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# Production of High Surface Area Activated Carbon from Grass (Imperata)

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**Abstract**

In this work the production of activated carbon (AC) from Imperata is done by microwave assisted Potassium hydroxide (KOH) activation and using this activated carbon for the purpose of the uptake of amoxicillin (AMX) by adsorption process from aqueous solution. The effects for irradiation power (450-800W), irradiation time (6-12min) as well as impregnation ratio (0.5-1 g/g) on the AMX uptake and yield AMX uptake at an initial concentration of AMX (150 mg/g). The optimum conditions were 700 W irradiation power, 10 min time of irradiation, as well as 0.8 g/g impregnation ratio with 14.821% yield and 12.456 mg/g AMX uptake. Total volume of hole and the area of the surface (BET) are 0.3027 m³/g, and 552.7638 m²/g respectively. The properties for the activated carbon were examined via Scanning Electron Microscopy (SEM).

*Keywords: Adsorption, Activated carbon , KOH*

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## 1- Introduction

Pharmaceutical components are found as wastes in water and have been known as the dangerous chemical matters [1], [2]. Lately, Amoxicillin was found in hospital waste water [3]. Several techniques such as coagulation, biodegradation and chemical oxidation etc., are used for the elimination of antibiotics from wastewater. The process of adsorption have proven to be a well technique, thanks to major advantages like applicability of a large concentration extent of adsorbate, efficient elimination capacity, low equipments price , as well as the existence of much rate-controllable parameters [4],[5].

Amoxicillin is one of the industrial antibiotic which was high microorganism resistance. AMX wastes cause a distasteful odor, lacing condition, and can reason microbe impedance between pathogen organisms or the disease for microorganism that are efficient in wastewater handling.

The impedance microorganism might be the reason of sickness which can't be handled via traditional antibiotics. Because of those causes, AMX has to be compelled to be treated before dislodge to the ambience [6]. Activated carbon could be a pored carbon matter that has a high sorption capability which is wide utilized such adsorbent within the filtrate for liquids and gases [7].

Some of wide classification is manufactured for popular purposes depend on their physical properties as Powder Activated Carbon (PAC) or Granular Activated Carbon (GAC).

GAC is made from a crushed material, granular raw material, or pellets produced from fine raw material and a binder, whereas PAC is from crushed or ground carbon particles.

The particle size of PAC is commonly less than 100 μm; whereas the particle size of GAC is commonly around 1 to 5mm. Generally, PAC is widely used for water treatment. GAC is used for adsorption from the gas phase. Either PAC or GAC may be utilized for both gas and liquid phase applications [8].

It is well known that AC may be produced from a great set of net materials with low levels of inorganic compounds and high carbon content. Several materials of carbonaceous nature like coal, wood, nutshells, lignite and peat are utilized in the manufacture of commercial activated carbon. Gathering and handling of several crops of agricultural materials result in great amounts of agricultural by-products [9]. In the industrial operation of activated carbon, there are two strategies of activation, chemical and physical [10]. Microwave heating is more and more used in several scientific fields and technologies for an assortment of implementations, as a result of its feature of uniform heating rate and faster compared with the conventional heating process. The transfer of energy does not depend on the effluent of convection or conduction, however, it is converted into heat inside the particles via rotation of dipole and conduction of ions [11].

## 2- Experimental Work and Materials

### 2.1. Adsorbate

Amoxicillin AMX ($C_{16}H_{19}N_3C_5S$) was utilized as an adsorbate. Its molecular weight is 365.4 g/mol. A stock solution of 400 ppm was produced via dissolving a suitable amount for AMX in distilled water; it is taken

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from the Arab Company for the manufacture of antibiotics and accessories form (Akai) and thereafter diluted for the desirable concentrations.

### 2.2. Adsorbent

Raw materials Imperata is utilized for production of activated carbon, it can be re used, it is vastly obtainable, inexpensive as well as ecological amiable resources. It was washed with filtered water to eliminate dust, dehydrated by placing at 80°C for 24h as well as cut to a very small size. Then it was sifted into a regular size for (300-1000) µm. Potassium hydroxide was employed as a chemical activator.

### 2.3. Activated Carbon Production

Weighed quantity (5g) of dried Imperata was impregnated with 25 ml from potassium hydroxide solution at several impregnation ratios (IR) (0.5 – 1g/g) of 24 hr in room temperature. The specimens are put in oven at 90°C till fully desiccated thereafter the dried specimens were activated via utilizing a quartz glass column (3cm diameter x 15cm height ).

The column was closed at the bottom and open from the top end to allow the run off the vapors that result from pyrolysis. A modified microwave was utilized where a movable overlay linked to a stainless steel tube of 5mm inner diameter from that pyrolysis gases are way out. It is shown in Fig. 1.

Fig. 1 A photographic modified microwave heating oven

The column was put inside the microwave and adjusted to several irradiation power (450 – 800W) and several irradiation time (4 – 12 min) are shown in Table 1

Table 1. Activated Carbon produced via microwave heating

| Experiment Number | Irradiation Time (min) | Irradiation Power(Watt) | Impregnation ratio(gm/gm) |
|---|---|---|---|
| 1 | 6 | 450 | 0.8 |
| 2 | 8 | 450 | 0.8 |
| 3 | 10 | 450 | 0.8 |
| 4 | 12 | 450 | 0.8 |
| 5 | 8 | 450 | 0.8 |
| 6 | 8 | 600 | 0.8 |
| 7 | 8 | 700 | 0.8 |
| 8 | 8 | 800 | 0.8 |
| 9 | 8 | 450 | 0.4 |
| 10 | 8 | 450 | 0.5 |
| 11 | 8 | 450 | 0.8 |
| 12 | 8 | 450 | 1 |

After activation, the specimen was taken out from microwave to cool. To separate residue Potassium hydroxide activator, the specimen was impregnated with HCl solution 0.1M about 10 mg/l activated carbon to HCl solution ratio.

The blend was left 24 hours, and then purified and thereafter the specimen was frequently washed with filtered water until eliminating residue alkalis and organic substances and pH of filtrate reached (7-7.5).

The specimen was desiccated at 110C° until dry completely.

Then it was weighed to measure the yield of the activated carbon. The yield of activated carbon produced was determined such as [12].

$$\text{yield} = \frac{W_f}{W_o} * 100\% \quad (1)$$

Where $W_f$ and $W_o$ (g) are the weight of KAC and dried Imperata, respectively.

The AMX uptake was determined by following method: 0.1g of the produced AC, with particle size about 300 µm, was placed in 200 ml flask with 100ml for AMX sol and primary concentration (150 ppm).

Those flasks were shaken with 200 rpm to reach equilibrium. After filtration, the concentrations for AMX in filtrates were measured using UV -Visible Spectrophotometer at wavelength about 272nm. The AMX uptake, $q_e$ (mg/g), was measured as follows [13].

$$q_e = \frac{(C_o - C_e)V}{W} \quad (2)$$

Where $C_o$ initial concentration of AMX and $C_e$ (mg/l) equilibrium concentration of AMX, respectively, V is the volume of AMX solution (l), and W is the weight of activated carbon (g).

## 3- Results and Discussion

### 3.1. Influence of Irradiation Time

Affect irradiation time on the AMX uptake and AC yield were performed in constant impregnation ratio 0.8g/g at 450 W irradiation powers.

From Fig. 2, it is shown that increasing microwave irradiation time from 6 to 10 min displayed a reinforcing of AMX uptake from 0.588 to 6.18 mg/g, at 10 min.

Obviously, lengthened irradiation time leads to lowering of power or temperature, which in turn increases the interaction rates and leads to the opening of holes that caused the extension of the diameter, which will increase porosity of the hole structure [14].

Thus, additional heat handling may reproduce local hotspots, leading to the shrinkage and ablation of the activated carbon structure broadening its internal diameter.

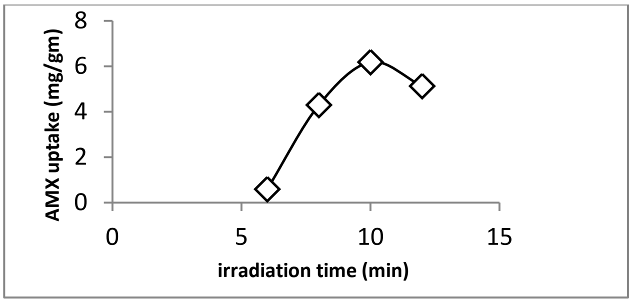


Fig. 2. Influence of irradiation time on amoxicillin uptake (at irradiation power 450 W and Impregnation ratio 0.8g/g)

Fig. 3 indicates that the yield for activated carbon decreased with increasing of irradiation time. An increase in irradiation time from 6 to 10 min at 450 W irradiation power and IR of 0.8 g/g leads to an increase the yield from7.6 % to 43.9%, when irradiation time increased from10 to 12 min the yield decreased from 43.9% to 25.6%. A decline decrease happen after 10 min, this is perhaps because of the swift expansion of volatile matter to compose stabilized compounds illustrated by Foo and Hameed [13].

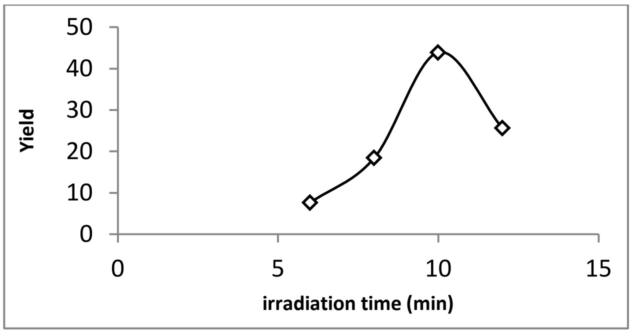


Fig. 3. Influence of irradiation time on yield (at IR 0.8 g/g and irradiation power 450 W)

3.2. Influence of Irradiation Power

Fig. 4 shows that the AMX uptake increases with irradiation power about 700 W, thereafter decreased.

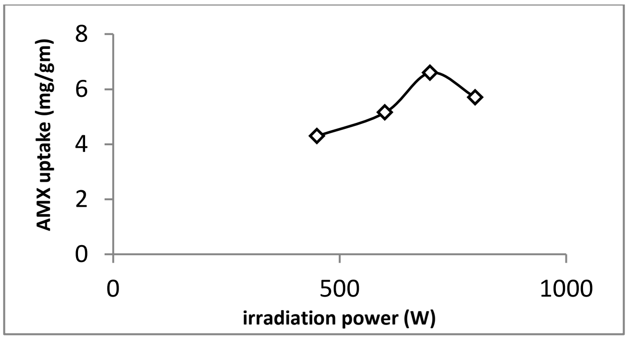


Fig. 4. Influence of irradiation power on AMX uptake (irradiation time of 8min and IR 0.8g/g)

A raise in power from 450 to 700 W at irradiation time 8min and IR of 0.8 g/g causes an increase in AMX uptake from 4.2933 to 6.5964mg/g.

Increasing power from 450 to 700 W resulted in an extravagant raise in AMX uptake; which may be attributed to the joint influence of volumetric and internal heating accountable of the extension of the activated carbon structure, build up porosity and a bigger area of the surface [14]. However, at un irradiation power behind 700 W leads to reason the extravagant demolition of hole structures; and a gradual reduction in AMX uptake [15].

Fig. 5 indicates that as the irradiation power raised from 450 to 800 W at irradiation time (8min) and IR 0.8 g/g, the yield decreased from 18.4 to 8.25%. This may be due to the wastage of the volatile material with raising power. After 700W it was observed that the yield of activated carbon decreased, but at a high irradiation power behind 700 W, gasification may be happen it causing the demolition of hole structures, thus the yield of activated carbon and the AMX uptake decreased progressively. The weight wastage of activated carbon raised proportionally to the irradiation power, at most attributable to the strong reaction in a high thermal irradiation which formidable dehydration, putrity, disintegration, and devolatilization [14].

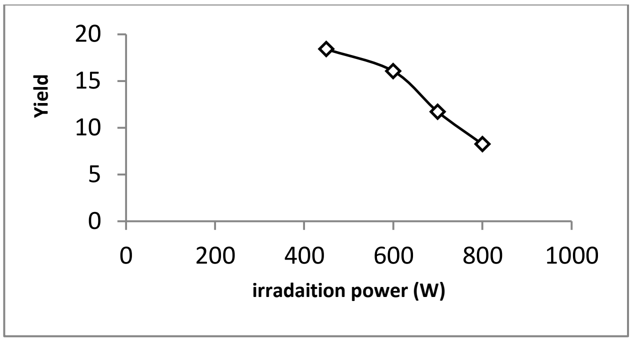


Fig. 5. Influence of irradiation power on yield (IR 0.8g/g and irradiation time 8)

3.3. Influence of Impregnation Ratio (IR)

Fig. 6 indicates the impact of IR on AMX uptake at irradiation time 8min and irradiation power 450. Increasing IR from 0.4 to 1g/g gave rise in AMX uptake from 3.7539 to 7.0371mg/g, and thereafter reduction.

The potassium ingredient created through the activation stage should spread into the inside structure of Impetara and expansion present holes. Thence, via rising the proportion of KOH to Imperata the activation stage should play a major part in hole modeling. The hole was expanded and new micropores - mesopores were created in the center pore walls, granting a rise in the total volume of pore and the area of surface. On the other hand the AMX uptake was extra improved. The increment of KOH and mineral potassium in the surface of activated carbon which leads to clogging of the holes leads to a great decrease in the attainable area. Furthermore, an extra increase in IR should increase a strong energizing reaction, it causes to burn off the activated carbon and transformation from micropores-mesopores into macropores decreasing the AMX uptake [14].

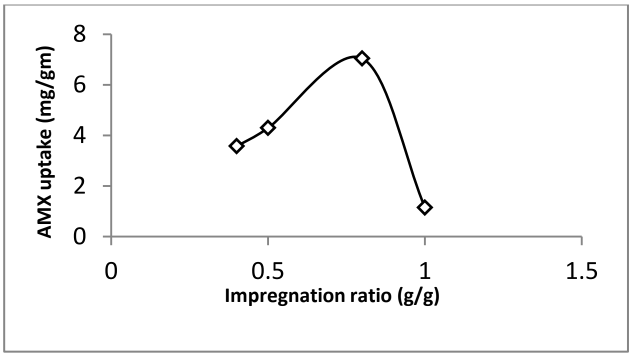


Fig. 6. Influence of IR on AMX uptake at irradiation time 8 min and irradiation power 450W)

Fig. 7 it will be plainly deduced that the yield of activated carbon decreased from 49.06 to 6.75% with raising impregnation ratio (0.4-1 g/g). Correspondingly, the increase in impregnation ratio explained a good reduction of the yield of activated carbon. This is agree with results gained by Sudaryanto, 2006 [16] for AC production by chemical activation from cassava peel with potassium hydroxide.

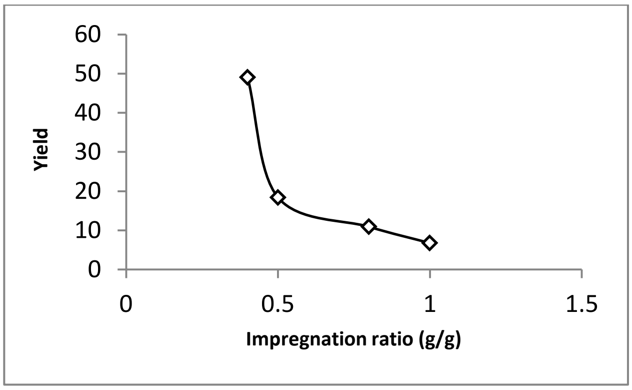


Fig. 7. Influence for impregnation ratio on the yield (irradiation power 450W, irradiation time8 min)

3.4. Characteristic for AC

The most features for AC are total pore volume and specific surface area. The total volumes of pore as well as the area of surface of activated carbon produce at better conditions are 0.3027 $m^3/g$ and 552.7638$m^2/g$, respectively.

Those results were best compared with of that prepared activated carbon via Huda (2016), the area of surface of activated carbon produce from scrap tires via utilizing KOH as activation reagent were 374.594 $m^2/g$. Table 2 shows the comparison of structure textural of prepared activated carbon with carbons from various raw materials.

From this table it can be shown that the Imperata is known as a suitable raw material for AC with high volume of pores and the high surface area.

The chemical energizing operation has participation in the high area of surface and the volume of pore of the activated carbon produced.

Table 2. Compare between pore structures for activated Carbons produced from several raw materials

| Raw materials | Activation | BET surface area $m^2/g$ | Reference |
|---|---|---|---|
| Imperata | KOH | 552.7638 | This study |
| Cotton stalk | KOH | 729.33 | Deng, et. al.2010 |
| Scrap tires | KOH | 374.594 | Huda 2016 |
| Oil palm stone | $Co_2$ | 320 | Guo and Lua 2000 |
| Bamboo charcoal | $Co_2$ | 263 | Wang et., 2012 |

3.5. Surface Morphology

The SEM images of Imperata and KAC are displayed in Fig. 8 and Fig. 9. It will be noted that the Imperata surface is planar, intensive, shrunken and plugged by deposited substance. However, the microwave irradiation specimen gave a good expanded and regular surface formation a organized hole structure. The development for porosity of AC by KOH activation. It was supposed which KOH was decreased to compose mineral potassium through the carbonization operation. The interaction among the carbon and KOH happen as follows:

$$2C + 6KOH \rightarrow 3H_2 + 2K_2CO_3 + 2K$$

$K_2CO_3$ was decreased via carbons to compose CO, K, $CO_2$, and $K_2O$ so that many holes were formed:

$$2C + K_2CO_3 \rightarrow 3CO + 2K$$

$$K_2CO_3 \rightarrow CO_2 + K_2O$$

It was supposed that mineral potassium formed through the gasification operation would spread into the interior activated carbon matrix structure expansion the existing holes and formed new holes [17]. Fig. 9 also indicate which the produced activated carbon surface comprise several chambers that were created from the vaporization of saturated KOH leaving the gab already taken via the agent. These gabs supply channels to the micropores and mesopores internal an activated carbon particle.

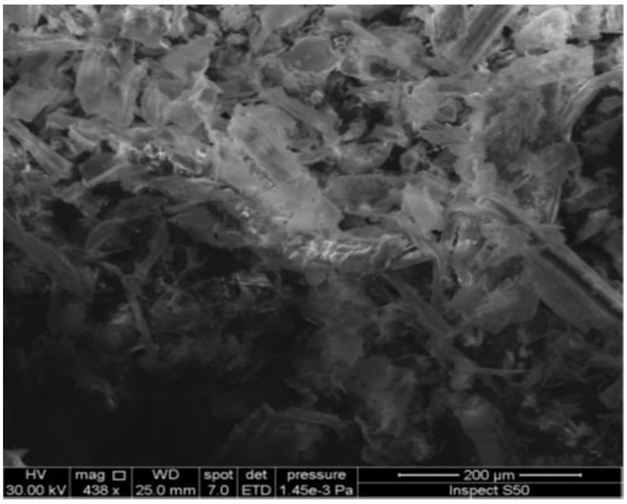


Fig. 8. SEM micrographs(200μm) of Imperata

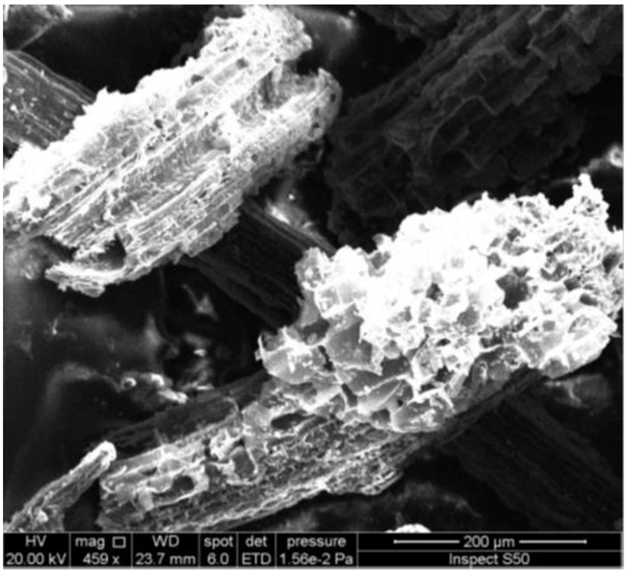

Fig. 9. SEM micrographs (200 µm) of AC

## 4- Conclusion

From this work, it can be concluded that the activated carbon with the area of surface 552.7638m$^2$/g as well as the volume of pore 0.3027m$^3$/g was produced from Imperata by potassium hydroxide microwave heating. AMX uptake of 12.456( mg solution /g adsorbent ) with the yield of activated carbon 14.821% were attained in the best condition of irradiation time(10min),irradiation power(700W) as well as IR (0.8g/g).The yield of activated carbon reduced with raising irradiation time, irradiation power and IR . The AMX uptake is directly proportional to irradiation time, irradiation power and IR.

## References

[1] Chatterjee, R., (2008)," Fresh produce from wastewater", Environ. Sci. Technol, 42 (21), 7732-2008.

[2] ElSayed, E.M., Prasher, S.O. and Patel, R.M., (2013)," Effect of nonionic surfactant Brij 35 on the fate and transport of oxytetra cycline antibiotic in soil", J. Environ. Manage, 116, 125-134

[3] Ghauch, A., Tuqan., A., and Abou Assi, H., (2009)," Antibiotic removal from water: Elimination of amoxicillin and ampicillin by microscale and nanoscale iron particles Environ". Pollut, 157,1626–1635

[4] Bajpai, S.K., Bajpai, M. and Rai, N., (2012)," Sorptive removal of ciprofloxacin hydrochloride from simulated wastewater using sawdust: Kinetic study and effect of pH Afr. j, 38 N° 5", 673-682

[5] Zha S.X., Zhou Y., Jin X. and Chen Z., 2013,"The removal of amoxicillin from wastewater using organobentonite",Journal of Environmental Management 129 ,569,576.

[6] Aksu Z., (2005), Application of biosorption for the removal of organic pollutants: a review, Process Biochemistry, 40, 997-1026.

[7] A .Mohammad-Khah and R. Ansari, (2009)," Activated Charcoal: Preparation, characterization and Applications", A review article International Journal of ChemTech Research CODEN( USA): IJCRGG Vol.1, No.4, 859-864

[8] Bandosz T. J., 2006,"Activated carbon surfaces in environmental remediation", 7, T.J. Bandosz, ed, Oxford, United Kingdom ,Elsevier.

[9] Ioannidou O., and Zabaniotou A., (2007), "Agricultural residues as precursors for activated carbon production –A review" , Renewable and Sustainable Energy Review, 11, (2007),1966,(2005).

[10] Bansal R.C., Donnet J.B and Stoeckli H.F., (1988)"Active Carbon", Marcel Dekker, 9-28285-537-6 New York , United States.

[11] Duan Xin-hui , C. Srinivasakannan , Peng Jin-hui , Zhang Li-bo, Zhang Zheng-yong, (2011),"Preparation of activated carbon from Jatropha hull with microwave heating: Optimization using response surface methodology", Fuel Processing Technology 92 394–400.

[12] Marwa Ahmed Chaied., (2015)," Microwave-Assisted Preparation of Activated Carbon from Phragmites (Reed) for Antibiotics Adsorption", A thesis Submitted to the College of Engineering of the University of Baghdad in Partial Fulfillment of the Requirements for the Degree of Master of Science in Chemical Engineering

[13] Foo K.Y. and Hameed B.H.( 2012c)," Porous structure and adsorptive properties of pineapple peel based activated carbons prepared via microwave assisted KOH and K2CO3 activation", Microporous and Mesoporous Materials ,148 , 191–195

[14] Foo K.Y. and Hameed B.H. (2012a)," Potential of jackfruit peel as a precursor for activated carbon prepared by microwave induced NaOH activation", Bioresource Technology,112 , 143–150.

[15] Foo K.Y. and Hameed B.H., (2012b)" Textural porosity, surface chemistry and adsorptive properties of durian shell derived activated carbon prepared by microwave assisted NaOH activation", Chemical Engineering Journal,187, 53– 62.

[16] Sudarynoto Y., Hartono S. B., Irawaty W. and Ismadji S., (2006),"High Surface Area Activated Carbon Prepared from cassava peel by chemical activation", Bioresour. Technol., 97,734-739.

[17] Deng H. , Li G.X. , Yang H.B. , Tang J.P., and Tang J.Y., (2010a), "Preparation of activated carbons from cotton stalk by microwave assisted KOH and K2CO3 activation", Journal of Chemical Engineering,163,373–381.