

Preparation of Activated Carbon from Green Coconut Shell and its Characterization

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Abstract

In this work activated carbon was prepared from green coconut shells by chemical activation method. It was then characterized by XRD, SEM, FT-IR and TGA. Different physical properties such as bulk density, moisture content, volatile matter content, ash content, and surface area and porosity were also determined. The activated carbon was used as adsorbent for the removal of carbon dioxide from flue gas as well as various inorganic contaminants from waste water. Removal efficiency of Methylene blue was studied by changing various parameters such as adsorbent dosage and contact time. This study showed that for the removal of various pollutants from flue gas and waste water treatment activated carbon can be used as a good adsorbent.

Keywords: Activated carbon; Adsorption; Methylene blue; XRD; FTIR; TGA

Introduction

Adsorption has appeared as one of the promising methods for CO₂ capture and sequestration due to its low energy consumption, cost-effectiveness, relatively simple technological process, non-corrosive to the equipments and it is applicable over a relatively wide range of temperatures and pressures [1-3]. Development of new and high-efficient solid adsorbents is crucial to enhance competitiveness of this process. Zeolites or activated carbons are good adsorbents that are used for capturing CO₂ from flue gas through physical adsorption [4], due to its porous structure. The extensive use of activated carbon is used now days, due to its large micro porosity, large specific surface area [5]. The activated carbon is seems to be black in color and have large micro porosity. Activated carbon adsorption also used for treating low concentrations of wastewater streams at extremely low cost. The removal efficiency of Activated carbon is very high for harmful pollutants [3]. As environmental pollution is the major problem now a day's so need of activated carbon is growing day by day. Its texture characteristics and surface properties depend on the raw material and on the method used for its preparation [6]. For AC, the removal of moisture is not required and it is easy for regeneration and has a high CO₂ adsorption capacity at ambient pressure [7] and also its good adsorption properties. Activated carbons can be prepared by either a physical method or chemical method [8]. It is produced from a large number of carbonaceous raw materials like coal, lignite, wood and some agricultural product like rice husk [9,10], nut shell [11], coconut shell [12,13], pea nut [14,15], sugarcane bagassage [16], tamarind wood [17], saw dust [18] and industrial waste products. The cost of Agricultural wastes are very low so it is considered to be a very important feedstock for preparation of AC [19]. To prepare activated carbon from chemical activation, the steps are carbonization step and activation step. Raw material is impregnated with an activating reagent before carbonization step in chemical activation method. The effect of different chemical reagents on the production and quality of activated carbon was studied extensively by different researchers [20,21]. Different chemicals are used like Zinc chloride (ZnCl₂), phosphoric acid (H₃PO₄), Sulphuric acid (H₂SO₄), potassium hydroxide (KOH), sodium hydroxide (NaOH), and K₂CO₃[22-24]. Chemical activation is better than physical activation process because it generally takes place at a lower temperature and shorter time and the development

of a porous structure is better than that of physical activation. For preparation of AC, Zinc chloride is the most widely used chemical studied by various authors [6,11,25-32]. In this study green coconut shell has been chosen as raw material for preparation of AC because it is considered superior to those obtained from other sources mainly due to small macrospores structure which renders it more effective for the adsorption of gas/vapour and for the removal of colour and odour of compounds. And it has high fixed carbon and low ash content. The adsorption behaviour of activated carbon is determined not only by their porous structures but also by the chemical nature of its surface. Adsorption capacity is determined by porosity, and the functional group present on the surface, is getting affected by polar and non-polar structure of the adsorbate [33]. In adsorption process surface chemistry and porosity plays an important role, so activated carbons are investigated by different characterization method. The objective of this study was to prepare relatively well developed porosity activated carbons from green coconut shell by chemical activation methods. The adsorbents were characterized by using Scanning electronic microscope (SEM), Fourier transfer infrared spectroscopy (FTIR), X-Ray diffraction (XRD), Accelerated surface area and porosimetry analyzer (ASAP2020). Also its performance for liquid adsorption capacity was tested by Methylene blue adsorption by using UV Spectro photometer.

Materials and Methods

Preparation of activated carbon

Green Coconut Shells were collected from nearby Technology market of IIT Kharagpur Campus and then cut into small pieces,

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followed by washing with simple tap water for removal of dust adhere to it. Then it was dried in the sunlight for 15-20 days. Dried materials were kept inside the furnace at 150°C for 24 hours for removal of moisture and other volatile impurities. After that it was crushed with a locally made crusher and sieved to 300-700 µm size range. Chemical Activation of the powdered precursor was done with ZnCl₂ to make the Impregnation Ratio (Activating agent/Precursor) 100% (500 gm of dried precursor was well mixed with 3000 ml of concentrated solution of ZnCl₂ that contains 500 gm of ZnCl₂). The slurry form of powder precursor was properly mixed and kept for 24 hours for proper soaking of ZnCl₂ on its surface. The slurry was kept inside the oven at 100°C for 24 hours [34]. In this work we have used ZnCl₂ because it acts as dehydration reagent that lowers the carbonization temperature during chemical activation and restricts the formation of tar as well as promotes charring of carbon [25,35]

The resulting chemical impregnated samples were kept inside the muffle furnace after putting inside galvanized iron pipe of dimensions (ID 3.7 cm, OD 4.4 cm, Length 24 cm). The material inside the furnace was heated at a heating rate of 10°C min⁻¹ to the final carbonization temperature of 650°C under the Nitrogen flow rate of 120 cm³ min⁻¹ STP. The material was kept inside the furnace for 1 hr at 650°C. Then it was cooled under the constant flowing of Nitrogen Gas till it was completely cooled. The dried material was washed with 0.5 N HCl for 2-3 times and then washed with warm distilled water to remove different residual organic and mineral matter. Then it was finally washed with cold water till the solution becomes neutral. Finally the sample was dried for 24 hours at 100°C inside an oven and packed in an air tight container [36].

Characterization of activated carbon

Physical characterization: Bulk density of the prepared activated carbon was done by water displacement method and was found to be 2.1 gm/cc. Textural characteristics were determined by nitrogen adsorption at 77.35 K with an automatic adsorption instruments (ASAP 2010, Micromeritics) in this case the samples are out gassed at 300°C under the flow nitrogen for 6.5 hours before measurement. The surface area of the activated carbon samples were calculated by Brunauer-Emmet-Teller (BET) method in the relative pressure (P/P^o) range of 0.05-0.30. The adsorption isotherms were analysed and BET method [37] for determination of surface area. The pore size distribution was determined by using Barrett-joyner-Halenda (BJH) model [38] and t-plot for determining volume of the micropore [39]. The cross sectional area of nitrogen molecule is assumed to be 0.162 nm. The micropore volume corresponds to volume adsorbed in the pore less than 2 nm. Mesopore corresponds to volume adsorbed in pores between 2 and 50 nm. The mesopore surface area was calculated by subtraction of BET surface area and micropore surface area [25]. The Dubinin-Radushkevich (DR) method is used to calculate the micropore volume. The micropore size distribution calculated by Density functional theory (DFT). The total volume was calculated by converting the amount of Nitrogen gas adsorbed at a relative pressure of 0.95 to the equivalent liquid volume of the adsorbate. The mesopore volume is calculated by subtracting the micropore volume to the total volume. Scanning electron microscope (ZEISS EVO 60) was used to visualize the morphology of the raw material (green coconut shell), activated carbon.

Chemical characterization: Elemental analysis was carried out using CHNS/O analyser. Proximate analysis was carried out to find out percentage moisture, volatile matter, ash and fixed carbon content. An X-Ray diffract meter (XRD PW 1710) was used to investigate the

surface inorganic component of the prepared activated carbon. The X-ray patterns were recorded under the scan rate of 0.1°C/min. The surface organic functional groups and structure were studied by FTIR spectroscopy (NEXUS 870). The FTIR spectra of activated carbon and amine impregnated activated carbon were recorded between 400 and 4000 cm⁻¹. In the FT-IR analysis Solid powder sample was first mixed with solid KBr in mortar pestle and a thin pellet is made using a hydraulic press. Such pellet can be placed in the IR beam for analysis.

Results and Discussion

Proximate and ultimate analysis

The results of proximate and ultimate analysis were shown in Tables 1 and 2. From this analysis it was observed that the fixed carbon content of AC was very high as compared to raw precursor, which results in better adsorbent for adsorption purpose. From the analyses it has been shown that the high volatile matter and low ash content of the precursor make it a good starting material for preparation of activated carbon [25]. The carbon content of AC increases with the increasing of activation temperature from 500 to 600°C due to an increasing degree of aromaticity [36]. In activated carbon hydrogen, nitrogen, sulphur and oxygen contents have been decreased because during pyrolysis and activation process, the coconut shell has been decomposed. During the decomposition, the volatile compounds containing mainly H, O, and N leave the carbonaceous product and the coconut shell becomes rich in carbon. Because ZnCl₂ removed the H and O away from the green coconut shell as H₂O and H₂ instead of removal of CO, CO₂ or hydrocarbons [25,40]. From Table 2, it was observed that, the carbon content of activated carbons has been risen to from 41.185% to 72.04 wt%; however, a decrease in the hydrogen and oxygen content from 9.416 to 2.963 wt%, and 10.024 to 0.046 wt% respectively.

BET surface area and pore size distribution

Table 3 Shows the data related to total surface area, micropore volume and micropore surface area for activated carbon particle and raw precursor. Figure 1 gives the N₂ adsorption isotherms at -196°C of the activated carbon and raw precursor produced at the activation temperature of 650°C. Micropore plays an important role for adsorption. Nitrogen adsorption was more in the case of activated carbon because of the presence of excess pores that developed during activation and carbonization that of raw precursor. In the case of activated carbon the small pore is called micropore and the external surface areas consist of the mesopores and macropores [41]. Thus from

Sample	Moisture (wt. %)	Volatile matter (wt. %)	Fixed Carbon (wt. %)	Ash (wt. %)
Raw Precursor	11.834	44.969	40.633	2.564
Activated carbon	2.983	17.208	78.907	0.902

Table 1: Proximate analysis of raw precursor and Activated carbon.

Sample	C (%)	H (%)	N (%)	S (%)	O (%)
Raw Precursor	41.185	9.416	5.99	33.385	10.024
Activated carbon	72.04	2.963	1.16	23.791	0.046

Table 2: Ultimate analysis of raw precursor and Activated carbon.

Sample	BET SSA (m ² /g)	V _{Tot} (cm ³ /g)	V _{Micro} (cm ³ /g)	A _{micro} (m ² /g)	Avg. Pore radius (Å ^o)
Raw Precursor	59.728	0.05321	0	0	16.8461
Activated carbon	995.799	0.4487	0.372	921.71	9.01198

Table 3: Pore structure parameter for raw precursor and Activated carbon sample.



Figure 1: Pictorial view of raw green coconut shell.

table we can infer presence of micro pore is nil in raw green coconut shell hence the least Nitrogen adsorption. The shape of the adsorption isotherm can provide preliminary qualitative information on the adsorption mechanism as well on the pore structure of the carbon. According to IUPAC classification, these isotherms are of Type I, which represents dense micropore structures [25]. The surface area and pore volume of activated carbon prepared under optimum conditions were 995.799 m²/g and 0.372 cm³/g, respectively. Average pore radius was 9.011 Å. It found to be very high as compared to raw precursor, which shows better adsorbent characteristics. Due to the larger the specific surface area of the adsorbent, the better its adsorption performance as obtained literature [42].

Thermo gravimetric analysis

The two graphs in the Figures 2-5 is the plot for original weight verses temperature. We observe weight loss for activated carbon was 28.75% compared to 47.15% for raw precursor when temperature was raised from room temperature 27°C to very high temperature 800°C. So activated carbon has high thermal stability and thus preferred over raw precursor as suitable adsorbent. In minutely observing, both graphs showed similar trends. Two graphs can be divided into three stages. In 1st stage, decreasing slope was maximum which accounts to removal of volatile matter, moisture and other impurities. In this stage the organic matter decomposes releases the gaseous volatiles [43]. For activated carbon the temperature range was from 27°C-110°C to that of raw precursor which was from 27°C-65°C. In 2nd stage the decreasing slope was reduced a bit which accounts to slow decomposition of both raw precursor (65°C-300°C) and activated carbon (110°C-660°C) and the intermediates further decompose to form other volatile species, tar and char also the chemical activating agent ZnCl₂ begins to melt or decompose [43,44]. In 3rd stage with further rise in temperature the downward slope again increases rapidly for both cases that account to severe erosion of material and the char reacts with ZnCl₂, causing the pores to open [43,44]. By analyzing both graphs we observed the activated carbon has very high resistant to weight loss compared to raw precursor for the same rise in temperature change from room temperature to 800°C. Thus the activated carbon can be subjected till 650°C without much significant weight loss of adsorbent.

Fourier transform-infrared analysis

FTIR is a well-known method for analyzing surface chemistry. It can be used to detect the functional groups present in the sample, The atoms in molecules are not static, but vibrate about their equilibrium positions, even in the solid state. Each atom vibrates with a frequency which depends on its mass and the length and strength of any bonds

it has formed. Molecular vibrations are stimulated by bonds absorbing radiation of the same frequency as the natural frequency of vibration of the bond (i.e., in the range 1.20×10^{13} - 1.20×10^{14} Hz) which is in the infrared region of the electromagnetic spectrum. Surface functional group containing oxygen have a major role in AC properties such as surface behaviour and reaction, hydrophobicity, surface charges, electron density of graphene layer and may be further used for surface modification. Different functional group present in activated carbon sample were carboxylic, carbonyl, phenol and lactones etc. The



Figure 2: Pictorial view prepared Activated carbon.

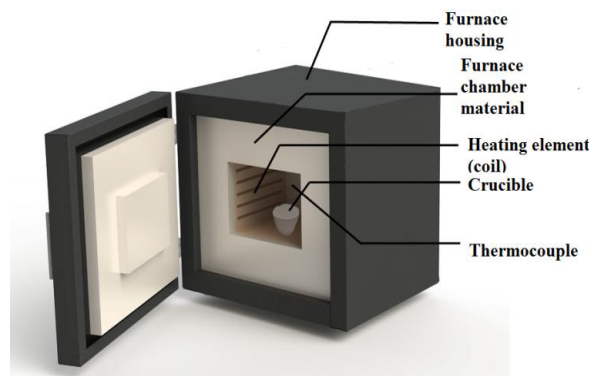


Figure 3: Pictorial view of Muffle furnace.

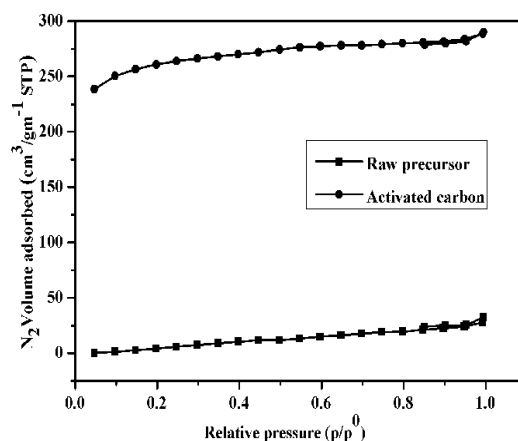
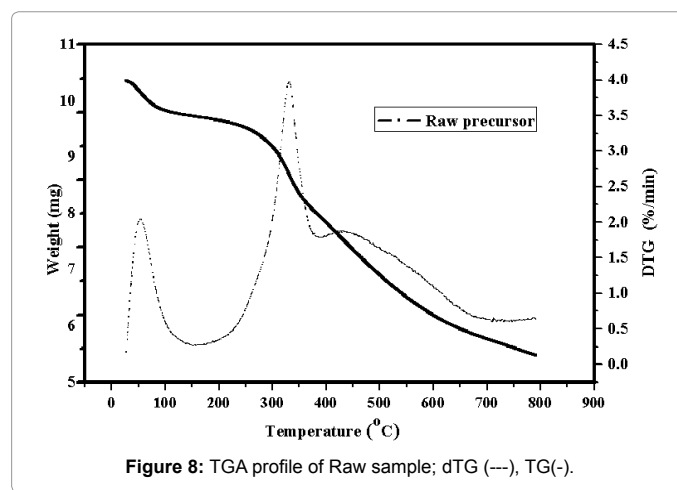
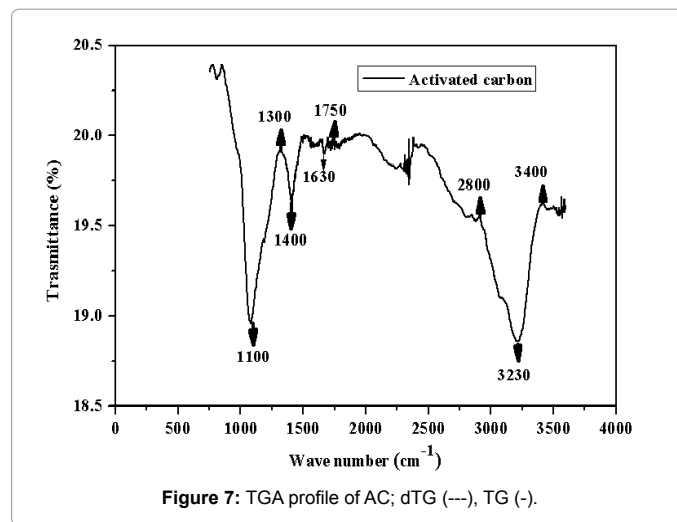
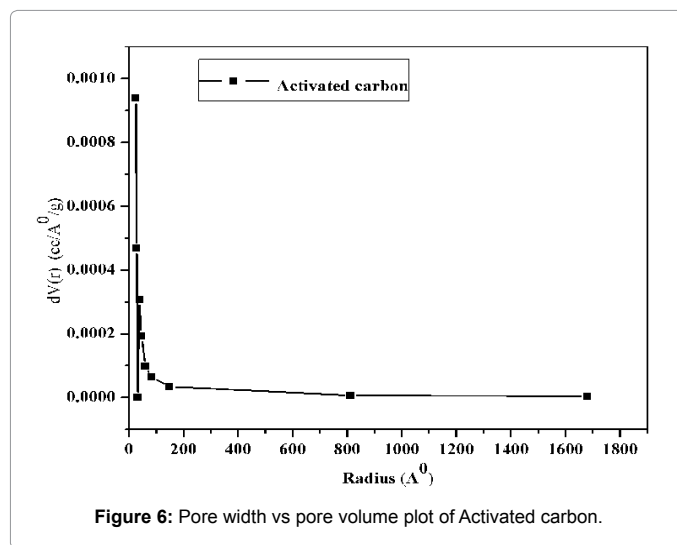
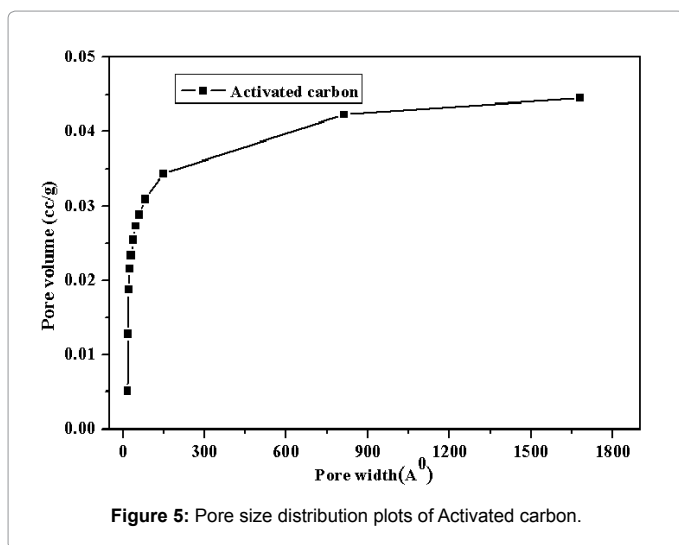


Figure 4: Nitrogen adsorption isotherm of Activated carbon.

functional group present in the activated sample were as shown in the table. The range 3500-3200 cm^{-1} indicates the presence of O-H stretch bonded, alcohol, phenol functional group. 3300-2500 cm^{-1} shows the presence of O-H stretch and carboxylic group. At 1320-1000 cm^{-1} (strong) frequency shows C-O stretch and functional group presents are alcohol, carboxylic acid, esters and ethers [45]. A band at 1400 cm^{-1} was only can be attributed to the plane N-H bending vibration [46,47]. A band at 1100 cm^{-1} is attributed to C-O stretching vibration in hydroxyl and phenol groups. Combination of bands at 1750 and 1100 cm^{-1} suggested that it is predominantly carboxylic groups. The frequency range 1470-1450 cm^{-1} shows the presence of alkanes (C-H bond). Other group's presences were shown in the Table 4.

Scanning electron microscope studies

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. SEM micrograph of activated carbon produced at 650°C for 1 hr was depicted in Figures 6-10. Different pores were observed in activated carbon sample. Due to carbonization and activation, volatiles are removed producing a fixed carbon mass with widening of pore networks that are present in activated carbon sample. The micropore presences contribute towards gas adsorption [48].

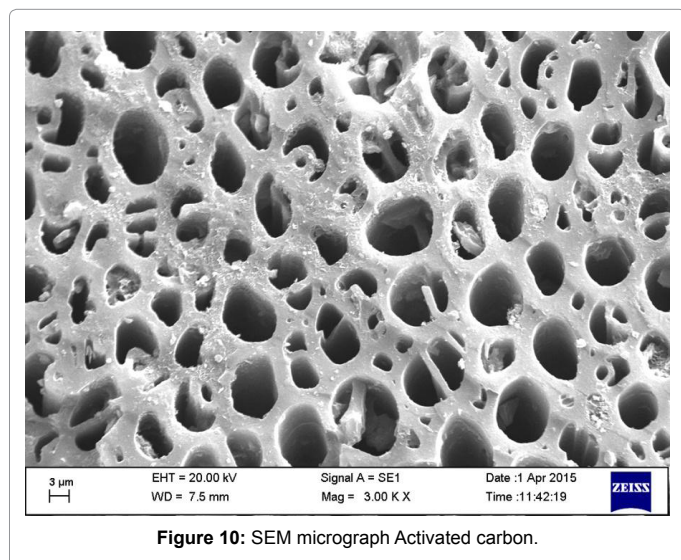
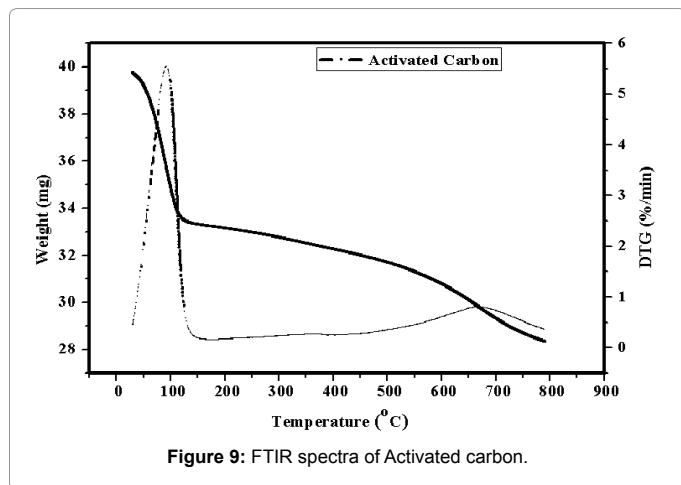


Wave number (cm^{-1})	Bond	Functional Groups
1100	C-H wag (CH_2X)	Alkyl halides
1300-1400	C-O stretch	Alcohols, ethers, ester, carboxylic acid
1630	N-H bend	1° amine
1750	C=O	Esters, carboxylic acids, aldehydes, carbonyl
2800	H-C=O	aldehydes
3230	\equiv C-H stretch	Alkyne
3400	O-H stretch, H bonded	Alcohols, phenols

Table 4: FT-IR peaks assignments of functional group on Activated carbon surface.

X-Ray diffraction

From the above graph we observed that for raw green coconut shell we obtain three sharp peaks at angle 25°, 40°, 50° respectively that revealed crystalline carbonaceous structure. The sharp peak is produced



due to better layer alignment which is the characteristic of a crystalline structure. Absence of sharp peaks in activated carbon suggests it is predominantly amorphous structure which is advantageous property for well-defined adsorbents [49,50].

Methylene blue adsorption

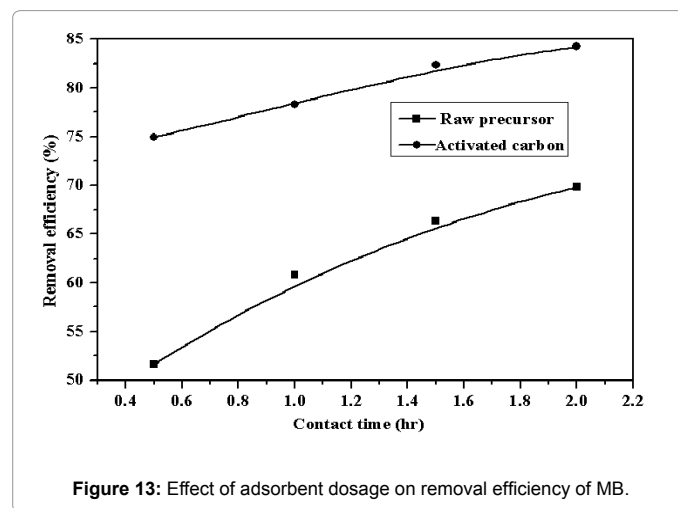
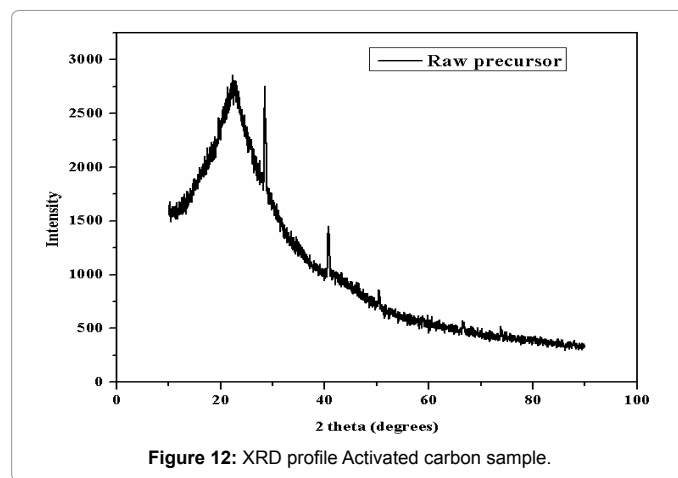
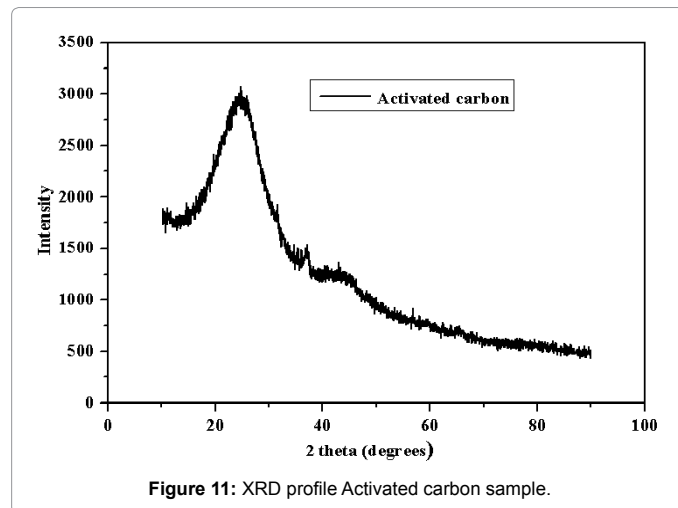
Due to its strong adsorption onto solids, Methylene blue dye serves as a model compound for adsorption of organic contaminant. The methylene blue dye solution was prepared by 2 drops of MB in 100 ml of distilled water and was taken in conical flask. For batch adsorption studies, One gram of activated carbon was added to a series of conical flasks filled with MB solution. It was sealed properly and then shaken in a thermal shaker at 30°C until equilibrium was reached. The sample solution was then removed and analyzed with a UV-Visible spectrophotometer at a wavelength of 660 nm [51]. A previously established linear Beer-Lambert relationship was used for the concentration determination. The Removal efficiency was determined by eq.1 as shown below.

The efficiency of dye removal was calculated as

$$\text{Efficiency (\%)} = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

Where C_i = Initial concentration of MB (mg/L), C_e = Final concentration of MB (mg/L)

Similarly the removal efficiency was calculated by taking constant concentration of Methylene blue with different quantity of adsorbent dosage. It was found from the Figures 11-14 that the percentage removal of Methylene blue was increased by increasing adsorbent



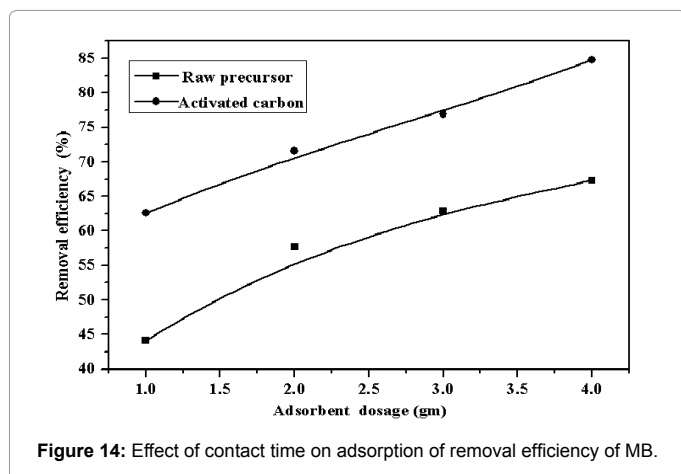


Figure 14: Effect of contact time on adsorption of removal efficiency of MB.

(activated carbon) dosage. Because with increase in adsorbent dosage the numbers of available adsorption sites were increased [52]. Thus percentage removal efficiency is more than that of raw precursor. Similarly with increase in time duration the percentage removal efficiency of Methylene blue increases [53]. After sufficient time (9-10 hrs) and under equilibrium condition the removal efficiency of Methylene blue shows >90%. So activated carbon shows good adsorbent characteristics as compared to raw precursor.

Conclusion

Green coconut shell based activated carbon was successfully prepared by chemical activation method. The results indicated that the adsorption capacity depends upon the pore size, surface area micropore volume of the activated carbon. Thermal stability was also found to be very good by thermo gravimetric analysis method. Removal efficiency of Methylene blue for activated carbon was >90% after sufficient time (9-10 hrs) under equilibrium condition. So the activated carbon can act as a good adsorbent for the adsorption of carbon dioxide from flue gas as well as waste water treatment.

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