INTERNATIONAL JOURNAL OF RESEARCH IN PHARMACY AND CHEMISTRY

Available online at www.ijrpc.com

Research Article

NEW CHEMICALLY PREPARED - WASTE COOKED TEA BASED ACTIVATED CARBON: FTIR, XRD, AFM, AND SEM SPECTROSCOPIC STUDIES

Kafa Khalaf Hammud[1], Abdulatif Mohammed Raouf[1],
Adulkareem Mohammed Ali Al-Sammarrie[2] and Ryadh Raheem Neema[1]
[1]Ministry of Science and Technology, Baghdad, Iraq.
[2] Department of Chemistry, College of Science, Baghdad University, Baghdad, Iraq.

ABSTRACT
Waste black tea- based activated carbon samples were prepared with conventional and / or ultrasound activation processes using Clorox or concentrated H_2SO_4- HNO_3 mixture as individual activating agent. The effects of activation process and applied activating agent were studied by FTIR, XRD, SEM, and AFM spectroscopies. The qualitative and quantitative analysis with statistical information of several prepared samples were summarized and discussed. The obtained results demonstrated the existence of micro- and nano- structural particles. The choice of starting material, activation process, and activating agent with their spectroscopic results suggested that the prepared activated carbon representing an economically promising material with wide spectrum of applications.

Keywords: Activated carbon, consumed black tea, FTIR, XRD, SEM, AFM.

INTRODUCTION

Activated carbon as amorphous carbon with high qualities in porosity surface area, penetration, adsorption capacity, and regenerations can be prepared from agricultural, food processes, and human consumption wastes as friendly environmental material with less cost by applying chemical ,physical,and physico-chemical activation methods[1-22].

Chemical activation process based on prior material and accomplished with thermal decomposition of the raw agricultural industrial residues with acidic or basic reagents [10, 21].

The aim of this study was directed to prepare activated carbon from largely consumed product in Iraq that is black tea after cooking with different activation agents and procedures, and characterize it with available FTIR, XRD, SEM, and AFM techniques.

EXPERIMENTAL PART

Chemicals (used as received)

Sulfuric and nitric acids were from BDH. Clorox bleaching solution containing 5% sodium hypochlorite was purchased from local market in Baghdad, Iraq. Consumed black tea samples were collected at home after usual drinking step with major notice that black tea sample where purchased from local market in Baghdad city, Iraq with different commercial marks.

Equipment and Instruments

Ultrasonic bath, F5200b Decon Laboratories Ltd., East Sussex, United Kingdom were used. FTIR spectra were recorded using infrared spectrophotometer Shimadzu, Japan. X-Rays Diffraction also recorded using a Lab X (XRD-6000), Shimadzu, Japan and using the operational details of the following: Target: Cu Kα radiation, Wave: 1.54060 A, Voltage: 40.0 kV, Current: 30.0 mA, Speed: 5.0000 deg / min. Scanning Electron Microscope (SEM) type VEGAS III TescanAlgiua origin. Atomic Force Microscope**,** AA3000 - 220V, Angstrom Advanced Inc., USA**.**

To obtain a clear quantitative image of grain size and surface roughness, AFM measurements of several prepared samples of activated carbon with different applied conditions were conducted with an AFM –

AA3000 of Angstrom advanced Inc., USA) in ambient conditions.

Modified Multistep Preparation of activated carbon

Sample A

Cooked or consumed black tea was dried in open air vessel at room temperature then in electrical oven at 70 °C for five hours then grinded with domestic grinder to fine powder.

Sample A1

unweight quantities of sample A were heated in muffle furnace at 500 °C for (15 minutes) the cooled to room temperature.

Sample B

7.5 gm of sample A1 were impregnated in 70 mL of Clorox solution for 24 hrs. then filtered off, washed several times with deionized water to neutral pH, and dried.

Sample C

20 gm of sample A1 were 20 mL of H_2SO_4:HNO_3 concentrated mixture (1:1) by adding 10 mL of concentrated sulfuric acid then 10 mL of concentrated nitric acid. The resulting paste was left to stand at lab temperature with occasionally stirring for more than 100 hrs. then diluted with deionized water, filtered off, washed with deionized water to pH 7, and dried.

Sample D

The same procedure of preparing sample C was repeated except that the resulted diluted mixture with deionized water was left for 48 hrs. at lab temperature, filtered off, washed with deionized water to pH 7, and dried.

Sample E

0.36 gm of sample A1 and 5 mL of Clorox solution were mixed for one hour with ultrasonic apparatus, filtered off, washed with deionized water to pH 7, and dried.

RESULTS AND DISCUSSION

Non-crystalline or porous activated carbon material with surface area ranged (300-5000) m^2/gm used in adsorption processes, surface reactions, ion exchange, and mechanical filtration can be classified to granular and powder where the first class used in gases adsorption while the second class for purification of liquids[23].

Activated carbon can be prepared from raw materials with varying in quality and composition depending on chemical treatment methods based upon hydrogen removal linked to raw materials :Dates stones and walnut shells (as primary carbon materials) Asphalt (binding material) , Waste Tar refineries, Baiji / Iraq, Petroleum charcoal[24-26].

Figure 1 shows the modified multistep preparation of activated carbon from waste cooked black tea with different activation agents (Clorox solution and mixture of concentrated sulfuric and nitric acids) and using muffle furnace in carbonization step beside ultrasonic bath for activation step with Clorox solution.

According to our research with electronic and hard published scientific articles, Clorox solution as a commercial bleach trade mark of 5.25% of sodium hypochlorite or mixture of concentrated sulfuric and nitric acids have not been applied as an activation agent for preparation of activated carbon. Conventional (muffle furnace) heating was putted in use to achieve this modified multistep work and comparing the obtained results of each final step product with others. The reasons that encourage us to choose these modified multistep (Figure 1) can be summarized as below:

1. Choosing available and low cost material thrown to trash that is largely consumed waste cooked black tea.
2. Selection of newly activation agents may applied and comparison their results.
3. To achieve point 2, sodium hypochlorite was our first choice. Clorox as a obtainable commercial bleach solution containing 5.25% of sodium hypochlorite provided us low cost activation agent.
4. Sulfuric acid or nitric acid was individually applied as activation agent but not as a concentrated mixture of them.

Availability of ultrasonic irradiation bath gave us another point of view that may take place in comparison with other above points. Table 1 shows the effect of applying the above points through residual percentage calculated from the difference between material weights after and before each identical single step in percentage state.

Analysis of Infrared spectroscopy (FTIR)

Infrared spectra of tea residues before and after carbonization and activation processes were depicted in Figure 2. They showed several characteristic vibration bands such as hydroxyl, carbonyl, C-O, N-O, S=O, and C-H. in this matter, sample (A) as a prior material (before carbonization and activation steps) showed hydroxyl group at 3417 cm^{-1}, carbonyl stretching at 1728 cm^{-1} (for ketone, aldehyde, or acetyl derivatives) and 1648 cm^{-1}, bending of (C-O) group at 1452 cm^{-1}, stretching of (N-O) group at 1375 cm^{-1}. While stretching vibration of (S=O and Si-O-Si) noticed at (1064 and 601) cm^{-1} respectively [27].

In this matter, asymmetrical and symmetrical vibrations of (C-H) appeared into (2924 and

2854) cm^{-1} as identical characterization of methyl and alkyl methylene beside that the broad (OH) band indicated high degree of hydrogen bonding. FTIR spectrum of sample (A) depicted in Figures 3 and 4 showed all previous mentioned bands beside $1633cm^{-1}$ that belong to stretching vibration of (C-O).

The prepared activated carbon samples (A1, B, C, D, E) were also characterized with FTIR instrument and showed the all/ or some of the mentioned vibration modes with some shifting in wavenumbers (Figure 2). The shifting or disappearance reflected the influence of the acting step (carbonization, activation, and using assistant tool (ultrasonic irradiation bath).

FTIR results can be compared with other published results of preparing activated carbon from residual tea with different activation methods such as using potassium carbonate, potassium acetate with tube furnace, or sodium hydroxide or sodium carbonate with microwave irradiation or heating with muffle oven[21, 28-33]. This comparison showed that presence of different hetero-atoms (N, S, O) as functional groups with demand oxygen in tea structure represented an important notice of effective centers in surface interactions and activated carbon behavior.

Analysis of X-rays diffraction (XRD)

One of the most effective technologies that used to identify the nature of crystalline materials is X-Rays Diffraction. XRD patterns (Figure 3) show three different Bragg angle (2θ,deg.) with maximum intensity in all measured samples (24-29) degrees and around (44) degrees corresponding to (002) and (100) diffraction of the disordered stacking of the microstructures. Also, the obtained results of prepared activated carbon samples (Figure-3, Table2) illustrated that spaces between interlayers with weak crystalline character as non-graphitized carbonaceous structure are larger than graphite as calculated by Scherrer equation [34]:

$$d=k\lambda/\beta.\ \cos\theta$$

where:

d= average crystallite size (or mean size of the ordered crystalline) in (nm).

λ= X-ray wavelength in (nm)

β= the peak width of the diffraction peak profile (or the line broadening) at half maximum (FWHM) in (radius);[β(radian)=2π β(deg.)/ 360].

K= shape factor (or constant related to crystalline shape= 0.91).

θ= Bragg angle in (radius); θ(radian)=2π θ(deg.)/ 360.

From Table 2, it can be concluded that muffle furnace carbonization sample and the using of Clorox as an activation agent (**sample E, 41.4 nm**) was more performance in producing large average size than concentrated acids mixture with direct dilution with water (**sample D, 27.9 nm**) or after (48) hours of dilution with water (**sample C, 36.9 nm**).These results coincide with many published documents of preparation and characterization of activated carbon[1, 21, 25, 26].

Analysis of Scanning Electron Microscopy(SEM)

Analysis of Scanning Electron Microscopy (SEM) of the prepared activated carbon samples after treatment with Clorox or concentrated acid mixture depicted in Figure4 had been done.

The obtained results clarify that activated muffle furnace carbonization samples with Clorox and concentrated acids mixture (B and D) respectively have porous structures outputted from heating and activation agentseffects on residual tea (Figure 4). These (B and D) samples had gaps, channels, or slits of porous structure as Microscopic porous character. This character conjugated with size and shape irregularities was clearly affected with activating materials.

The configuration of pores is linked to the formation of carbon dioxide and carbon monoxide. The formation of these gases resulted from oxidation processes of carboxylic and/or lactone (for CO_2 formation), phenol, carbonyl ether, and/ or quinone (for CO formation), and/or anhydride (for both CO_2 and CO formations) functional groups[35]. Previous mentioned notices of our prepared activated carbon were similar in characteristic properties with others that prepared from tea or vegetarian sources[21, 26, 33, 36 -38].

Analysis of atomic Force Microscopy(AFM)

To understand the small scale phenomena, micro- and / or nano- tribological studies are needed to be applied especially with Atomic Force Microscopy instrumentations. In order to establish the effect of muffle, Clorox – concentrated acids mixture, conventional – ultrasonic mixing, and exposure time to concentrated acid mixture on the morphology of our newly prepared samples, AFM technology was applied. The AFM morphological results are presented in Table-3 and Figures (5 and6).

The effects of the applied conditions (type of carbonization process (muffle), activation agent, time with same activation agent, ultrasonic, …) on the obtained characteristics were summarized in Table 3 through AFM surface roughness parameters [roughness average (S_a, nm), root mean square (S_q, nm), surface kurtosis (S_{ku}), Surface area ratio (S_{dr}, nm), Fractal dimension, Avg. diameter in nm] beside other calculated data from the AFM images and the instrument software [∑ of volume (%), highest volume (%)].

These tabulated data cannot consider as representative absolute surface values because of sample, image, and instrument processing.

From amplitude or height parameters of all measured samples with AFM instrument, the increasing sequence of roughness average (**S_a**) which depends on the average profile of heights was [**S_a (D) > S_a (E) > S_a (B)].**

Root mean square (S_q) as a function of the square root of the measures is more sensitive to peak and valley than roughness average (S_a) because S_q represented the standard surface height deviation (as square root) not the height deviation. Sq increasing series was [S_q**(D)** >S_q**(E)** >S_q**(B)].** Also, Sq to Sa ratio as an indication to the Gaussian distribution of surface was less than 1.31 (**ranged from 1.197 to 1.155**)[39, 40].

With muffle furnace carbonization and two different types of activation agents [Clorox and concentrated H_2SO_4-HNO_3 mixture], the resulted acid mixture sample (**D**) gave the higher roughness average (S_a) value compared with (**E or B**) and this notice reflects the importance of activation time. These observations can be repeated in their effects with root mean square (S_q)value.

The distribution of the spikes above and below a mean line for all measured samples, which is represented by roughness kurtosis (S_{ku}), were less than3as bumpy or flat surface (Platykurtic surface) not spikes or surface with more peaks than valleys(S_{ku} value is higher than3) or perfectly random surface as Gaussian amplitude distribution (Mesokurtic surface, S_{ku}= 3). S_{ku} increasing sequence were **[S_{ku} (E) >S_{ku} (D) >S_{ku} (B)**]. This numerical increasing sequence of S_{ku} (or flatness parameter) is more apparent to eye by observing the obtained AFM images especially as 3D (Figures 4 and 5)[39, 41, 42].

Surface area ratio (S_{dr}) as a hybrid parameter increased with different sequence that was [**S_{dr} (D) >S_{dr} (E) >S_{dr} (B)**] which is in the same direction of S_a or $S_q$increasing sequence. From this parameter, roughness ratio can be calculated with (r=1+S_{dr}/100) equation [43]. Our calculated (r) was more than (1) as another indication of roughness.

The resulted fractal dimensions were varied from **2.53 to 2.79; close to 3**; with increasing sequence of our prepared activated carbon (**D >E> B**) as the same of conclusion of S_{dr}, S_a,or$S_q$which is another characteristic of rough three dimensional surface not of high self-affinity smooth surface when fractal dimension is close to 2. To understand the effects of the applied conditions on the resulted activated carbon samples, other main values came to light with AFM science and technology that are sum of volume (%) and highest volume (%) that computed with nano – diameter (or less than 100 nm), beside average diameter as presented in Table 3 and Figures (5-6).

The tested samples showed important values with diameter **(≤100 nm)** that were total volume % (**50.97-82.6**) % and highest volume % (**9.66-13.04**) in this range with increasing sequence (**D >E > B)** and **(D > B >E)** respectively. Also, average diameter varied from (**82.61-95.88**) nm with increasing series [**B (95.88 nm) >E (91.28 nm) > D (82.61 nm)**].

Table 1: Residual percentage of the prepared materials according to their conditions

Process direction	Used equipment	Activation agent	Residual percentage, %
A1 to C and D	Muffle furnace	Acid mixture	75
A1 to E	Muffle furnace, ultrasonic (1 hr.)	Clorox	38

Table 2: XRD results according to Scherrer equation

Sample	2θ, deg.	β(FWHM), deg.	d=kλ/β. cosθ
A1	29.4088	0.3105	26.74646
B	29.2844	0.4041	20.54545
C	43.9521	0.2346	36.92305
D	43.9727	0.3098	27.96248
E	43.9923	0.2090	41.45155

Table 3: AFM results of several prepared samples

		B	D	E
Amplitude parameter, nm	**Roughness Av. (S_a)**	0.445	0.901	0.693
	Root mean square(S_q)	0.514	1.06	0.83
	Surface kutosis (S_{ku})	1.8	1.97	2.26
Hybrid parameter	**Surface area ratio (S_{dr})**	0.0535	0.248	0.206
Fractal dimension		2.53	2.79	2.69
Avg. diameter, nm		92.21	82.61	95.88
Notes		Results of (**65-100**) nm Σ of Volume(%) : **65.85** highest volume (%) : **12.15** at **85** nm	Results of (**55-100**) nm Σ of Volume(%) : **82.6** highest volume (%) : **13.04** at **75** nm	Results of (**25-100**) nm Σ of Volume(%) : **50.97** highest volume (%) : **9.66** at **90** nm

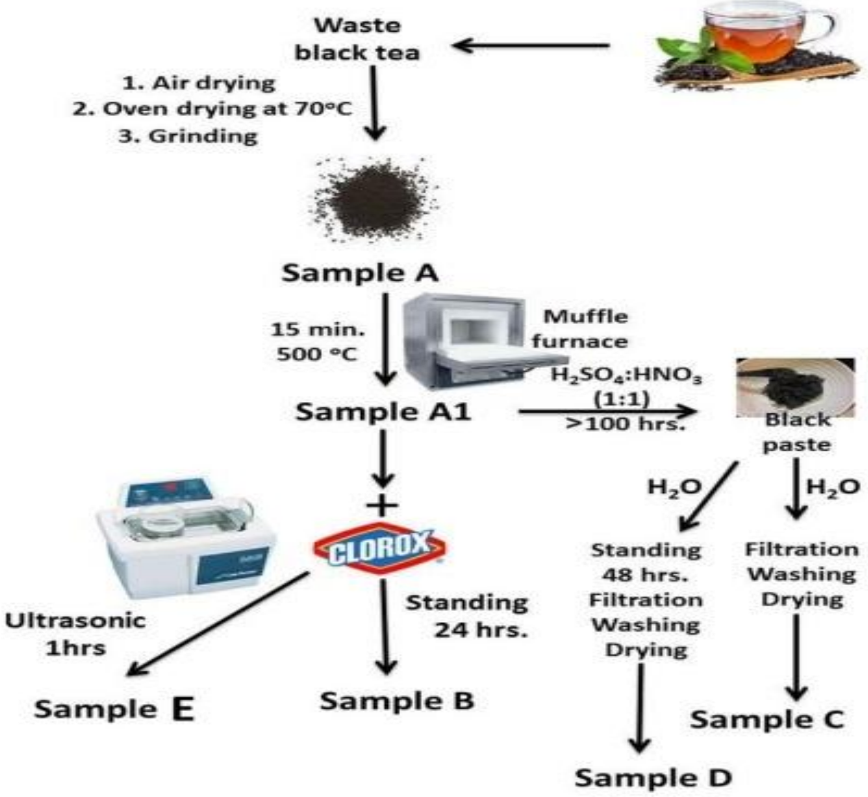


Fig. 1: Modified multistep preparation of activated carbon from waste cooked black tea with different activation agents and muffle furnace

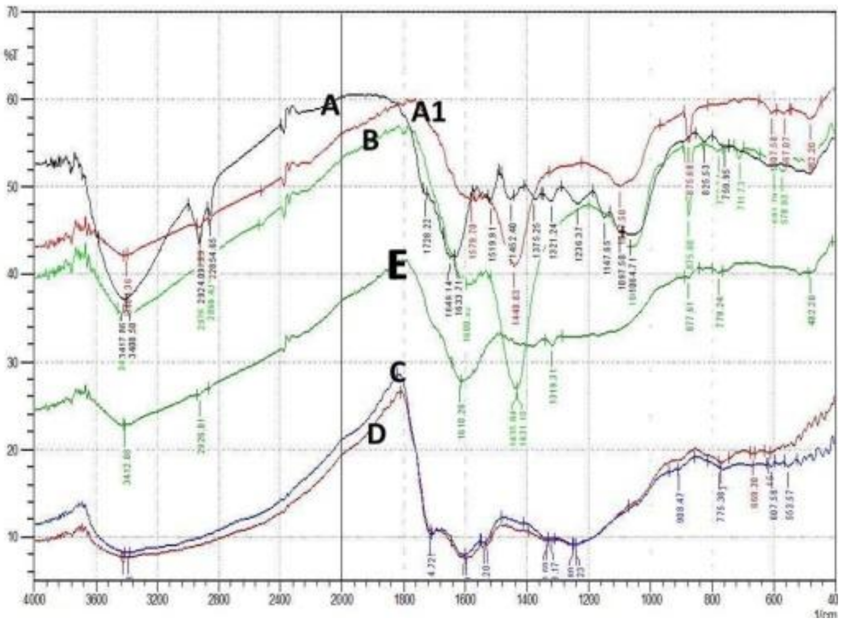


Fig. 2: FTIR spectrum of activated carbon after carbonized tea residues with muffle furnace with different activation agents and processes

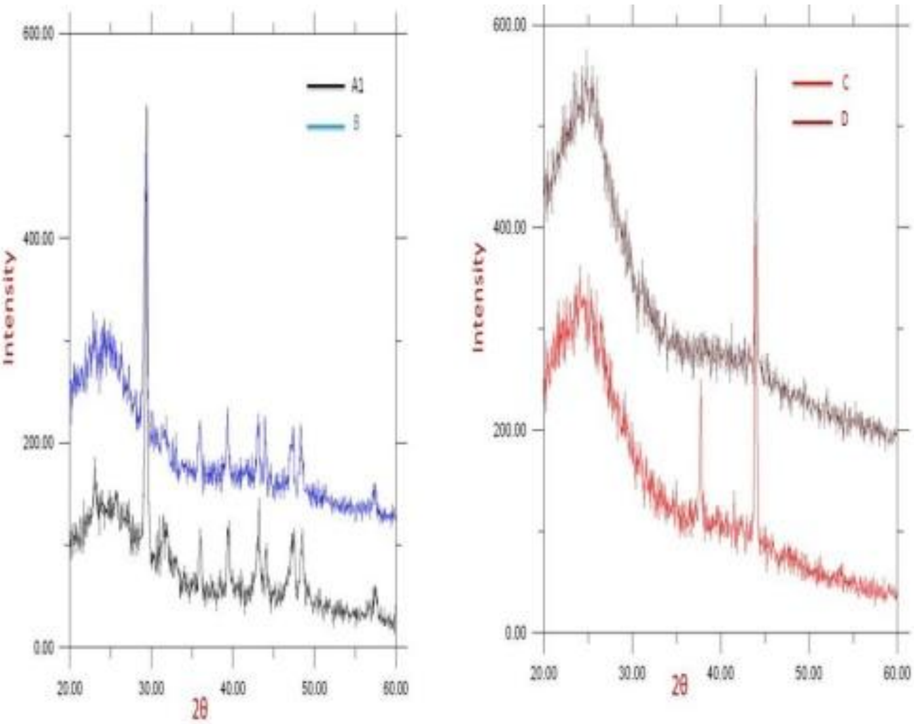


Fig. 3: XRD analysis of prepared activated carbon with muffle furnace carbonization (A1), and Clorox (B), concentrated acids mixture (C and D) activations

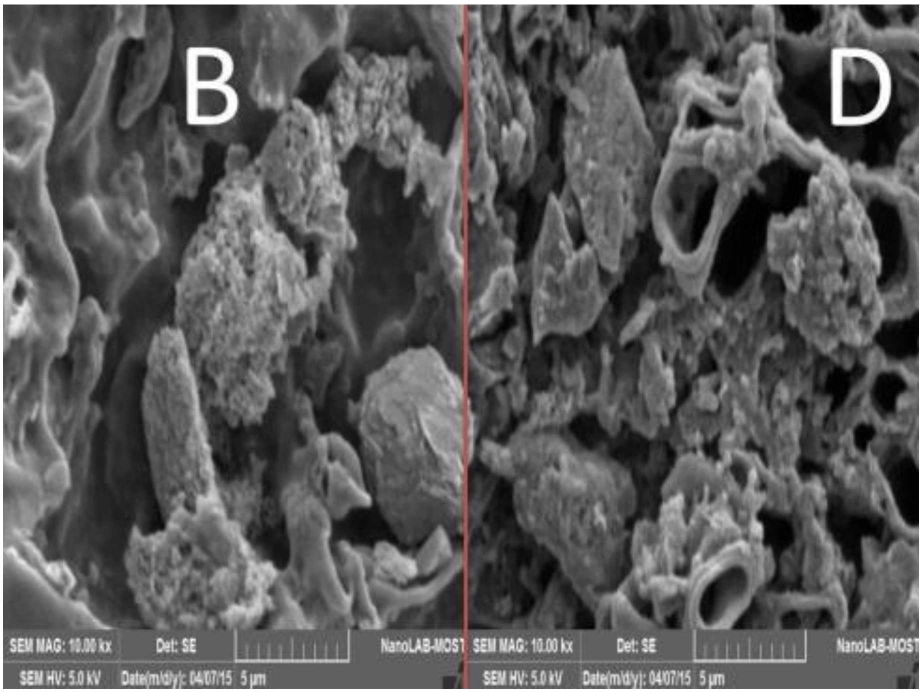
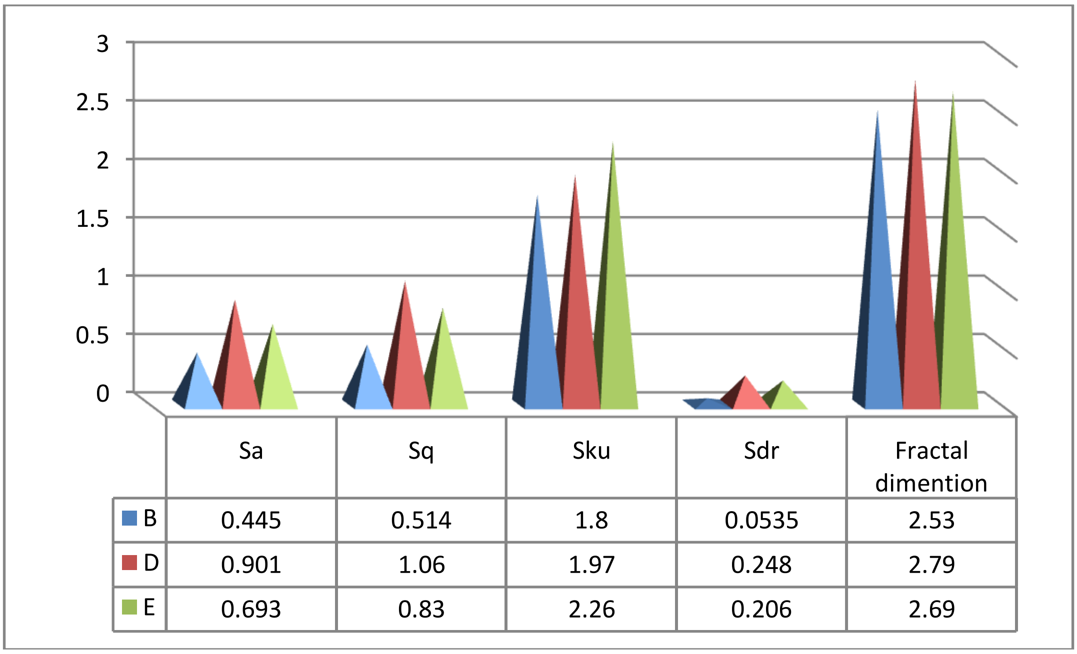
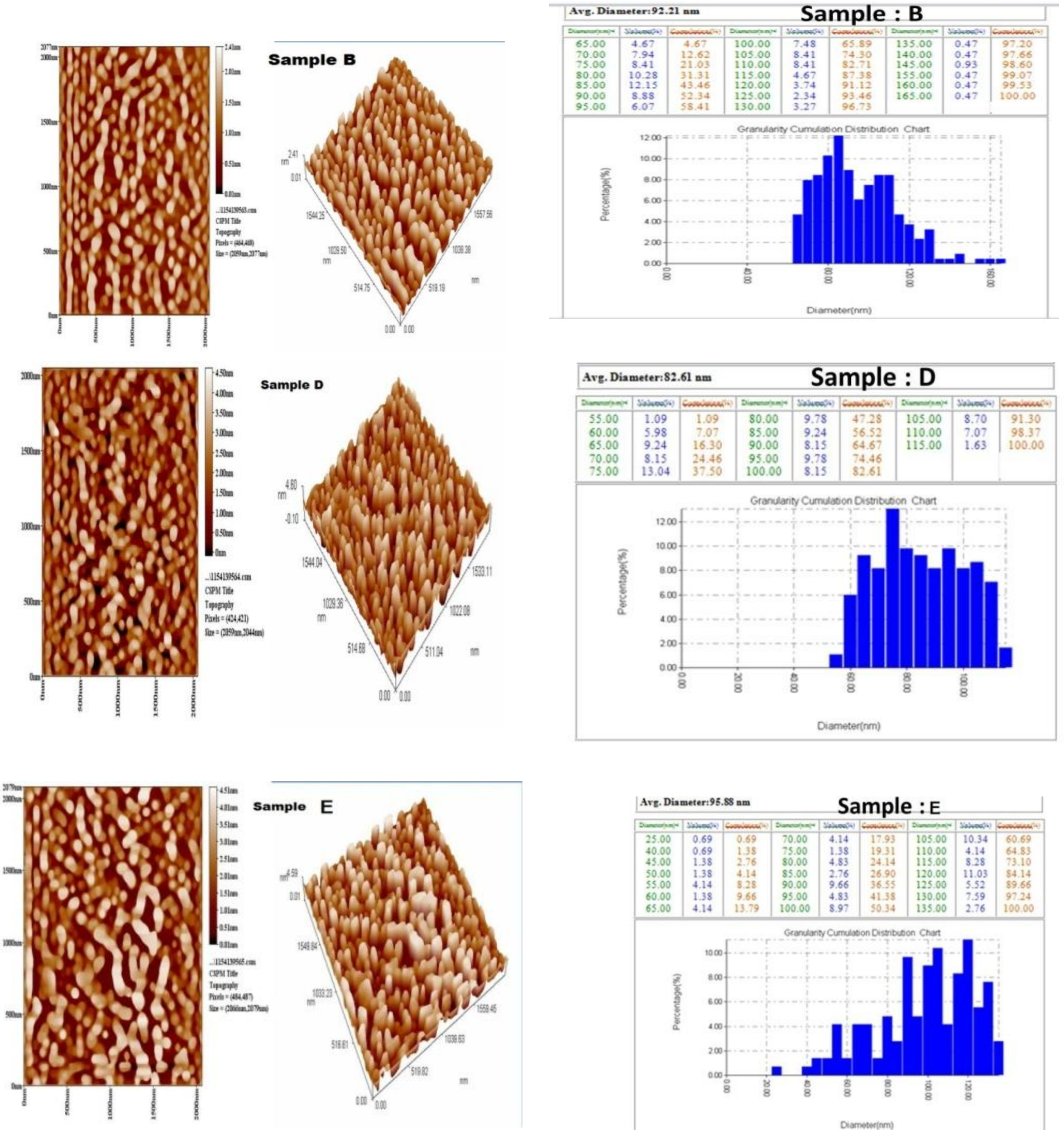


Fig. 4: SEM images of the prepared activated carbon samples after muffle furnace carbonization and activation with Clorox (B) or concentrated H_2SO_4:HNO_3 mixture (D)



	Sa	Sq	Sku	Sdr	Fractal dimention
🟦 B	0.445	0.514	1.8	0.0535	2.53
🟥 D	0.901	1.06	1.97	0.248	2.79
🟩 E	0.693	0.83	2.26	0.206	2.69

Fig. 5: AFM Fractal dimension, Amplitude (S_a, S_q, and S_{ku}) and (S_{dr}) Hybrid parameters of (B, D, F, G, H, I, and K) prepared activated carbon samples



Avg. Diameter:92.21 nm **Sample : B**

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
65.00	4.67	4.67	100.00	7.48	65.89	135.00	0.47	97.20
70.00	7.94	12.62	105.00	8.41	74.30	140.00	0.47	97.66
75.00	8.41	21.03	110.00	8.41	82.71	145.00	0.93	98.60
80.00	10.28	31.31	115.00	4.67	87.38	155.00	0.47	99.07
85.00	12.15	43.46	120.00	3.74	91.12	160.00	0.47	99.53
90.00	8.88	52.34	125.00	2.34	93.46	165.00	0.47	100.00
95.00	6.07	58.41	130.00	3.27	96.73			

Avg. Diameter:82.61 nm **Sample : D**

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
55.00	1.09	1.09	80.00	9.78	47.28	105.00	8.70	91.30
60.00	5.98	7.07	85.00	9.24	56.52	110.00	7.07	98.37
65.00	9.24	16.30	90.00	8.15	64.67	115.00	1.63	100.00
70.00	8.15	24.46	95.00	9.78	74.46			
75.00	13.04	37.50	100.00	8.15	82.61			

Avg. Diameter:95.88 nm **Sample : E**

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
25.00	0.69	0.69	70.00	4.14	17.93	105.00	10.34	60.69
40.00	0.69	1.38	75.00	1.38	19.31	110.00	4.14	64.83
45.00	1.38	2.76	80.00	4.83	24.14	115.00	8.28	73.10
50.00	1.38	4.14	85.00	2.76	26.90	120.00	11.03	84.14
55.00	4.14	8.28	90.00	9.66	36.55	125.00	5.52	89.66
60.00	1.38	9.66	95.00	4.83	41.38	130.00	7.59	97.24
65.00	4.14	13.79	100.00	8.97	50.34	135.00	2.76	100.00

Fig. 6: 2D - 3D images and Granularity-Cumulation Distribution results of B, D, and F prepared activated carbon samples

REFERENCES

1. Zhong Z, Yang Q, Li X, Luo Liu Y, and Zeng G. Preparation of Peanut hull-based activated carbon by Microwave-Induced Phosphoric acid activation and its application in Remazol Brilliant Blue R adsorption. Ind Crops Prod. 2012;37(1):178-185.
2. Hayashi J, Kazehaya A, Muroyama K and Watkinson A. Preparation of Activated Carbon from Lignin by chemical Activation. Carbon 2000; 38(13):1873-1878.
3. AlSlaibi T, Abustan I, Ahmad M and Abu Foul A. A Review: Production of Activated Carbon from Agricultural byproducts via Conventional and Microwave Heating. J ChemTechnolBiotechnol. 2013;88(7):1183-1190.
4. Al-Slaibi T, Abustan I, Ahmad M and Abu Foul A. Cadmium Removal from Aqueous Solution Using Microwaved Olive Stone Activated Carbon. J Environ Chem Eng. 2013;1(3):589-599.
5. Klijanienko A Lorenc-Grabowska E and Gryglewicz G. Development of Mesoporosity during Phosphoric Acid Activation of Wood in Steam

Atmosphere. Bioresour Technol. 2008;99(15): 7208-7214.

6. Zubizarreta L, Arenillas A, Pirard J, Pis J and Jo N. Tailoring the Texture Properties of Activated Carbon Xerogels by chemical Activation with KOH, Microporous Mesoporous Mater. 2008;115(3):480-490.
7. Urabe Y, Ishikura T and Kaneko K. Development of Porosity in Carbons from Yeast Grains by Activation with Alkali metal Carbonates. J Colloid Interface Sci. 2008;319(1):381-383.
8. Hameed B and El-Khaiary M. Equilibrium, Kinetics, and Mechanism of Malachite Green Adsorption on Activated Carbon Prepared from Bamboo by K_2CO_3 Activation and Subsequent Gasification with CO_2. J Hazard Mater. 2008;157(2-3):344-351.
9. Hameed B, Ahmad A and Latiff K. Adsorption of Basic Dye (Methylene Blue) onto activated Carbon Prepared from Rattan Sawdust. Dyes Pigments. 2007;75(1):143-149.
10. Singh C, Sahu J, Mahalik K, Mohanty C, Mohan B and Meikap B. Studies on the Removal of Pb(II) from Wastewater by Activated Carbon developed from Tamarind Wood Activated with Sulfuric Acid. J Hazard Mater. 2008;153(1-2):221-228.
11. Ugurlu M, Gurses A and Acikyildiz A. Comparison of Textile Dyeing Effluent Adsorption on Commercial Activated Carbon and Activated Carbon Prepared from Olive Stone by $ZnCl_2$ Activation. MicroporousMesoporous Mater. 2008;111(1-3):228-235.
12. Petrov N, Budinova T, Razvigorova J and Parra P. Conversion of Olive Waste to Volatiles and Carbon Adsorbents. Biomass Bioenerg. 2008;32(12):1303-1310.
13. Cabal B, Budinova T, Ania C, Tsyntsarski B, Parra J and Petrova B. Adsorption of Naphthalene from Aqueous Solution on Activated Carbons Obtained from Bean Pods. J Hazard Mater. 2008;161(2-3): 1150-1156.
14. Abe I, Fukuhara T, Iwasaki S, Yasuda K, Nakagawa K, and Iwata Y. Development of a High Density Carbonaceous Adsorbent from Compressed Wood. Carbon. 2001;39:1485-1490.
15. Sanchéz-Polo M and Rivera-Utrilla J. Adsrobent-Adsorbate Interactions in the Adsorption of Cd(II) and Hg(II) on onzonied Activated Carbons. Environ Sci Technol. 2002;36(17):3850-3854.
16. Román S, González J, González-García C and Zamora F. Control of Pore Development During CO_2 and Steam Activation of Olive Stones. Fuel Process. Technol. 2008;89(8):715-720.
17. Alcañiz-Monge J, Lillo-Ródenas M, Bueno-López A and Illán-Gómez M. The Influence of Iron Chloride Addition to the precursor Pitch on the Formation of Activated Carbon Fibers. MicroporousMesoporous Mater. 2007;100(3):202-209.
18. Zhang T, Walawender W, Fan L, Fan M, Daugaard D and Brown R. Preparation of Activated Carbon from Forest and Agricultural residues through CO_2 Activation. ChemEng J. 2004; 105(1):53-59.
19. Alvim-Ferraz M and Gaspar C. Catalytic Activity of Active Carbons Impregnated before activation of Pinewood Sawdust and nutshells to be used on the Control of Atmospheric Emissions. J Hazard Mater. 2005;119(1-3):135-143.
20. Tan I, Ahmad A and Hameed B. Adsorption of basic Dye on High-Surface-Area Activated Carbon Prepared from Coconut Husk: Equilibrium, Kinetic, and Thermodynamic Studies. J Hazard Mater. 2008;154(1-3):337-346.
21. Rauof A, Hammud K, Mohammed J, Akosh N, Hamza F, Hassan M, Omran N, Zanad D and Ammen E. Removal of Benzene and Toluene from their Polluted Aqueous solutions by using Natural Adsorbent. Third Iraq Oil and Gas Conference, Amara, Iraq, 28-29. October, 2015.
22. Hameed B, Tan I and Ahmad A. Adsorption Isotherm, Kinetic Modeling, and Mechanism of 2,4,6-Trichlorophenol on Conconut Husk-Based Activated Carbon. ChemEng J. 2008;144(2): 235-244.
23. Stoeckli H. Microporous Carbons and their Characterization: The Present State of the Art. Carbon 1990;28(1):1-6.
24. Qasim L. The production of activated carbon from plant sources, MSc Thesis, University of Mosul, Mosul, Iraq, 1991.
25. Ramadhan O, Hamdoon A, and Mahmood E. Production of Activated Carbon via Grafting and Chemical

Activation. National J Chem. 2006;24:580-586.

26. O'Grady T and Wennerberg A. High Surface Area Active Carbon", ACS symposium series 303, ACS Publication, Washington, DC, 1984.
27. Terzyk A. The Influence of Activated Carbon Surface Chemical Composition on the Adsorption of Acetaaminophen (Paracetamol) in vitro: Part II: TG, FTIR, and XPS Analysis of Carbons and the Temperature Dependence of Adsorption Kinetic at the Neutral pH. Colloids Surf A: PhyscochemEng Aspects. 2001;177(1): 23-45.
28. Gundogdu A, Duran C, Senturk H, Soylak M, Imamoglu M and Onal Y. Physicochemical Characterizatics of a Novel Activated Carbon Produced from Tea Industry Waste. J Anal ApplPyrol. 2013;104:249-259.
29. Duran C, Ozdes D, Gundogdu A, Imamoglu M, and Senturk H. Tea – Industry Waste Activated Carbon, as a Novel Adsorbent for Separation, Preconcentration, and Speciation of Chromium. Anal Chim Acta. 2011;688(1):75-83.
30. Yagmur E, Ozmak M and Aktas Z. A Novel Method for Production of Activated Carbon from Waste Tea by Chemical Activation with Microwave Energy. Fuel. 2008;87(15-16):3278-3285.
31. Auta A and Hameed B. Preparation of Waste Tea Activated Carbon Using Potassium Acetate as an Activating Agent for Adsorption of Acid Blue 25 Dye. ChemEng J. 2011;171(1):502-509.
32. Tsubaki S and Azuma J. Total Fractionation of Green Tea Residue by Microwave – assisted Alkaline Pretreatment and Enzymatic Hydrolysis. Bioresour Technol. 2013;131:485-491.
33. Akar E, Altinişik A and Seki Y. Using of Activated Carbon Produced from spent Tea Leaves for the Removal of malachite Green from Aqueous solution. Ecol Eng. 2013;52:19-27.
34. Marsh H and Reinoso F. Activated Carbon, Elsevier, London, 1st Edition, 2006.
35. Figueiredo J Pereira M, Freitas M and Orfao J. Modification of the Surface Chemistry of Activated Carbons. Carbon. 1999;37(9):1379-1389.
36. Hejazifar M, Azizian S, Sarikhani H, Li Q and Zhao D. Microwave assisted Preparation of Efficient Activated Carbon from Grapevine Rhytidome for the Removal of Methyl Violet from Aqueous solution. J Anal ApplPyrol. 2011;92:258-266.
37. Maldhure A and Ekhe J. Preparation and Characterization of Microwave Assisted Activated Carbon from Industrial Waste Lignin for Cu(II) sorption. ChemEng J. 2011;168(3):1103-1111.
38. Deng H, Zhang G, Xu X, Tao G and Dai J. Optimization of Preparation of Activated Carbon from Cotton Stalk by Microwave Assisted Phosphoric Acid – Chemical Activation. J Hazardous Mater. 2010;182(1-3): 217-224.
39. Thomas T. Rough Surface, Imperial College Press, London, 2nd Edition, 1999.
40. Kumar B and Rao T. AFM Studies on Surface Morphology, Topography, and Texture of Nanostructured Zinc Aluminum Oxide Thin Films. Digest J NanomaterBiostr. 2012;7(4):1881-1889.
41. Magonov S and Whangbo M. Surface Analysis with STM and AFM, Expeimental and Theoritical Aspects of Image Analysis. VCH publishers, Weinheim, 1996.
42. Sulaymon A, Mohammed S and Abbar A. Characterization and Electrochemical Preparation of Thin Films of Binary heavy Metals (Cu-Pb, Cu-Cd, Cu-Zn) from simulated Chloride Wastewaters. Int J Electrochem Sci. 2014;9:6328-6351.
43. Palacios-Cuesta M, Cortaiarena A, Garcia O and Rodriguez-Hernández J. Fabrication of Functional Winkled Interfaces from Polymer Blends: Role of the Surface Functionality on Bacterial Adhesion. Polymers. 2014;6(11):2845-2861.