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Research article

Production and Evaluation of Activated Carbon from Saudi Arabian Acacia Tortilis Tree Bark by Microwave and Low TemperatureActivation Process

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Keywords: Waste material, Acacia Tortilis bark, Activated carbon, Chemical activation, Microwave irradiation, Carbonization

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In the present work, activated carbon was prepared from *Acacia Tortilis* tree bark, utilizing phosphoric acid chemical activation, and microwave irradiation (AC-MWI). Activated carbon was also prepared by conventional phosphoric acid chemical activation and low temperature carbonization (AC-CA). Characterization of the activated carbons were performed by proximate analysis adopting ASTM standard procedures. BET-specific surface area, total ash content, bulk density, moisture content, pH, pore volume and iodine number were determined. Comparison of characteristics for both carbons, with the characteristics of AC available in the literature and commercially available in the market was performed. Comparison showed that activated carbon produced from *Acacia Tortilis* tree bark is well comparable with the reported characteristics of AC in literature and commercially available in market. It was found that AC-MWI has higher BET-specific surface area than AC-CA (836 m2/g and 687 m2/g respectively). Results depict that there is a n increase of 22.3% in microspores component and 21.7% BET- specific surface area obtained in AC-MWI as compared to AC-CA. Similarly, the corresponding pore volume obtained (4.97 cm3/g and 4.07 cm3/g respectively) demonstrating better values as compared to the commercial ACs (<1 cm3/g). Additionally, it was found that AC can be produced by mi crowave irradiation in about 48.5% less time when compared to conventional low temper ature heating. The adsorption study of produced ACs was performed using methylene blu e as a contaminant. Study showed that MB removal rate initially increased with increase in contact time, which decreased with time until steady state was reached. Adsorption da ta of MB was fitted to Freundlich, Langmuir and Temkin adsorption isotherm models. All models show reasonable correlation, however, Freundlich isotherm is best to describe the MB adsorption on AC-MWI based on correlation coefficient R2 value (0.9985). The results revealed the feasibility of microwave heating for preparation of high surface area activated carbons from *Acacia Tortilis* tree bark.

1. INTRODUCTION

Activated Carbon (AC), is a form of carbon that has been processed to make it extremely porous, thus having a very large surface area available for adsorption or separation of components. Due to its high degree of porosity, one gram of AC may has a surface area in excess of 1000 m^2/g.[1] Activated carbon can be produced from different raw carbon sources such as lignite, peat, coal, and biomass sources such as wood, sawdust, bagasse, tree pruning, and tree bark.[2] Acacias, in general, are the most commonly found tree species in Saudi Arabia and in the Arabian Peninsula. Acacia; generally known as thorn tree, whistling thorn, or wattle which; is a genus of shrubs and trees belonging to the sub-family *Mimosoideae* of the family *Fabaceae—Leguminosae*.[3] Among other Acacia tree species *Acacia Tortilis* is an abundantly found Acacia tree species in Saudi Arabia.[4]

Activated carbon is a well known adsorbent used in water, wastewater and other effluents espacially in industrial applications.[5] As activated carbon is an expensive adsorbent Saudi Arabia invests huge amount of foreign exchange to fulfill the requirements of the country. Activated carbon can be produced from various carbonaceous materials such as wood, rise husk, and other biomass.[6-8] In the past, researchers tried to produce low cost activated carbon from waste precursor materials to reduce the cost of production.[9] In addition to that various production processess and methods were applied to achive better and economical product.[10]

In the presnt study *Acacia Tortilis* tree bark was selected as a raw material as it could be financially more viable

as compared to other materials which require manufacturers extra amount of money for procurement. Furthermore, studies in the past shows promissing results in producing activated carbon from various Acacia species.[11-14] However, there is limited information available about Acacia tree species utilized as a precursor material for the AC preparation. During preparation of AC, precursor materials are generally carbonized by heating in a furnace to obtain extra-porous material and chemical agents are used as catalysts to activate the carbonaceous material.[11] Use of phosphoric acid gained attention of researchers because it has various economic and environmental benefits such as ease of recovery, low energy cost, and high char yield over other activation processes.[15] Microwave heating technology is extensively used in material science, food processing and analytical chemistry. In the recent past, application of microwave heating in AC production shows promising results as compared to conventional activation.[16] However, its application is still in investigation phase.[17]

Due to the promising results obtained in the past studies about production of AC and the unavailability of sufficient information about utilizing *Acacia Tortilis* tree as a precursor material motivated them to carry out research to evaluate the possibility of utilizing microwave irradiation to produce AC from *Acacia Tortilis* tree bark.[18,19] Therefore, in the present study AC was produced with locally available waste material Acacia Tortilis bark using chemical activation with phosphoric acid (H_3PO_4) followed by carbonization via two methods first by low temperature thermal heating, and secondly using MWI on separate chemically activated carbon. Comparison between produced ACs was made. BET-specific surface area, total ash content, bulk density, moisture content, pH, pore volume, and iodine number for both ACs were determined. Prepared AC's were characterized and characteristics were compared with the commercially available AC in market and reported in literature. Finally, adsorption study of produced ACs performed using MB as a contaminant and data also fitted to famous adsorption models.

2. MATERIALS AND METHODS

The following section describes the materials and methods used in the preparation and characterization of activated carbon by conventional heating and by microwave irradiation technique.

2.1. PREPARATION OF ACTIVATED CARBONS

Precursor material Acacia Tortilis bark was prepared as described by Saleem et al.[20] In the chemical activation step, one kilogram of the prepared precursor material was mixed with 40% V/V phosphoric acid having actual concentration of 85.5 wt%.[20] The mixture was kept in the oven at equlibrium temprature of 110 ± 5 °C for 24 hours based on the studies conducted in the past.[12,21] After 24 hours, mixture was poured into a stainless steel hollow cylinder having 8 cm diameter and 15 cm length and having two narrow exhaust holes of 5 mm diameter and two removable end caps

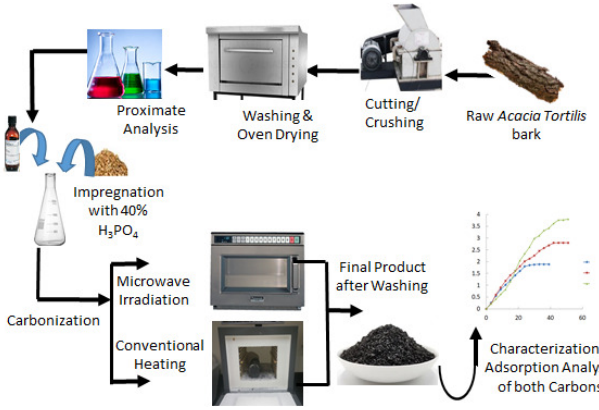

Fig. 1. Schametic diagram for the production of Activated Carbon from conventional and microwave irradiation process.

also provided at both ends of cylinder. After preparation, cylinders were placed inside a muffle furnace (TMAX-1700, Muffle, China) and were heated at a slow rate, allowing free evolution of volatiles up to a held temperature of 600°C. Soaked material was kept at final temperature for 2 hours. The second sample was kept in microwave oven (Panasonic, model NN-ST34NB) with input power of 900 W for 5 to 15 minutes, in several runs.

The cooled material was then washed with hot water to neutralize the samples then dried at 120 ± 5 °C for 2 hours. The dried sample were used for characterization and further study. Complete schamatic flow diagram of the study is presented in figure 1.

2.2. CHARACTERIZATION OF ACTIVATED CARBONS

Proximate and detailed analysis of produced AC samples were also performed. Results were obtained for bulk density, pore volume, pH, BET-specific surface area by utilizing BET Specific Surface Area Analyser (Macsorb, HM-Model 120, Japan). Iodine number, Moisture and Ash content were also measured. The produced ACs were characterized by adopting the standard ASTM procedures described elsewhere.[11,20]

2.3. REMOVAL AND ADSORPTION PROCESS EFFICIENCY

In order to evaluate the removal efficiency of produced AC batch study was performed. Methylene blue was used as an adsorbate in three sets (Commercial Filtrasorb® AC and both AC-MWI and AC-CA) of six 250-mL Erlenmeyer flasks. Each bottle contained 200 mg of AC in 200 mL of MB solutions with various initial concentration ranges from 10 mg/L to 50 mg/L. Set of flasks were agitated in an Orbital shaker OS-1400 at 120 rpm and 30°C. Each set was left to equilibrate for a period of 48 hours. At the end of equilibration period, samples were withdrawn from each bottle, filtered through 0.45 µm filter (Millipore), and analyzed for the MB residual concentrations using the UV-spectrophotometer at a wavelength of 664 nm. Methylene blue standard solutions between 10 to 50 mg/l were prepared and

used to obtain calibration curve. In the equilibrium time study 30 mg/l MB standard solution was used. The amount of MB adsorbed by the adsorbent and process efficiency was calculated with the following formula:

$$\text{Removal Efficiency } (\%) = [(C_o - C_e)/C_o] \times 100 \quad (1)$$

$$q_e(\text{mg/g}) = [(C_o - C_e)/M] \times V \quad (2)$$

Where, C_e is the equilibrium concentration of the adsorbate in the solution (mg/L), C_o is the initial concentration of the adsorbate in the solution (mg/L), q_e is the amount adsorbed (mg/g) on the surface of adsorbent, *V* is a volume of adsorbate used in the adsorption process, and M is the mass of the adsorbent.

2.4. ADSORPTION ISOTHERM STUDIES

Equilibrium data was fitted to three famous isotherm models Langmuir, Freundlich and Temkin isotherm. Expressions for these models are represented in equation 3, 4 and 5 respectively:

$$(C_e/q_e) = (1/q_m)C_e + 1/(K_a q_m) \quad (3)$$

$$\log(q_e) = \log(K_F) + (1/n)\log(C_e) \quad (4)$$

$$q_e = B_1 \ln K_t + B_1 \ln C_e \quad (5)$$

and,

$$B_1 = (RT)/b$$

Where, K_F and n are Freundlich constants. Where in Langmuir model q_m is the saturated monolayer sorption capacity and K_a is the sorption equilibrium constant. In Tempkin model, R is the universal gas constant and T is the absolute temperature. B_1 is Tempkin isotherm constants, K_t is the equilibrium binding constant (L/mol) corresponding to the maximum binding energy and constant B_1 is related to the heat of adsorption.[17]

During the experimentation strict quality control practices were adopted. In order to assure the quality control in the characterization and adsorption analysis, quality control (QC) samples (duplicate samples) were prepared and analysis were performed on both samples in parallel. Each quality control sample was assigned with a unique number, with the details of each sample recorded on a Sample Log Record. All the analyses were performed under strict quality control and utilizing standard ASTM procedures and methods. The known reference standards were used for the preparation of calibration curve of MB to determine the concentration of MB in the solution.

3. RESULTS AND DISCUSSION

This section discusses the results obtained in the study and analysis of results. Further, the comparison with the results reported in the past studies also discussed.

3.1. RESULTS OF PROXIMATE AND DETAILED ANALYSIS

The elemental analysis of *Acacia Tortilis* tree bark was performed and result obtained were compared with the constituent of other Acacia tree species repoted in the literature. Result reported in table 1 show that, the dominating constituent of the sample is carbon which is 53.9%, making the material a promising precursor material to prepare a good adsorbent. The range of carbon in other species reported in the literature (48% to 52.7%) is similar for various species of *Acacia* wood as shown in table 1.

Results from the proximate and detailed analysis of both carbons (AC-MWI and AC-CA) are presented in table 2. It can be seen from table 2 that the results obtained for both type of ACs are comparable with the properties of AC produced from other species of Acacia tree family. In addition, comparison made with the commercially available ACs in the market Filtrasorb® 400 and QAC-400.

3.2. CHARACTERIZATION OF ACTIVATED CARBONS

3.2.1. TOTAL ASH CONTENT

A good AC must have low ash content.[23] Ash content of AC-CA is 6.1% as compared to AC-MWI which has 5.2% (table 2). The ash content of AC-CA is slightly higher than the typical values reported in the literature. This may be attributed to the higher heating rate (i.e. 600°C) and impregnation volume ratio (i.e. 40%, V/V), as depolymerisation reactions between the volatile materials and phosphoric acid during the carbonization were affected. However, this parameter may be improved by lowering the heating rate and adjusting the impregnation ratio.[24] However, the ash content of AC-MWI is even lower than the commercial activated carbons and was reported in literature. This may be attributed to the fact that during microwave irradiation heating accomplished by molecular vibration which produced less ash than heating in furnace. It is because, bulk heating generates more ash content as compared to microwave heating. Similar findings are reported by researchers elsewhere.[25]

3.2.2. MOISTURE CONTENT

Moisture content affects the porosity of AC, as the moisture content increases, the porosity of AC also increases and vice versa. It can be seen from table 2 that the moisture content of both ACs are comparable (i.e. 3.6% and 4.4%). Lower moisture content is desirable for a good quality AC. Values obtained for both activated carbons are better than the reported values of commercial activated carbons. Similar conclusion reported elsewhere[26]

3.2.3. SURFACE AREA

Surface area indicates the capacity of AC to adsorb pollutants; larger the surface area of AC, more capable it is to remove the pollutants. BET-specific surface area obtained for AC-CA produced by using low temperature carbonization and AC-MWI by microwave heating was found to be 687 m^2/g and 836 m^2/g respectively. It can be seen from table 2 that the values for both activated carbons are reasonable as compared to BET-specific surface area of activated carbons reported in literature and even higher than commercial activated carbon (table 2, QAC-400).

Table 1. Results of elemental analysis for *Acacia Tortilis* bark with the values reported in literature

Constituent (weight %)	*Acacia Tortilis* bark	[11]*Acacia seyal*	[12]*Acacia asak*	[22]*Acacia nilotica*
C	53.9	51.3	52.7	48
K	1.26	1.82	0.93	--
Al	0.13	0.17	ND	--
N	0.40	0.33	0.42	0.4
H	5.5	5.8	5.3	6
Zn	0.02	0.01	0.01	--
S	0.03	0.03	0.02	--
P	0.07	0.09	0.1	--
O	39.7	32.9	41.5	44

Table 2. Comparison for the characteristics of AC-MWI and AC-CA with the values available in literature and commercially available

Property	AC-MWI	AC-CA	[11]*Acacia seyal*	[12]*Acacia nilotica*	*Filtrasorb® 400	#QAC-400
Ash (%)	5.2	6.1	5.9	5.8	5-6	6
Bulk Density (g/cc)	0.38	0.35	0.3	--	0.44	0.55
Moisture Content (%)	3.6	4.4	4.2	4.1	--	5
pH	6.9	6.5	6.5	7.0	6.2	9-10
BET-specific Surface Area (m^2/g)	836	687	762	590	944	400
Pore Volume (cm^3/g)	4.97	4.07	4.92	4.4	0.6	--
Iodine Number (mg/g)	870	819	827	480	1000	400

* Calgon Carbon Corporation, USA; # Quantum Active Carbon Pvt Limited;

3.2.4. *IODINE NUMBER*

Iodine adsorption is generally used to evaluate the effectiveness of activated carbon. The iodine number in the present study for AC-MWI and AC-CA found to be 870 mg/g and 819 mg/g respectively as compared to the typical values mentioned in the literature for other acacia species (table 2, 480 to 827 mg/g) and with the values of commercial grade activated carbons (400 to 1000 mg/g). Comparison in table 2 shows that both produced activated carbons meeting the typical values reported in literature for similar precursor materials and with commercially available activated carbons in the market. However, AC-MWI produced by microwave heating shows superior quality. Results show that during carbonization by microwave irradiation lesser percentage of ash formed and higher BET-specific surface area formed probably due to penetration of microwaves in deeper zones of precursor material which also results in a better iodine number value.[27]

3.3. ADSORPTION PROCESS EFFICIENCY

Process efficiency to remove MB while utilizing AC-MWI, AC-CA and commercial AC Filtrasorb ®-400 was evaluated in batch experiments as mentioned before. Temporal removal efficiency of AC-MWI, AC-CA and Filtrasorb ®-400 is presented in figure 2. The equilibrium time was found to be 55 minutes, 45 minutes and 65 minutes respectively. The corresponding amount of MB adsorbed at equilibrium on the surface of ACs was found to be 37.1 mg/g, 36.3 mg/g and 33.1 mg/g respectively. The efficiency of adsorption processes depends on various chemical and physical properties of both the adsorbate and adsorbent surface. However, in the present study, rate of removal was observed with time.[28] It can be seen from figure 2 that initially, commercial activated carbons removal is highest up to 35 minutes as compared to both ACs. However, the removal efficiency of AC-MWI becomes comparable to it. Furthermore, in about 45 minutes of experimental run the improvement in the removal efficiency became insignificant for AC-MWI. This may be attributed to the fact that, as the time progresses, initially the surface coverage of the adsorbent is high and afterward the adsorption decreases due to unavailability of adsorbent sites for MB molecules to adsorb.[29]

The final removal efficiencies of activated carbons achieved were 98.5%, 98.8% and 88.4% for Filtrasorb ®-400, AC-MWI and AC-CA respectively. The high removal efficiency of AC-MWI for MB dye may be attributed to the high BET-specific surface area and pore volume, which were produced by microwave heating.[23]

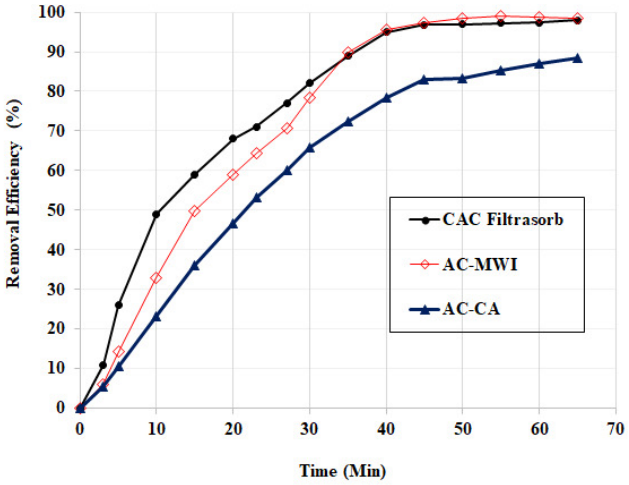

Fig. 2. Comparison of MB Removal efficiency of pr oduced ACs with commercial AC (initial concentration of 30 mg/L, 30°C)

3.4. ADSORPTION ISOTHERM ANALYSIS

The adsorption isotherm indicates the distribution of adsorbate molecules between the liquid phase and the solid phase when the adsorption process reaches the equilibrium state. To evaluate the adsorption capacity of AC- MWI produced from Acacia Tortilis tree bark, experimental runs were performed using various amount of AC- MWI and samples were allowed to reach equilibrium. Data generated was fitted to Freundlich, Langmuir, and Temkin isotherm.

Adsorption isotherms for all models are presented in figure 3, depicting that the equilibrium data is following Freundlich, Temkin as well as Langmuir isotherms to certain extent. However, data is best fitted to the Freundlich isotherm model as indicated by the value of correlation coefficient (R^2) (Table 3) which is nearly equal to one. Researchers in the past studies reported that the Freundlich constant K_F in the adsorption model (equation 3) gives indication of adsorption capacity. In the present study the K_F indicates that the process efficiency is high in the beginning and probably adsorbate molecules predominantly adsorb on the surface of outer and some macrospores. After sometime when the available sites are reduced, the adsorption takes place in the microspores, mesopores as well as in the nanopores until the steady state reach.[30,31]

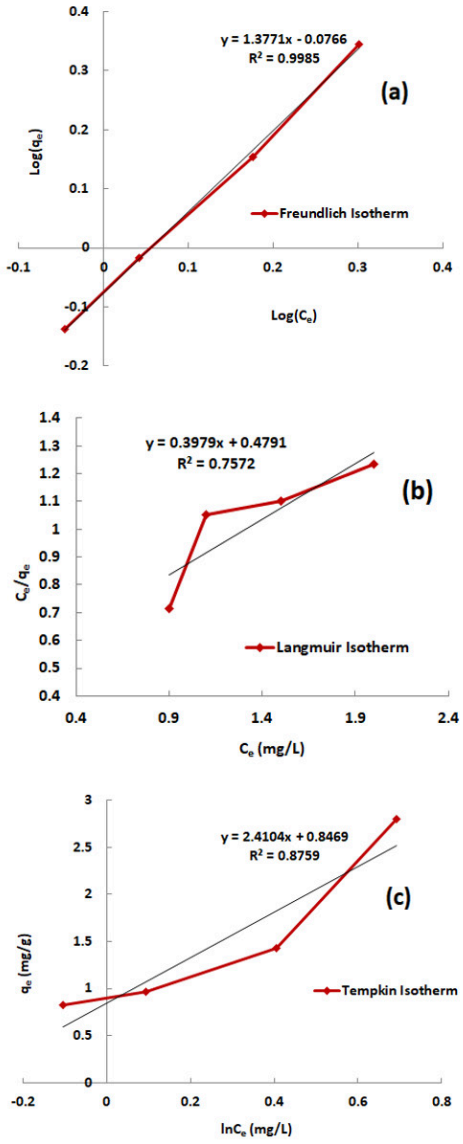

Fig. 3. Plot between log Ce vs log qe for (a) Freundlich isotherm (b) for Langmuir isotherm and (c) for Tempkin isotherm.

Table 3. Comparison of correlation coefficient (R^2) values obtained for all adsorption isotherm models

Isotherm Model	Correlation Coefficient (R^2)
Freundlich Isotherm	0.9985
Langmuir Isotherm	0.7572
Tempkin Isotherm	0.8759

4. CONCLUSION

Present study evaluates the preparation of activated carbon from Saudi Arabian *Acacia Tortilis* bark. *Acacia Tortilis* tree bark is local agricultural waste found to be promising raw material for the production of activated carbons. Characteristics of activated carbon produced by conventional chemical activation with phosphoric acid and carbonization, by low temperature heating and microwave irradiation, are well comparable with the characteristics of activated carbon produced by other species of Acacia tree branches reported in the literature and commercial activated carbon available in the market. It was found that AC-MWI has higher BET-specific surface area (836 m^2/g) as compared to AC-CA (687 m^2/g), demonstrating the effectiveness of the preparation method. Results revealed that there is an increase of 22.3% in micro-pores component and 21.7% BET-specific surface area obtained in AC-MWI as compared to AC-CA. Furthermore, AC-MWI and AC-CA, obtained 4.97 cm^3/g and 4.07 cm^3/g pore volumes respectively. These val-

ues are much better than the values of high quality commercial ACs (<1 cm^3/g). Temporal removal efficiency of AC-MWI, AC-CA and Filtrasorb ®-400 is presented in figure 2. The equilibrium time found to be 55 minutes, 45 minutes and 65 minutes respectively. The corresponding amount of MB adsorbed at equilibrium on the surface of ACs found to be 37.1 mg/g, 36.3 mg/g and 33.1 mg/g respectively. At equilibrium, removal efficiencies of activated carbons achieved were 98.5%, 98.8% and 88.4% for Filtrasorb ®-400, AC-MWI and AC-CA respectively. It was also found that AC can be produced by microwave irradiation in about 48.5% lesser time as compared to conventional low temperature heating. Adsorption data is fitted to Freundlich, Langmuir, and Temkin isotherm. However, it is best fitted to Freundlich isotherm model as indicated by values of correlation coefficients, reaching to unity (R^2 value 0.9985). Current study is intelligible with the ambitious Vision 2030 of Kingdom of Saudi Arabia by subsiding the import of activated carbon and expenditure on foreign exchange to meet the growing demand of activated carbon in the country. So the requirement can be met by producing low cost activated carbon from locally available waste materials in addition to managing the solid waste production by utilizing in beneficial purpose. Generally, the characteristics of activated carbon are judged by the large surface area, highly developed porous structures, and good mechanical stability. Therefore, the results obtained in this study suggest that *Acacia Tortilis* tree bark found in Saudi Arabia has potential as a precursor material to be used for the production of high quality green adsorbent by means of microwave induced phosphoric acid activation.

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ABBREVIATION

AC-CA Activated Carbon by Chemical Activation
AC-MWI Activated Carbon by Microwave Irradiation
MB Methyline Blue

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