10]. The high carbon content and active surface functional groups of bio char are suitable for the removal of heavy metals, organic and inorganic contaminants [11-15]. Biochar also plays a key role in catalytic degradation of toxic pollutants [16, 17]. The biochar is activated with an acid or base to enhance its adsorption capacity. Chemical activation process has shown improved surface area and porosity [18, 19]. A meta-analysis on the environmental and economic performance of biochar displayed minor impact on global warming [20]. Recent studies show the capability of biochar and its potential in wastewater treatment applications [21]. Biochar derived from landfill minimizes the environmental impact and offers a promising solution to address the environmental challenges. Pyrolysis and chemical activation will be considered as a better choice to manage solid waste and hence reduce the dependency of landfill [22]. The microwave assisted carbonizations of MSW have been studied by varying the temperature, particle size, carbonization period and electric power requirement [23]. Another study focused on the combined microwave irradiation and impregnation of KOH to activate heterogeneous composite wastes [24]. Thermochemical conversion of municipal solid waste into energy and hydrogen are assessed for an alternative technique for circular economy[25]. The recent study demonstrates that the pyrolysis product is a new and cost effective adsorbent with potential application in waste water treatment, production of polypropylene composites and to suppress carbon dioxide (CO_2) emissions [26].

Activated carbon is considered as one of the promising materials in removing organic pollutants and CO_2 adsorption due to its excellent performance and adsorbent regeneration capacity [27]. Municipal solid waste is generated in excess quantity; therefore, conversion of the waste into activated carbon could be a better alternative to address the issues related solid waste management by reducing the greenhouse gas emission. Additionally, the yield of pyrolytic char is nearly 35%, which can be used to produce AC [29]. The appealing properties of excellent adsorption capacity, cost effectiveness, safety and durability of AC make it suitable for wastewater treatment applications [30]. The ideology of sustainable development is to support people to lead a healthy, economically balanced and environmentally friendly life by enhancing the living standards [31]. Therefore, more emphasis must be given to process MSW, as it is one of the major concerns owing to the limited availability of disposal sites.

Petroleum refinery is one of the largest pollution causing industries and inadequate disposal of wastewater results in contamination of land and water. Therefore, the entire ecosystem gets polluted; this results in environmental issues and also affects human health. The main sources of water pollution in refinery are from crude oil distillation unit, thermal, catalytic and hydrocracking units with different organic and inorganic contaminants of phenols, mercaptans, oil, chlorides, cyanides, sulfides etc [32]. Hence it is essential to implement an appropriate water management system for the safe disposal of effluents into the environment. The refinery wastewater contains considerable quantity of organic and inorganic pollutants [33]. The typical refinery wastewater treatment technique consists of physical, chemical and biological methods utilizing activated sludge process, sequencing batch reactor (SBR), Electro-coagulation, Membrane bioreactor process, and Fenton Process [34]. Each treatment method has its advantages, shortcomings, and operational conditions [35]. These conventional treatment techniques produce huge quantity of oily sludge during each treatment steps and constructed wetland are in place to control the sludge volume [36]. Therefore, the current research investigates the conversion of MSW from Oman's landfill into activated carbon for refinery waste water treatment applications.

Experimental Section

The solid waste samples were collected from a local landfill in the Al-Amerat region of Oman. The MSW composed of polyethylene terephthalate bottles, poly vinyl chloride pipes, plastic wraps, dried date palm leaves, ligno-cellulosic fiber, paper and cardboard. Refinery wastewater was received from a leading refinery in Oman, Petroleum Development Oman (PDO). The pyrolysis process was performed in an Electric furnace (Nabertherm, 30-3000 ℃). KOH pellets were obtained from Sigma Aldrich, USA. The characterization methods employed are FTIR (Perkin Elmer Frontier), XRD (Malvern PANalytical) and SEM (JEOL JSM-7600F). For SEM analysis, the samples were sputter coated with gold to reduce the charging effect. TDS, TSS, DO are measured using Water analysis kit (Microprocessor Water & Soil Analysis Kit – 1160). Millipore water (18.2 MΩ resistivity) was used to perform experiments. COD measurements were carried out in a Thermo Reactor (Orion COD 125) and Turbidity meter (WTW Turb 550). The average particle size of the feedstock was 200 mm. All experiments were repeated four times to confirm the accuracy of the readings and the mean value of four data sets are considered as the final value. Table 1 represents the composition analysis of MSW samples collected from Al Amerat landfill. Table 2 illustrates the characteristics of the wastewater.

Table 1 — Composition of municipal solid waste collected from Al Amerat landfill

Solid waste category (w/w)	Percentage
Food	8.2
Paper	5.7
PET Plastic bottles	4.8
Plastic wraps	23.6
Metals	2.1
Cardboard	13.6
Glass	2.4
Wood	2.3
Textiles	6.9
Green waste	6.1
Dried date palm leaves	21.0
Other combustibles	2.1
Ligno cellulosic fibre	1.2

Table 2 — Characteristics of refinery wastewater

Parameters	Mean Value
COD, mg/L	2341
pH	6.0
TSS, mg/L	320
TDS, mg/L	432
DO, mg/L	209
Turbidity, NTU	100

Preparation of activated carbon

The pyrolysis of MSW was performed in a furnace operated at 550℃ for a period of 2 h. Chemical activation of biochar alters the functional groups and bonds and also helps to alter the surface properties and enhance its capability to adsorb certain species [37]. The chemical activation of biochar was carried out by mixing 1: 4 ratios of biochar and KOH (3% by weight) solution for 48 h and then heated in a furnace up to 600 ℃ for 1.5 h to obtain activated carbon. The resulting mixture was centrifuged at 5000 RPM for 5 min to separate the activated carbon followed by repeated washing with water and dried to 115 ℃ for 2 h. The dried sample was transferred to the furnace for thermal activation at 600 ℃ for 90 min before shutting off the furnace and allowing the sample to attain room temperature. The product, activated carbon was used for the characterization and batch treatment of refinery wastewater.

Characterization Techniques

Fourier transform infrared spectroscopic (ATR-FTIR) analysis of the prepared activated carbon was carried out at a resolution of 4 cm^{-1} and 32 scans with wavenumber between 400 cm^{-1} and 4000 cm^{-1}. The FTIR spectrum was recorded using KBr method. The various surface functional groups present in the sample and bonds were recorded using FTIR analysis. The surface morphology and microstructural characterizations were analyzed by scanning electron microscopy (SEM). The imaging was done by placing the sample in a stub followed by gold sputtering to make the sample conductive. The X-Ray diffraction (XRD) analysis was performed at a diffraction angle (2θ) ranging from 10–80° using a high-precision X-Ray diffractometer (Malvern PANalytical - AERIS). The wastewater was received from Petroleum Development Oman and the characterization was done for both raw and treated water for COD, DO, TSS, turbidity and TDS using APHA standards.

Refinery wastewater treatment

The treatment of refinery wastewater was performed by changing the processing conditions of wastewater pH, duration of mixing, speed of agitation, and mass of activated carbon and subsequently, the optimum processing conditions were established by recording the total dissolved solids (TDS), total suspended solids (TSS), chemical oxygen demand (COD), and dissolved oxygen (DO) and turbidity values. The wastewater treatment process was studied in a batch mode by mixing a

known amount of activated carbon with 250 mL of refinery wastewater under continuous stirring from 0 to 100 rpm speed followed by collecting samples at each time interval. The treatment efficiency was determined by collecting the samples at specified time intervals and analyzed for various process parameters of COD, DO, TSS, TDS, and turbidity. The pollutant removal efficiency of activated carbon was calculated using the Eq. (1).

$$\% \text{ Removal efficiency} = \frac{A-B}{A} \times 100 \quad \ldots(1)$$

Where, 'A' and 'B' are the initial and final values of COD, DO, TSS, TDS in mg/L.

The adsorption data were analyzed by Freundlich isotherm model and its mathematical form is

$$q_e = k \times C_e^{1/n} \quad \ldots(2)$$

where q_e is the adsorption capacity, and C_e is the equilibrium concentration of solution after adsorption,

The linearization of equation (2) shows,

$$\ln q_e = \ln k + \frac{1}{n} \ln C_e \quad \ldots(3)$$

The values of 1/n and the intercept ln k are obtained from the plot of ln q_e *vs* ln C_e.

Results and Discussion

Characterization of activated carbon

Fig. 1 represents the photographs of activated carbon prepared. The SEM micrographs of biochar sample and activated carbon are shown in Fig. 2a and 2b, respectively. It indicates that the surface morphological characteristics of the biochar were altered and the pores were gradually formed during the char formation reactions. This is due to the increased pyrolysis temperature. The structure and porosity of the biochar was influenced by the chemical activation process and the formation of volatile matter results in pore opening [38]. From Fig. 2a, it was observed that the biochar after activation results irregular globular shape with more porous nature and higher surface area than the sample before chemical activation with KOH. Chemical activation process influences the formation of pores and development of the cracks, thus enhances the surface area as visualized from the SEM images in Fig. 2b. The morphology of activated carbon differs from biochar due to its enhanced pore volume and surface area. SEM image exhibited a dissimilar and haphazard nature of pores after chemical activation. The SEM image established a substantial improvement in pore formation on the surface of activated carbon.

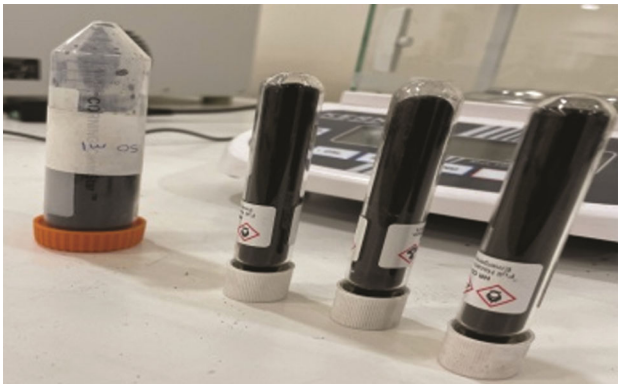

Fig. 1 ─ Photographs of synthesized activated carbon

The FTIR analysis of samples before and after activation process is shown in Figs. 3a and 3b, respectively. The FTIR spectrum shows the appearance of functional groups like – COOH, – OH, C=O, – CHO, C–N, etc. The appearance of multiple peaks explains the presence of different types of material in biochar from different sources. The

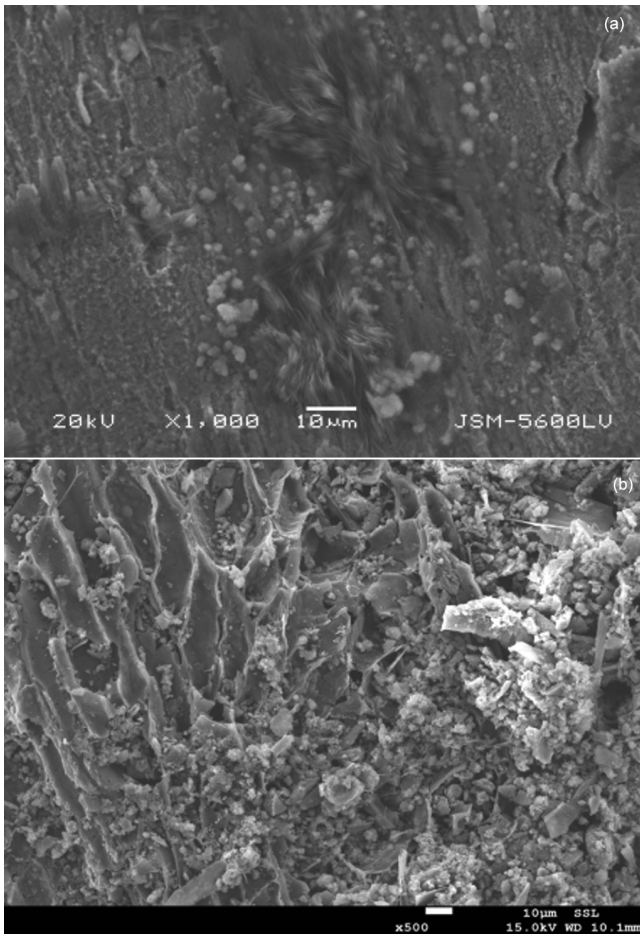

Fig. 2 ─ SEM micrographs of (a) biochar sample and (b) activated carbon

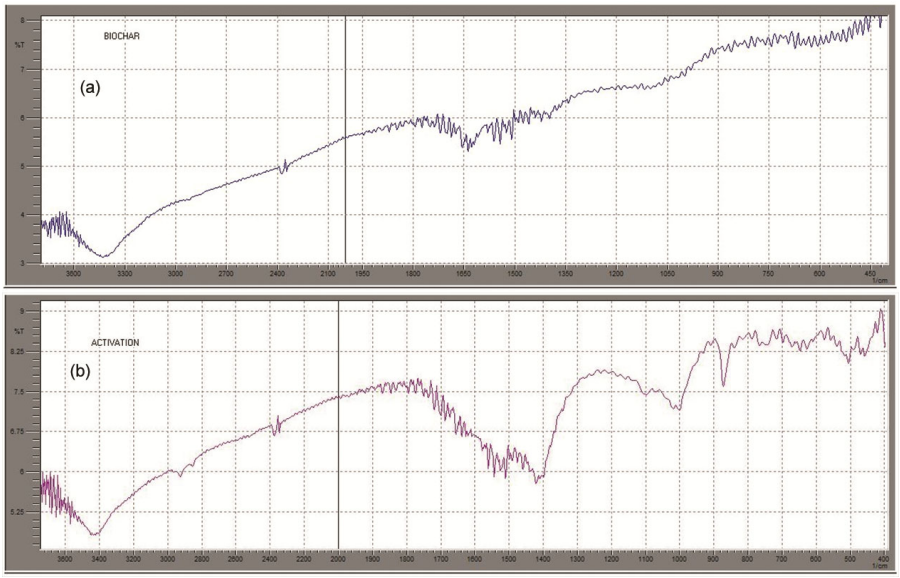

Fig. 3 ─ FTIR spectra of (a) biochar sample and (b) activated carbon

sample after activation with KOH shows a shift in the functional groups as illustrated in Fig. 3b. The changes in surface functional groups are evidenced through the transformation of peaks from one place to another. Further, thermal activation of the sample in the furnace resulted in shifting or disappearance of several peaks as observed in the FTIR spectrum presented in Fig. 3b. The peaks appeared at wavenumbers corresponding to 1650 cm^{-1} and 1865 cm^{-1} are due to the stretching vibrations of C = O and anhydrides with two carbonyl groups. Two bands at 2400 cm^{-1} and 2934 cm^{-1} are ascribed to asymmetric and symmetric stretching of aliphatic bands –CH, – CH_2 and $–CH_3$. The broad band from 3400 cm^{-1} – 3500 cm^{-1} represents the O–H stretching [39]. After chemical activation with KOH, the majority of the peaks are disappeared, particularly in the lower wavenumber ranges. The spectrum shown in Fig 3b indicates the existence of oxygen containing surface functional groups of carbonyl, hydroxyl, and carboxyl groups in the activated carbon sample. These peaks disappeared in the spectra of activated carbon, specifying the successful activation process by KOH treatment.

The X-ray crystal structure of the activated carbon sample after activation duration of 1.5 h is shown in Fig. 4. The diffraction pattern exhibits two prominent diffraction peaks at 28° and 42°, are characteristic of carbon and graphite[40]. A prominent peak at 2θ = 28° indicates the presence of carbon (C_{12} to C_{60}), and the augmentation of carbon content in biochar at higher temperatures[41].

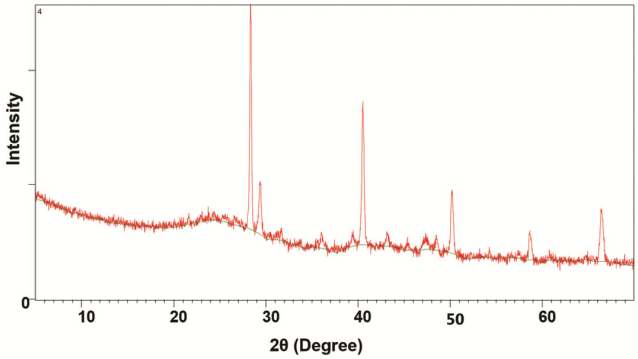

Fig. 4 ─ X-Ray diffraction pattern of activated carbon

Batch treatment of refinery waste water

Influence of solution pH

The influence of change in solution pH on reduction in parameters is carried out by changing the refinery effluent pH from 2.0 to 10.0. For this study, 100 mL of the effluent was mixed with a specified amount of activated carbon under stirring condition. The influence of solution pH with parameter reductions are shown in Fig. 5. It was identified that highest reduction in COD was achieved at a pH 6.0. At this pH, the activated carbon has more number of adsorption sites available for the adsorption of pollutants. A maximum percentage reduction in COD was obtained at pH 6.0 and the optimum COD reduction was 86%. So, pH 6.0 was considered as the optimum pH and the same pH was maintained in the rest of the experiments. Further, it can be noted that at pH 6.0, the highest removal efficiency of TDS, TSS, DO and turbidity was found to be 64%, 78%, 68% and 74%, respectively.

Influence of stirring time

Contact time is a significant parameter contributing the effectiveness of wastewater treatment system. Fig. 6 shows the effect of contact time with parameter reductions. From the experimental studies, the maximum COD reduction was obtained at 80 min, and this was considered as the optimal stirring time for the treatment. The percentage COD removal efficiency was 89% corresponding to a stirring time of 80 min. Additionally, at an optimum stirring time of 80 min, the highest removal efficiency of TDS, TSS, DO and turbidity was found to be 74%, 79%, 87% and 79%, respectively. It can be noted from Fig. 6 that an increase in contact time increases the removal efficiency up to 80 min. The study shows that the parameter reductions increased with enhanced mixing time up to 80 min. The breakdown of pollutant particles may lead to enriched surface area and hence result in increased adsorption rate. Above 80 min of stirring, the pollutant removal efficiency was diminished due to the weakening of the electrostatic force. At increased mixing time (up to 80 min), breakage of the pollutants is enhanced, thus reducing the particle size which results in more interfacial area for the adsorption to happen and encourages the adsorption. At the beginning stages of adsorption process, the collision between the adsorbent surface and pollutants starts, and adsorption sites were available for the adsorbate to get adsorbed on the surface, but at later stage these sites were reduced and repulsive forces may have started between the adsorbate molecules on the solid and bulk phases [42]. Therefore, mixing time is a decisive factor determining the pollutant reduction efficiency in wastewater treatment.

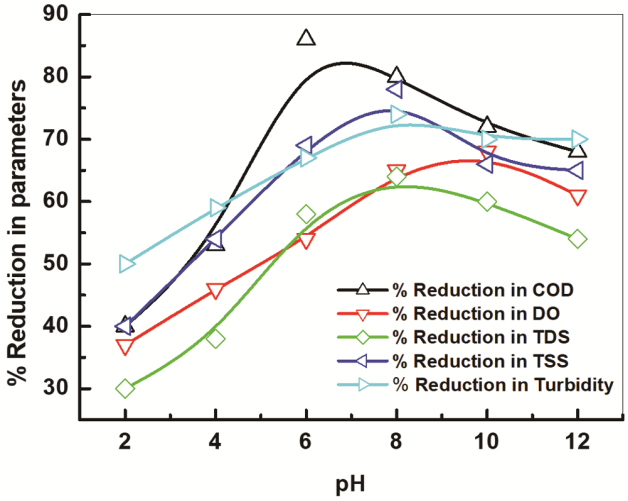

Fig. 5 — Influence of solution pH on reduction of different parameters

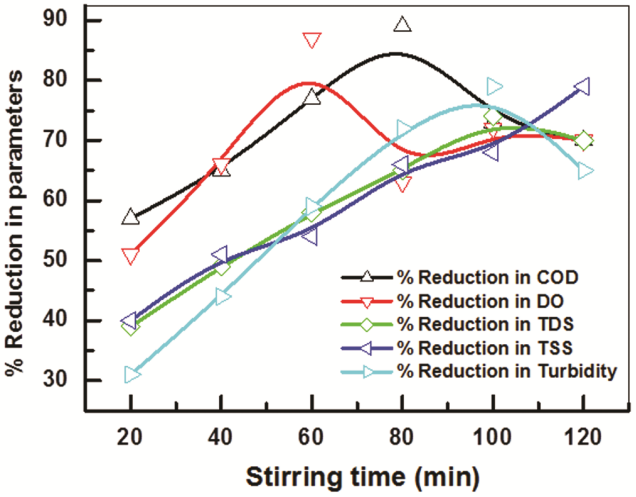

Fig. 6 — Effect of change in stirring time on reduction of different parameters

Influence of dosage

The variation of dosage of activated carbon was studied by altering the amount of activated carbon from 0.1 g to 2.2 g at optimized pH and stirring time. The influence of change in adsorbent dosage with percentage reductions in COD, TDS, TSS, DO and turbidity are illustrated in Fig. 7. The best results were achieved at an optimum dosage of 2.0 g and the adsorption sites are effectively utilized at this dosage with 80% reduction in COD. At this dosage the removal efficiency of TDS, TSS, DO and turbidity are 84%, 89%, 88% and 74%, respectively. An increase in the dosage of adsorbent resulted in enhanced surface area with increased number of functional groups and hence more interaction between pollutants

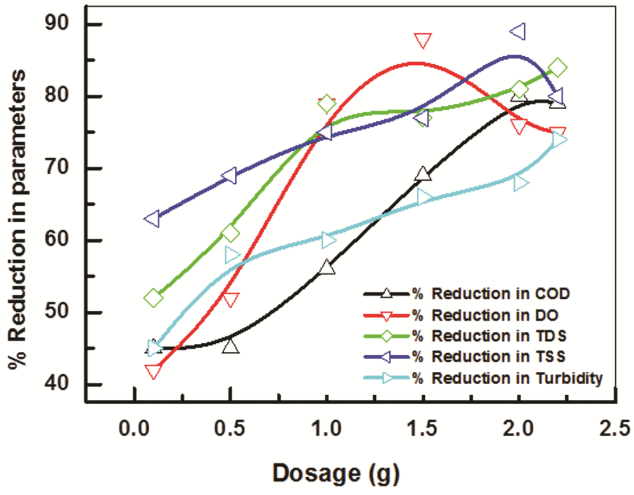

Fig. 7 — Effect of dosage on reduction of different parameters

and the adsorbent surface, leading to increased number of active sites and maximum adsorption capacity. A dosage above 2.0 g would result in overlapping of adsorption sites due to overcrowding of adsorbent particles[43,44]. This may be due to the aggregation of binding sites and hence lessening the surface area of particles available for adsorption process. The excess dosage has the possibility to get a charge reversal, which led to particle re-stabilization and flocs are produced rapidly and form a larger size, which can be easily settled. This could be attributed to a large number of vacant binding sites, which are available for adsorption during the initial stages, but thereafter the occupation of the remaining vacant sites will be difficult due to the repulsive forces between the adsorbent surface and the bulk effluent liquid. Insufficient dosage or over dosage of adsorbent would result in the reduced percentage removal efficiency. Therefore, it was crucial to determine the optimum dosage of adsorbent to minimize the cost and increased effectiveness.

Influence of agitation speed

Stirring speed is a key parameter determines the effectiveness of activated carbon in the treatment of refinery waste water. The effect of variation of stirring speed with parameter reductions are shown in Fig. 8. The COD removal efficiency was 89% corresponding to a stirring speed of 75 rpm, hence the best stirring speed for the reduction in COD was fixed as 75 rpm. The percentage reduction in COD was lowered when stirring speed was increased above 75 rpm. This may be due to the breakdown of the flocs and expanding chains, thereby the adsorbed pollutants are desorbed, which results in decreased pollutant removal efficiency. From the experimental study, the highest reductions in parameters are obtained at 75 rpm and this speed is considered as optimum agitation speed in the treatment. The enhanced stirring rate motivates improved diffusion of impurities on the surface of the adsorbent particles. The percentage reduction in TDS, TSS, DO and turbidity at an optimal agitation speed of 75 rpm are 89%, 72%, 78% and 90%, respectively.

Fig. 9 shows the morphological characteristic of the adsorbent after refinery wastewater treatment. The SEM image shows a clustered and thick structure, which indicates that most of the pollutants present in the refinery wastewater were adsorbed on the surface of activated carbon. The dark and thick deposition on the surface of adsorbent indicates the successful migration of pollutants from the bulk effluent solution to the adsorbent surface and stays on the adsorbent surface. The SEM image in Fig. 9 reveals the successful deposition of tiny particles as seen on the adsorbent surface as thick layer formation. This demonstrates that the adsorption sites of activated carbon were effective in removing the pollutants from the refinery wastewater. The improved adsorption capacity of activated carbon was due to the formation of large number of pores during the activation process.

The FTIR spectrum of the activated carbon after treatment with refinery wastewater clearly shows the appearance of additional functional groups due to the successful deposition of pollutants on the surface of the adsorbent material as shown in Fig. 10. This might be due to their large surface area, high adsorption

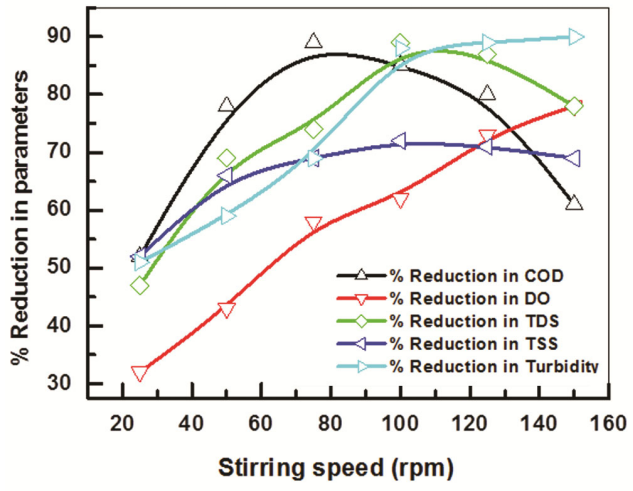

Fig. 8 — Effect of agitation speed on reduction of different parameters

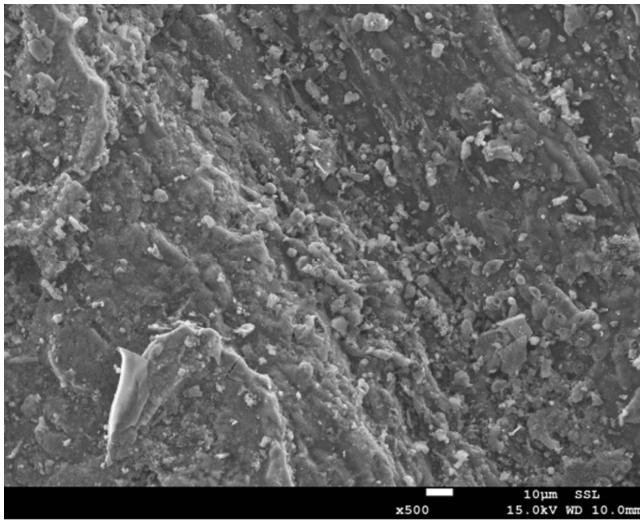

Fig. 9 — SEM micrograph of activated carbon after wastewater treatment

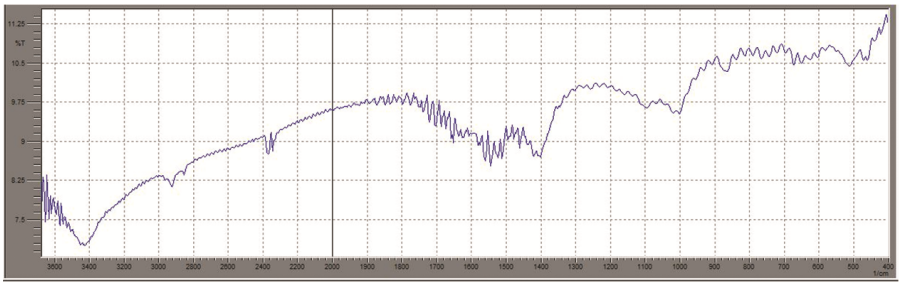

Fig. 10 ─ FTIR spectrum of activated carbon after refinery wastewater treatment

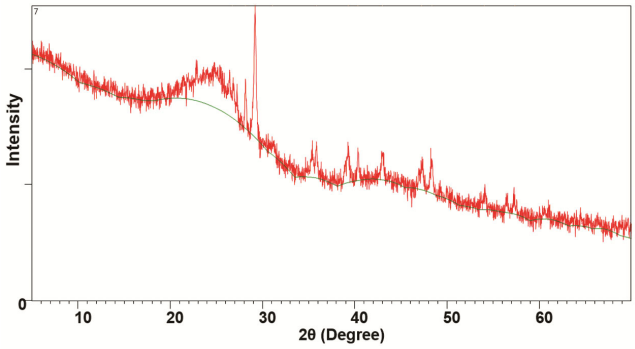

Fig. 11 ─ X-ray diffraction pattern of activated carbon after refinery wastewater treatment

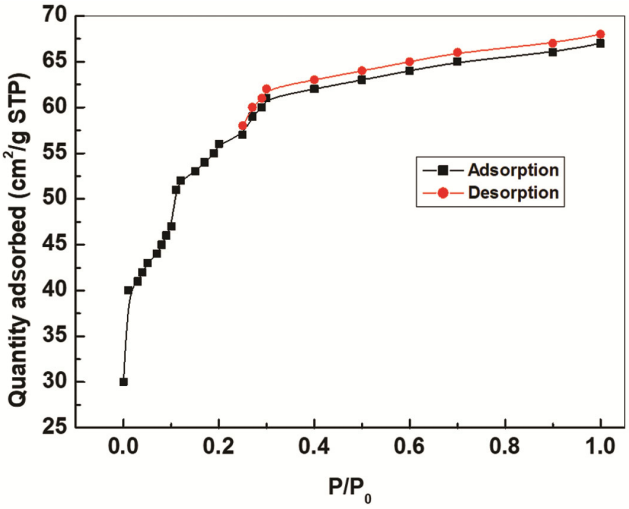

Fig. 12─ BET surface area plot of activated carbon before and after adsorption of wastewater

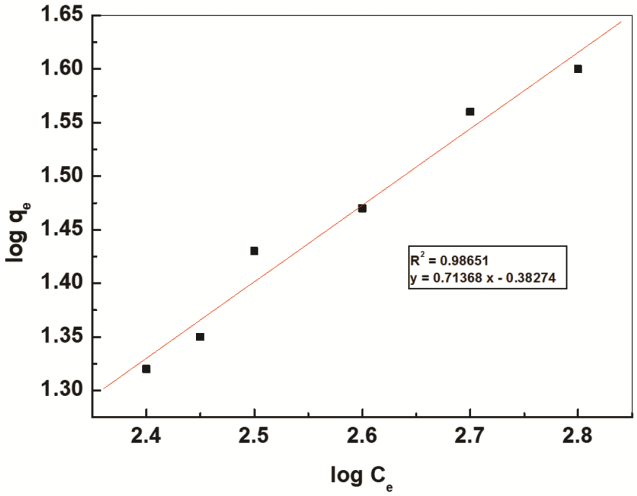

Fig. 13 — Freundlich isotherm model for the adsorption process

capacity, and presence of surface functional groups leading to enhanced adsorption[45]. The FTIR spectrum of the activated carbon after wastewater treatment revealed the appearance of several bands exhibiting functional groups of O = C = O carbon dioxide, carboxylic acid and aromatic compounds. The FTIR spectrum of material can provide valuable information about its chemical composition. During the process of adsorption, charge transfer occurs between the adsorbate and the adsorbent. This results in a dipole moment. Hence, there is the adhesion of ions, atoms and molecules from liquid, gas and dissolved solids to a particular surface. As a result, an adsorbate film is formed on the adsorbent surface.

The diffraction patterns of the adsorbent after waste water treatment are shown in Fig. 11. After adsorption process, there was a shift in the representative diffraction peaks and also appearance of new peaks, which indicates the deposition of pollutants present in the waste water to the surface of activated carbon.

The pore volume and active surface area of the adsorbent after wastewater treatment was scrutinized by Brunauer Emmett and Teller (BET) analysis, as shown in Fig. 12. The BET analysis displayed a surface area of 125.1 m^2/g and a pore volume of 0.561 cm^3/g. The Freundlich isotherm adsorption isotherm study was carried out to establish the equilibrium adsorption. The Freundlich adsorption isotherm model fits well showing the best correlation coefficient (R^2) value at 0.9955 and relates the experimental data. The adsorption isotherm shown in Fig. 13 signifies the Freundlich adsorption isotherm,

which is best fitted model with a high correlation coefficient, R^2 value of 0.9955 and also correlates the experimental data. The constants in the Freundlich isotherm was found to be k = 0.688, and n = 1.40.

Conclusion

In this research, the potential of activated carbon produced from municipal solid waste for wastewater treatment application was assessed. The activated carbon was employed in the batch treatment of refinery wastewater by altering the parameters of wastewater pH, stirring time, stirring speed and dosage of activated carbon. The experimental outcome demonstrates that the municipal solid waste is a cost effective alternative material of commercial carbon. The activated carbon exhibited enhanced adsorption capacity by reducing the pollutant concentration in refinery waste water. The effectiveness of pollutant removal was endorsed by SEM and the optimal adsorption capacity was achieved at pH 6.0 due to the shift in surface functional groups (e.g. –C=O, –C=N), as illustrated by FTIR analysis. The optimum treatment conditions were obtained at a solution pH of 6.0, 80 min stirring time, 75 rpm stirring speed with 2.0 g of activated carbon. The results of the batch study validate that the Freuendlich adsorption isotherm model as the best fit. The surface area and pore volume of the particles obtained from the adsorptions study using BET analysis are 125.1 m^2/g and a pore volume of 0.561 cm^3/g. The results validate that municipal solid waste can be converted into a value added activated carbon with high adsorption capacity in refinery wastewater treatment applications.

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