



Preparation and Characterization of Activated Carbon for Adsorption of Fluoroquinolones Antibiotics

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ABSTRACT

In this research, the preparation of a chemically activated carbon from date stones by using electric and microwave assisted K_2CO_3 activation was studied. The effect of radiation power, radiation time, and impregnation ratio on the yield and Iodine number on the activated carbons was investigated. The activated carbon characterizations were examined by its surface area, pore structure analysis, bulk density, moisture content, ash content, iodine number, FTIR, and scanning electron microscopy (SEM). The adsorption capacity was also studied by adsorption of fluoroquinolones antibiotics, CIP, NOR, and LEVO, by the prepared activated carbon.

Key words: Activated carbon, Date stones, fluoroquinolones antibiotics, Microwave.

تحضير وتوصيف الكربون المنشط لأمتزاز ملوثات الفلوروكوينولونس

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باحث

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الخلاصة

تم تحضير الكربون المنشط من نوى التمر بأستخدام الفرن الكهربائي و الميكرويف و باستعمال كاربونات البوتاسيوم كمادة منشطة.

تمت دراسة تأثير طاقة الاشعاع و زمن الاشعاع و نسبة التغطيس على انتاج الكربون المنشط و قيم رقم الايودين للكربون المنشط. خصائص الكربون المنشط التي تمت دراستها متمثلة بالمساحة السطحية و حجم المسامات و الكثافة و محتوى الرطوبة و محتوى الرماد و رقم الايودين و تحليل الطيفي بالأشعة تحت الحمراء و صورة المجهر الألكتروني. تم قياس سعة امتزاز الكربون المنشط من خلال امتزاز المضاد الحيوي الفلوروكوينولونس (السبروفلوكسن والنورفلوكسن و الليفوفلوكسن).

الكلمات الرئيسية : الكربون المنشط, نوى التمر, مضادات الفلوروكوينولونس, مايكرويف.



1. INTRODUCTION

Many precursors can be used successfully for the preparation of low cost activated carbon such as sugar beet bagasse **Demiral, and Gündüzoğlu, 2010**, coal **Teng, et al., 1998**, fruit peel **Kartika, et al., 2008**, Waste tea **Yagmu, et al., 2008**, olive stone **Blázquez, et al., 2005**, date stone **Foo, and Hameed, 2011**, and rice husk **Tyagi, et al., 2011**. Two processes are used for preparation of activated carbon: physical activation and chemical activation. Physical activation includes carbonization of carbonaceous content and then activation of resulting char by activation agent such as steam or CO₂ **Bouchelta, et al., 2008**. Chemical activation involves the impregnation of raw material by an activation agent such as KOH, H₃PO₄, ZnCl₂, etc. and then heated in an inert atmosphere. Chemical activation is more preferred than physical activation because of the higher yield, shorter time, simplicity, and lower temperature needed for activating material **Li, et al., 2010**. The preparation of activated carbon by microwave heating has many advantages. The important one is the reduction in activation time which results in lower energy consumption. There are many applications in using microwave heating for prepared activated carbon by KOH activation **Foo, and Hameed, 2011**, ZnCl₂ activation **Wang, et al., 2009**, phosphoric acid activation **Liu, et al., 2010**.

The fluoroquinolones are a series of synthetic antimicrobial agents which have a broad spectrum of activity against organisms **King, and Lan, 1986**. It is used in the treatment of several infections in human and animals. Hospitals and drug manufactures are the main sources of wastewater containing drug contamination. Also in wastewater treatment plants, antibiotics are partially eliminated and their main amount kept to the aquatic ecosystem. The presence of drug antibiotics in surface water and wastewater, even at low concentrations is considered an environmental hazard and can cause antibiotic resistance in bacteria **Bhandari, et al., 2008**.

In previous century, many techniques were enhanced to get rid of contaminants from the wastewaters, where they present at high concentrations in order of $\mu\text{g L}^{-1}$ to mg L^{-1} . The best drinking water treatment and wastewater treatments were found on coagulation, filtration, flocculation, biological degradation, and sedimentation methods that exposed ineffective removing for drugs counting antibiotics **Chayid and Ahmed, 2015**.

The aim of this work is to prepare a low cost activated carbon from natural precursors (date stones) for the adsorption of fluoroquinolones antibiotics from aqueous solution. The effect of impregnation ratio, radiation power, and radiation time are also investigated to obtain a high yield and adsorption capacity.

2. MATERIALS AND METHODS

2.1 Materials

In this study, date stones were used as precursor for the preparation of activated carbon. Firstly the stones were washed with tap water to get rid of impurities, washed with distilled water, dried in oven for 24h at 110°C, and crushed using disk mill, with the average particle size of 1 to 2 mm.



In this research, two chemical activators for preparation two types of activated carbon, K_2CO_3 (provided by Didactic Company, Espuma) of purity 99.9%. Three types of fluoroquinolones antibiotics were used: CIP of purity 99.9% are provided by Nanjing Huaxin Biofarm. Company Ltd., China, and NOR with purity 99.9% are provided by Ajanta Pharma Limited Company, India.

2.2 Preparation and Characterization of Activated Carbon

20 g of dried date stones was put in a stainless steel reactor (3 cm diameter * 15 cm length). The reactor was closed in one end and the other end had a removable cover which contains a 1mm hole in the middle to allow the escaping of the pyrolysis gases. The reactor is put in the middle of an electric furnace and heated at 500° C for 1 h. After that the reactor was taken out of the furnace and allowed to cool. Each 2g of the above sample was mixed with 10ml of K_2CO_3 solution with impregnation ratio (0.4 to 1.5 g/g) for 24h at room temperature. Then, these samples were dried in an oven at 110°C until well dried and stored in desiccators. In the activation step of dried impregnated samples, quartz glass reactor (3 cm diameter and 13 cm length) was used. The reactor closed at one end and the other end was opened to a stainless steel pipe with 5mm inside diameter. The reactor was put in a modified microwave heating oven (MM717CPJ, China), **Fig.1**, and run at different radiation power for different radiation time. At the end of activation step the samples were withdrawn from the oven and allowed to cool. After that the samples were soaked with 0.1 M HCL solution (10ml/g liquid to solid ratio). The mixture left at room temperature for 24 h and then filtered and washed with distilled water until the pH of solution reaches (6.5-7). Finally dried for 24h at 110°C and then the samples were weighed to calculate the yield of product and finally the activated carbon was stored in tightly closed bottles. The yield of prepared activated carbon is equal the ratio of final weight of product to the weight of precursor initially used which is based on this equation:

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

where W_f and W_o are the weight of activated carbon product g and the weight of date stone, g, respectively.

The characterization of activated carbon was represented by surface area, bulk density, ash content, moisture content, iodine number.

A set of batch mode experiments were carried out to study the uptakes of each antibiotics. 0.1g of prepared activated carbon was mixed with 100 ml samples of CIP, NOR, and LEVO with initial concentration 150 mg/l. The mixtures were shaken at 200 rpm for 24hours, and then the mixtures filtered. The concentrations of each antibiotic in filtrate was analyzed using UV- Visible Spectrophotometer with wave length 274, 272, and 290nm for CIP, NOR, and LEVO respectively. The adsorbed capacity or uptake of antibiotics q_t was calculated by:



$$qt = \frac{(C_0 - C_e)V}{W} \quad (2)$$

where C_e (mg/l) is the concentration of antibiotics at equilibrium, mg/l, V (ml) is volume of antibiotic solutions, and W (mg) is weight of activated carbon.

2.2.1 Bulk density

Apparent or bulk density was determined by this simple method, a 10 ml cylinder filled to a specified volume with prepared activated carbon that had been dried in an oven at 80°C for 24 h **Ahmedna, et al., 1997**, and then calculated as follows:

$$\text{bulk density} = \frac{W_c}{V_c} \quad (3)$$

where W_c is the weight of dried carbon (g), and V_c is the cylinder volume (ml) filled with dried carbon.

2.2.2 Ash content

The ash content was calculated as follows: 0.1 g of activated carbon with particle size of 250 μ m was dried at 80°C for 24h and put into weight ceramic crucibles. The sample was heated in an electric furnace at 650°C for 3h. Then it was left to cool to room temperature and then weighed. Then by using this equation, the percent of ash was determined **ASTM Standard, 2000**.

$$\text{ash}\% = \frac{W_{s3} - W_{s2}}{W_{s1}} * 100 \quad (4)$$

where W_{s1} is the weight of activated carbon (g), W_{s2} is the weight of ceramic crucible (g), and W_{s3} is the weight of ceramic crucible containing ash (g).

2.2.3 Moisture content

Oven drying method was used to calculate moisture content **Adekola, and Adegoke, 2005**. 0.1 g of prepared activated carbon with particle size 250 μ m was put into weighed ceramic crucible. The sample were dried at 110°C to constant weight, and then cooled to room temperature and weight.

$$\text{moisture}\% = \frac{W_{m3} - W_{m2}}{W_{m1}} \quad (5)$$

where W_{m1} is the weight of activated carbon sample (g), W_{m2} is weight of ceramic crucible with dried sample (g), and W_{m3} is the weight of ceramic crucible with original activated carbon used (g).

2.2.4 Iodine number

It is a measure of micropore content of activated carbon. It was calculated by standard method **Lubrizon Standard Test Method, 2006**. 10ml of 0.1 N iodine solution in a conical flask was titrated with 0.1N sodium thiosulfate solution with two drops of 0.1N starch solution as indicator, until the color of solution become colorless. 0.1g of activated carbon was added to 15ml of 0.1N iodine solution, and then the solution was shaken for 4minutes and then filtered. 10 ml of the filtered solution was titrated with 0.1N sodium thiosulfate in the presence of two drops of 0.1N starch solution as indicator. Then by using this equation:

$$\text{Iodine number} = \frac{(V_b - V_s) \cdot N \cdot (126.9) \cdot \left(\frac{15}{10}\right)}{M} \quad (6)$$

where V_b (ml) is the volume of blank sodium thiosulfate solution, V_s (ml) is the volume of titrated sodium thiosulfate solution, N (mol /L) is the normality of sodium thiosulfate solution, and M (g) is the mass of activated carbon used.

3. RESULTS AND DISCUSSION

3.1 Characteristics of Activated Carbon

The characteristics of prepared activated carbon were shown in Table 1. The surface area is the most important characteristics, 852 m²/g for activated carbon impregnated with K₂CO₃. This result may be in agreement with that prepared by **Foo, and Hameed, 2011**, who prepared an activated carbon from date stones by microwave induced chemical activation with surface area of 856 m²/g, **Bamufleh, 2009**, prepared activated carbon from date stones by chemical activation with ZnCl₂ with surface area 802- 1270 m²/g, and **Hameed, et al., 2009**, who showed a surface area of activated carbon by chemical activation of KOH of date stones was 763.4 m²/g. This may be due to the ability of K₂CO₃ to produce an activated carbon which had high micropore content.

3.2 SEM Image

The scanning electron microscope image in **Fig.2** of activated carbon with optimum operating conditions (t=8min, P=540W, IR=0.8g/g) for activated carbon activated by K₂CO₃. From SEM image, there were clear changes in the surface of date stones particles before and after chemical activation. It can be found that the surface of date stone is very flat and dense with loss of any pore, while the microwave irradiation forms a lot of pores structure with thin boundary.

3.3 FT-IR Analysis

Fourier transform infrared spectroscopy (FTIR) was used to show the carbons of any adsorbent in the range of (400-4000) cm⁻¹ wave number to get information about functional group and chemical

structure **Speight James, 2005**. **Fig. 3** shows the FT-IR analysis of activated carbon, from this figure the band between the range (3400-3500) cm^{-1} for prepared activated carbon is assigned to the O-H hydroxyl groups, the hydrogen bonding may be because of the adsorbed water from the surroundings **Vinke, et al., 1994**. The double peak at (2850–2950) cm^{-1} may be due to the C-H stretching vibrations **Jia, and Thomas, 2006**. The band at (1050–1200) cm^{-1} region is probably assigned to C-O stretching in phenols and alcohols **Phussadee, et al., 2008**. Also the peak at 1650 is the (-COOH and -COOCH₃) stretching vibration of nonionic carboxyl group, this may be due to carboxylic acids or esters **Li, et al., 2007**. The peak near 2360 cm^{-1} may be due to the band consideration of CO₂ **Gascoin, et al., 2008**. The bond occurring near 1400 cm^{-1} due to C=O stretching, C–O stretching in carboxylate moieties and carboxylic groups **Lua, et al., 2005**. The bands near 1750 cm^{-1} C=O group in configurations such as Quinone, lactones, and COOH groups which indicates the presence of ester and acetyl group that present in hemicelluloses **Chandrasekaran, et al., 2014**.

4. IODINE NUMBER, YIELD AND UPTAKE OF ACTIVATED CARBON

4.1 Effect of Radiation Time

The relation between the yield and time is shown in **Fig. 4**. From this figure the yield of activated carbon decreases when time increases from 4 to 12 minutes. After 10 min, a slow decrease occurred in yield during activation because of rapid formation of volatile material which form stable structure **Foo, and Hameed, 2012a**.

Fig. 5 also shows the relation between iodine No. and time. The iodine no. range 631.89 to 854.91 for prepared activated carbon. The optimum time of 8 min gave a higher iodine no. for activated carbons. It's higher than prepared by **Haimour, and Emeish, 2006**, 495mg/g for activated carbon that prepared from date stones and phosphoric acid by chemical activation. This is may be because of the ability of K₂CO₃ to form high micropore content in the active carbon.

The adsorption of fluoroquinolones antibiotics, CIP, NOR, and LEVO by activated carbon are shown in **Fig. 6**, where the uptake of all antibiotics increase when time increases and reach maximum at time of 8 min, then decreases until the time increase. The maximum uptake of fluoroquinolones antibiotics were 97.4, 93.9, 99.8 mg/g for CIP, NOR, and LEVO respectively. In this study the activation time of 8 min may be considered as a best time for preparation of both type of activated carbon. **Ahmed and Thydan, 2013**, showed that 8 min is the best time for preparation of activated carbon prepared from albizia lebbek seed pods by microwave activation.

4.2 Effect of Radiation Power

The yield of activated carbon versus radiation power is shown in **Fig. 7**. From this figure it can be see that the yield decreased with the increase in power from 380-700W. This decrease may be due to loss in volatile materials with high power value. Also, after 620W a steep decrease in yield

occurs because of formation of stable structure. **Fig. 8** also shows the relation between Iodine no. and radiation power. 540W power gave higher iodine no. for activated carbon activated by K_2CO_3 . The antibiotics uptake, **Fig. 9**, also shows the similar behavior as above. Its increase with increase in radiation power, reached maximum at 540 W for activated carbon promoted by K_2CO_3 and then increased with increasing radiation power. The maximum uptake of fluoroquinolones antibiotics were 97.4, 93.9, 99.8 mg/g for CIP, NOR, and LEVO respectively. This decrease at high power may be due to sintering effect followed by the shrinkage of the char, then realignment in the structure of the carbon that lead to reduce the pore areas and then volume **Foo, and Hameed, 2012b**.

4.3 Effect of impregnation ratio

Impregnation ratio of activator/precursor is an important factor in the chemical activation process **Ahmadpour, and Do, 1996**. The relation between yield and impregnation ratio is shown in **Fig. 10**. It can be seen that, as the impregnation ratio increases, the yield decreases. This is may be because of rapid removal of tar component from the pore. After 1g/g, a steep decrease in yield occurs due to stable structure. **Fig. 11** shows the relation between iodine no. and impregnation ratio. 0.8g/g ratio gave a higher iodine no. for activated carbon. The uptake of fluoroquinolones antibiotics also increases with increasing impregnation ratio, and reached maximum uptake at 0.8 g/g then decreased as shown in **Fig. 12**. The maximum fluoroquinolones antibiotics uptake were 97.4, 93.3, 99.8 g/g for CIP, NOR, and LEVO respectively. **Foo, and Hameed, 2012c** showed the same behavior, where the yield and uptake increased when increasing the impregnation ratio (0.25 to 2) g/g , reaching a maximum value, then decreased for their prepared activated carbon from empty fruit bunch waste by microwave heating.

4. CONCLUSIONS

A simple method is used in this work for preparation of activated carbon with high surface area and heterogeneous structure from date stones by conventional and K_2CO_3 microwave heating activation. The effect of different parameter such as radiation time, radiation power and impregnation ratio on the yield, iodine number, and fluoroquinolones antibiotics adsorption were studied. The maximum yield were 44%, 37.8%, 40% for optimum conditions of 8 min. radiation time, 540 W radiation power and 0.8 g/g impregnation ratio. The iodine no. for optimum condition was 854.91. The surface area is 852 m²/g and pore volume 0.761cm³/g. A good adsorption of fluoroquinolones antibiotics was obtained by the prepared activated carbon. CIP uptake of 97.4 mg/g, NOR uptake of 93.9 mg/g and LEVO uptake of 99.8 mg/g for AC- K_2CO_3 that obtained at optimum conditions.



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NOMENCLATURE

Ce	Equilibrium concentration of each antibiotics, mg/l
C _o	Initial concentration of each antibiotics, mg/l
M	Mass of activated carbon, mg
N1	normality of sodium thiosulfate solution, mol/l
qt	Uptake of antibiotic at equilibrium, mg/g
V	Volume of antibiotic solutions, ml
V _b	volume of blank sodium thiosulfate solution, ml
V _c	Cylinder volume, ml
V _s	volume of titrated sodium thiosulfate solution, ml
W _c	Weight of dried carbon, mg
W _f	Weight of activated carbon product, mg
W _{m1}	weight of activated carbon sample, g
W _{m2}	weight of ceramic crucible, g
W _{m3}	weight of ceramic crucible with original activated carbon used, g
W _o	Weight of date stones, g

ABBREVIATIONS

CIP	Ciprofloxacin
LEVO	Levofloxacin
NOR	Norfloxacin

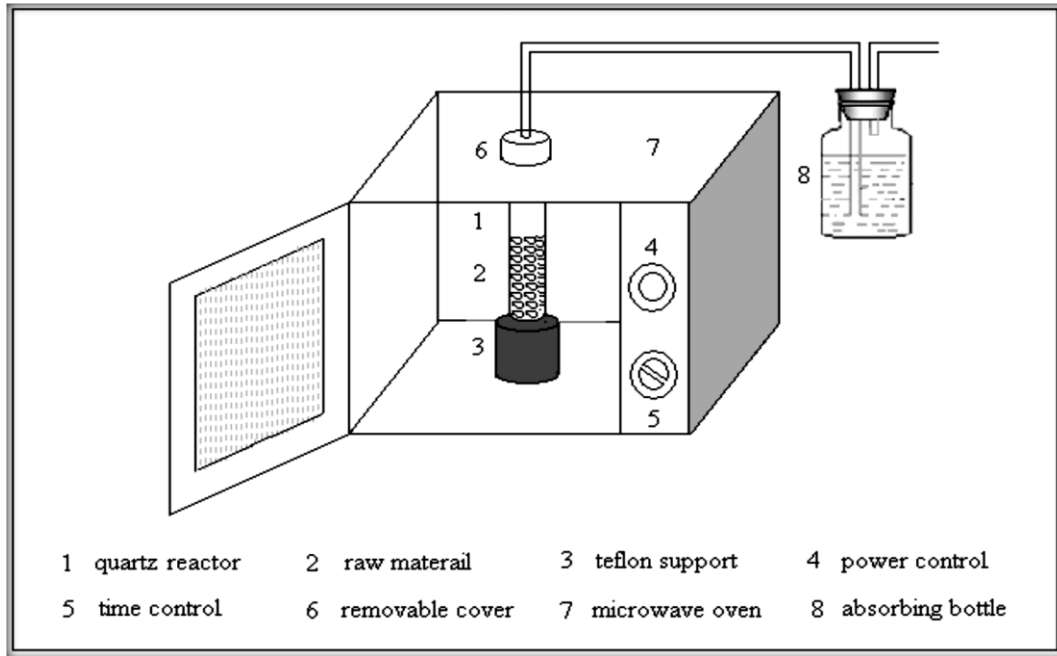


Figure 1, Schematic diagram of microwave unit for preparation of activated carbon.

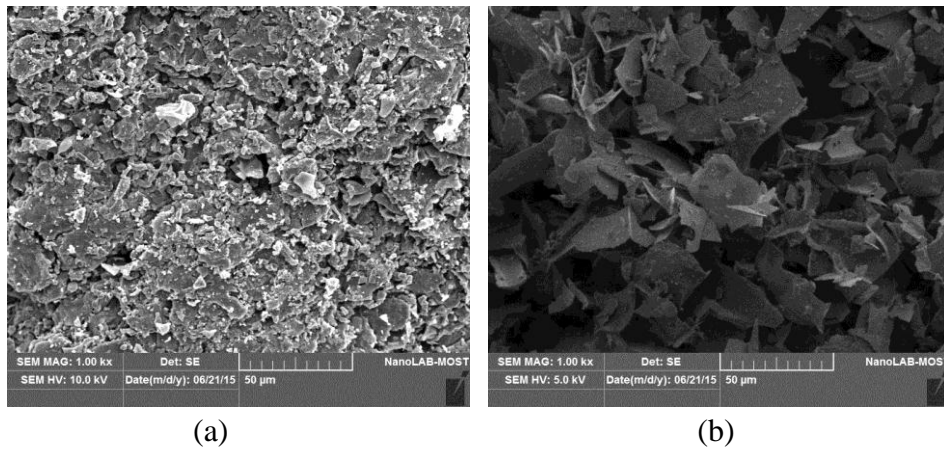


Figure 2. SEM images of (a) date stone and (b) activated carbon, respectively.

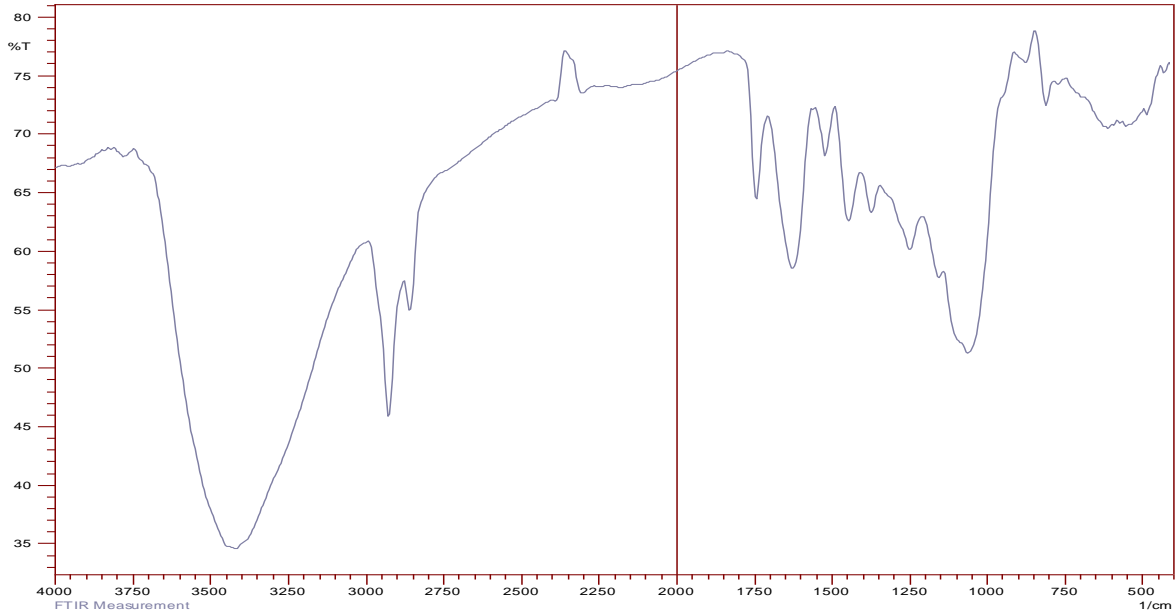


Figure 3. Activated carbon activated by K_2CO_3

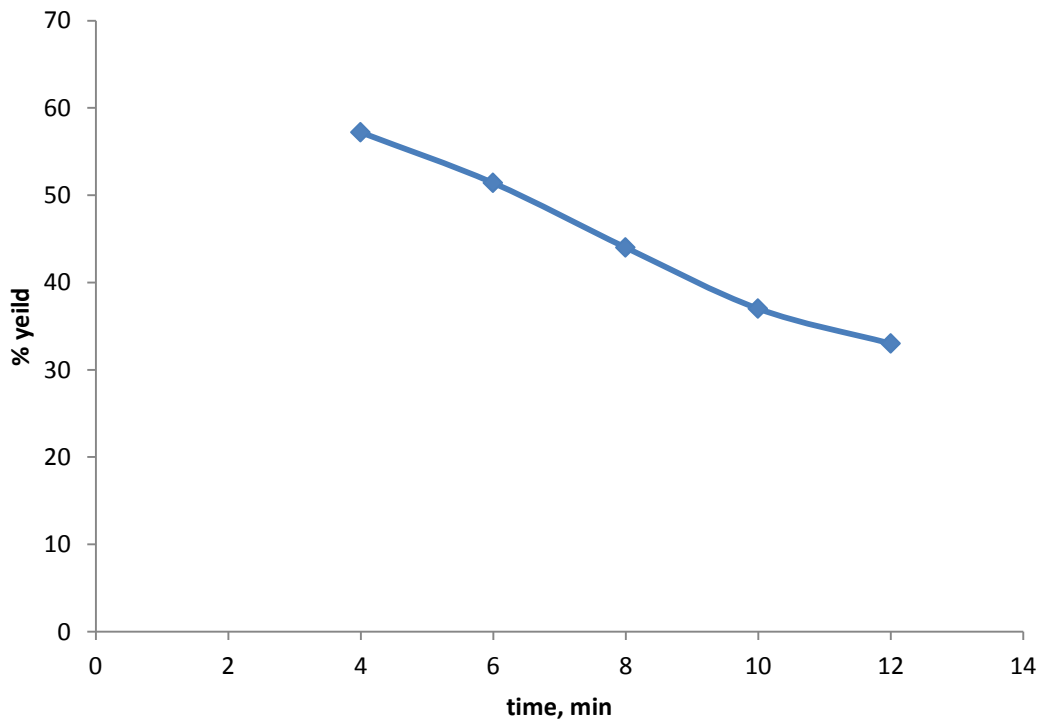


Figure 4. Effect of time on the yield of activated carbon.

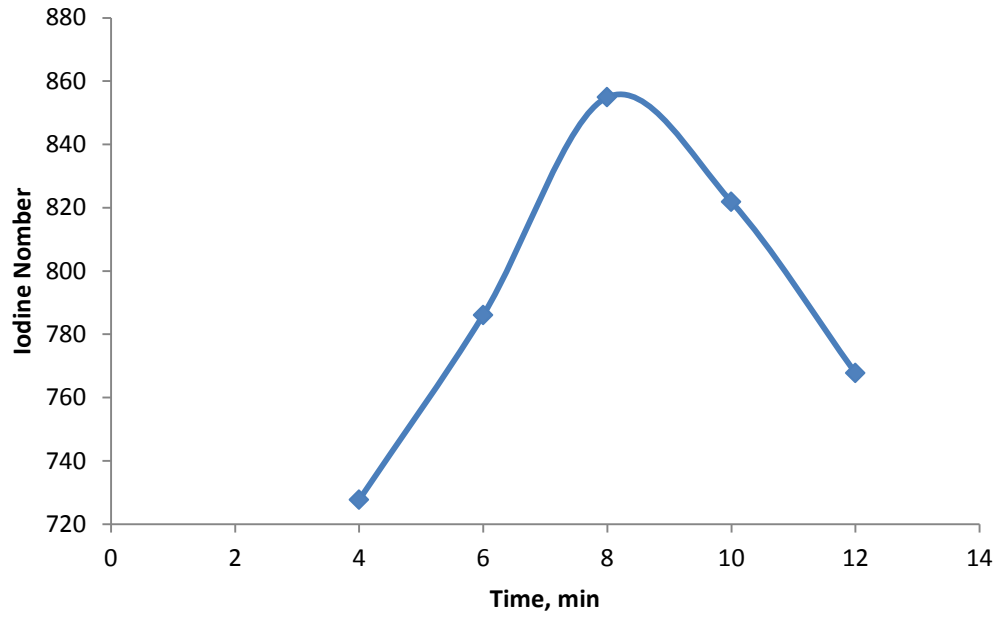


Figure 5. Effect of time on Iodine number

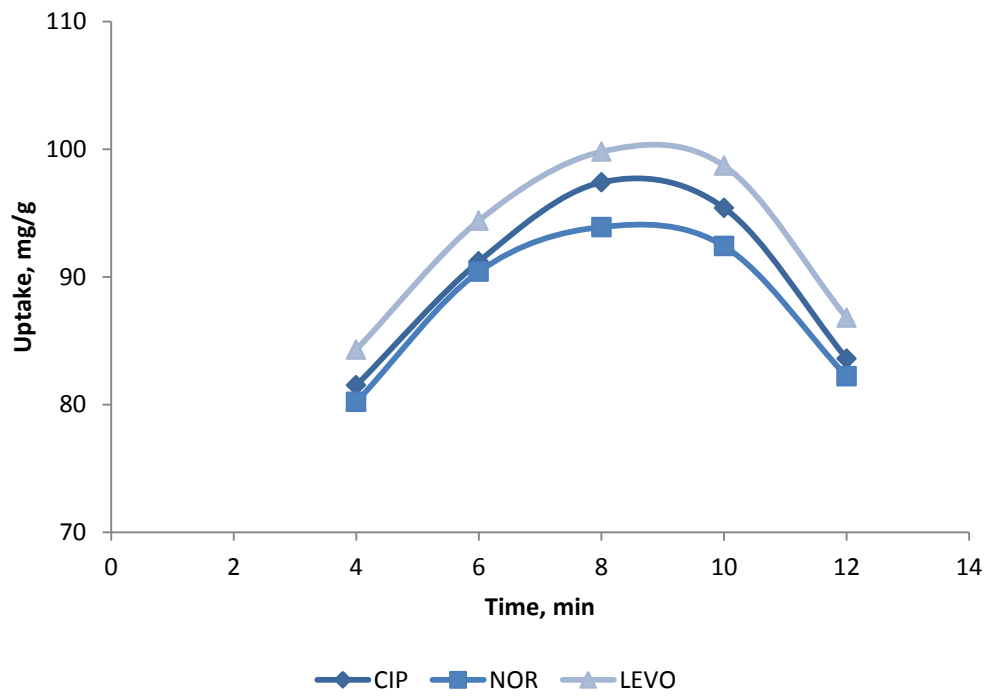


Figure 6. Effect of time on the uptake of antibiotics.

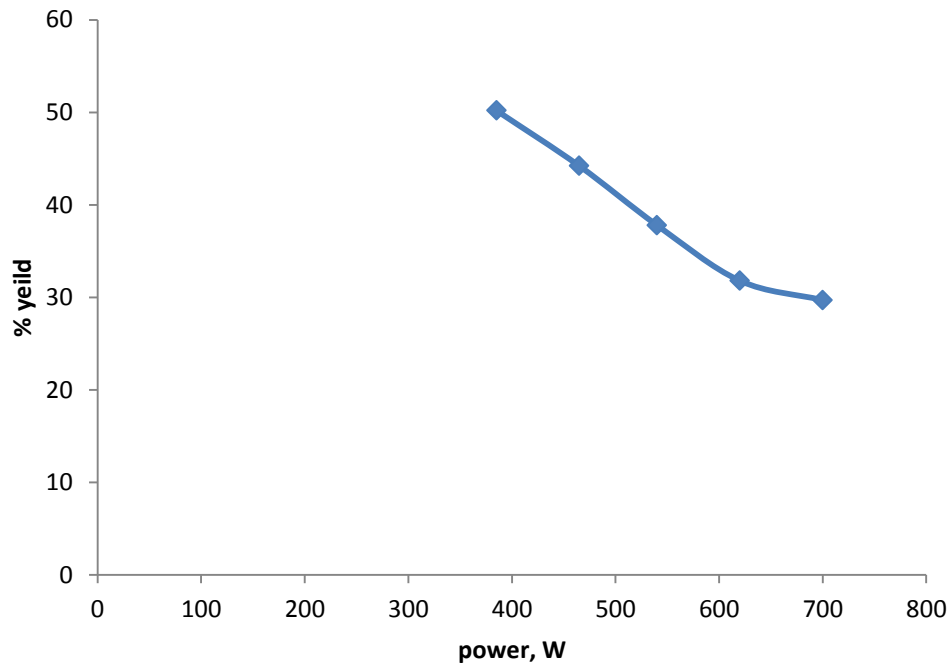


Figure 7. Effect of radiation power on the yield of activated carbon.

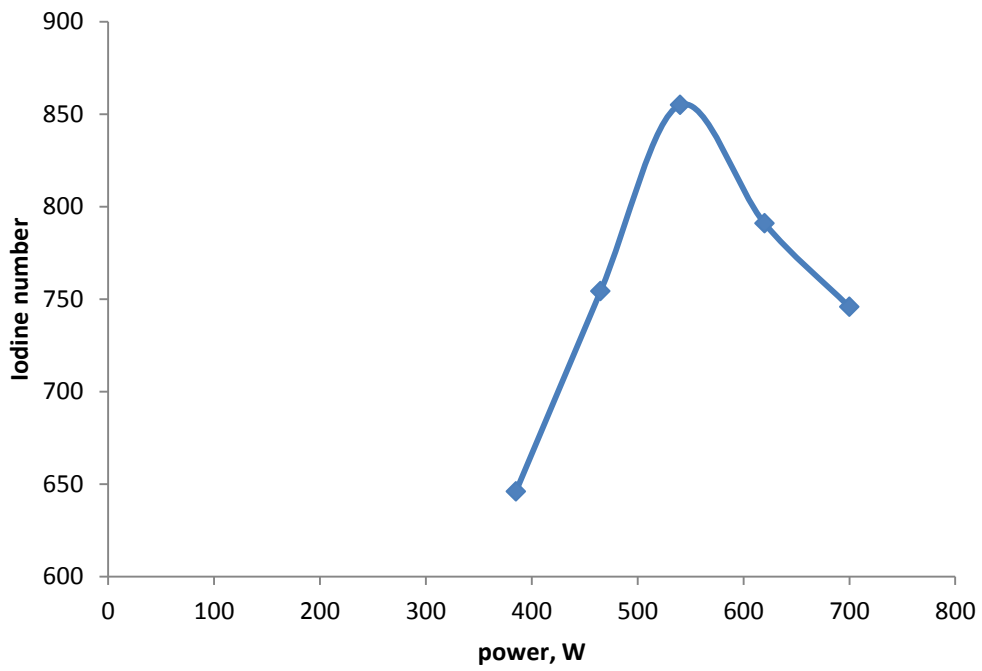


Figure 8. Effect of radiation power on Iodine number.

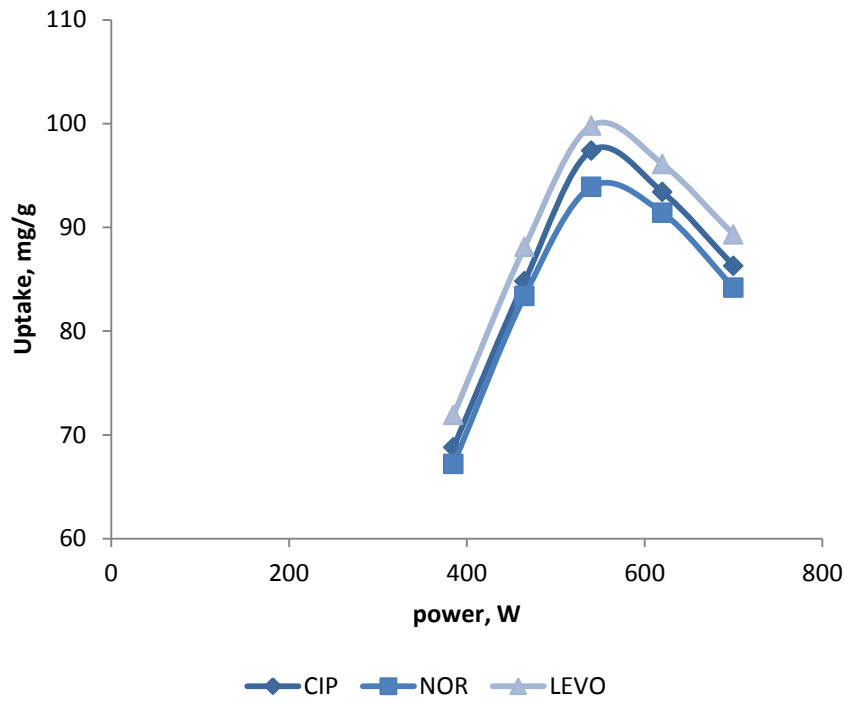


Figure 9. Effect of radiation power on the uptake of antibiotics.

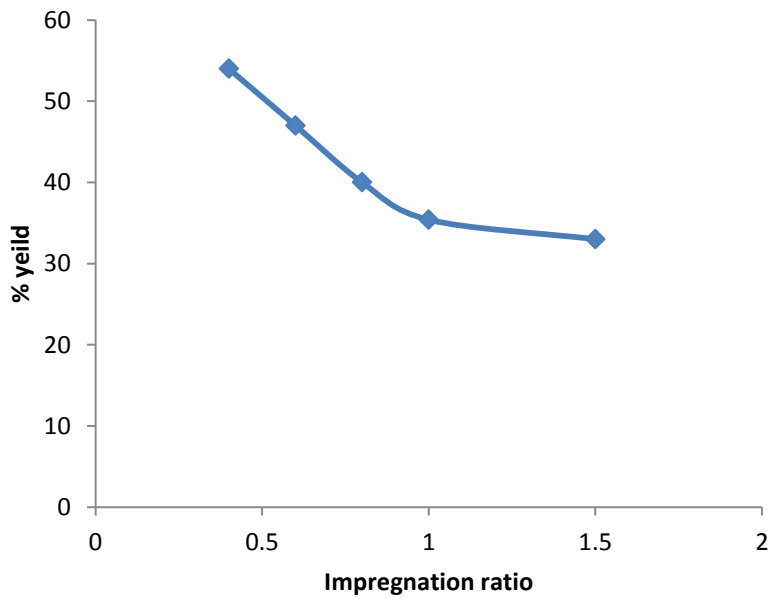


Figure 10. Effect of impregnation ratio on the yield of activated carbon.

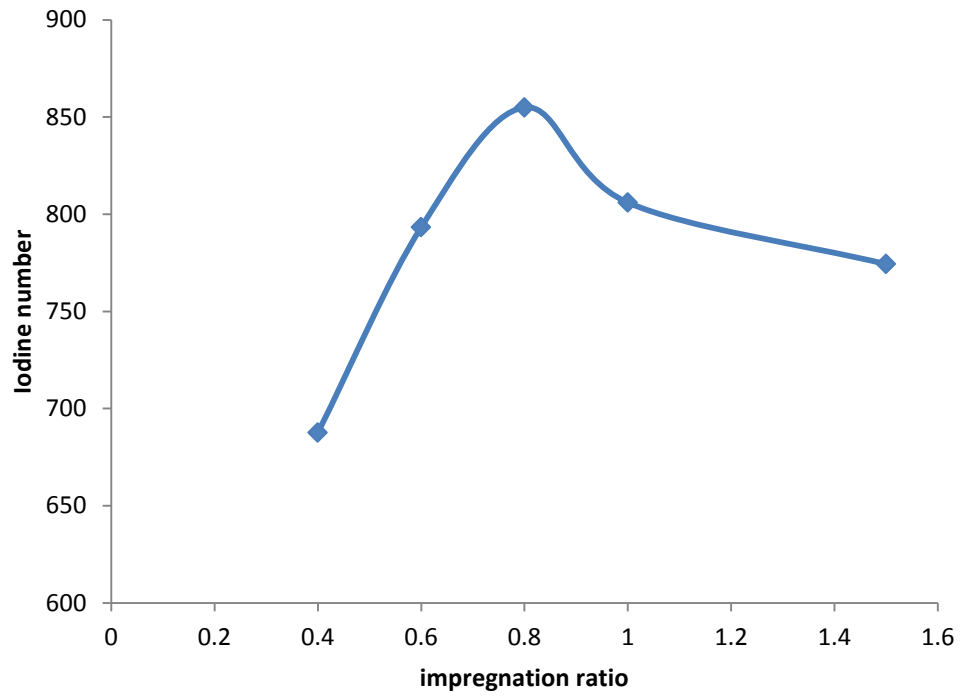


Figure 11. Effect of impregnation ratio on iodine number.

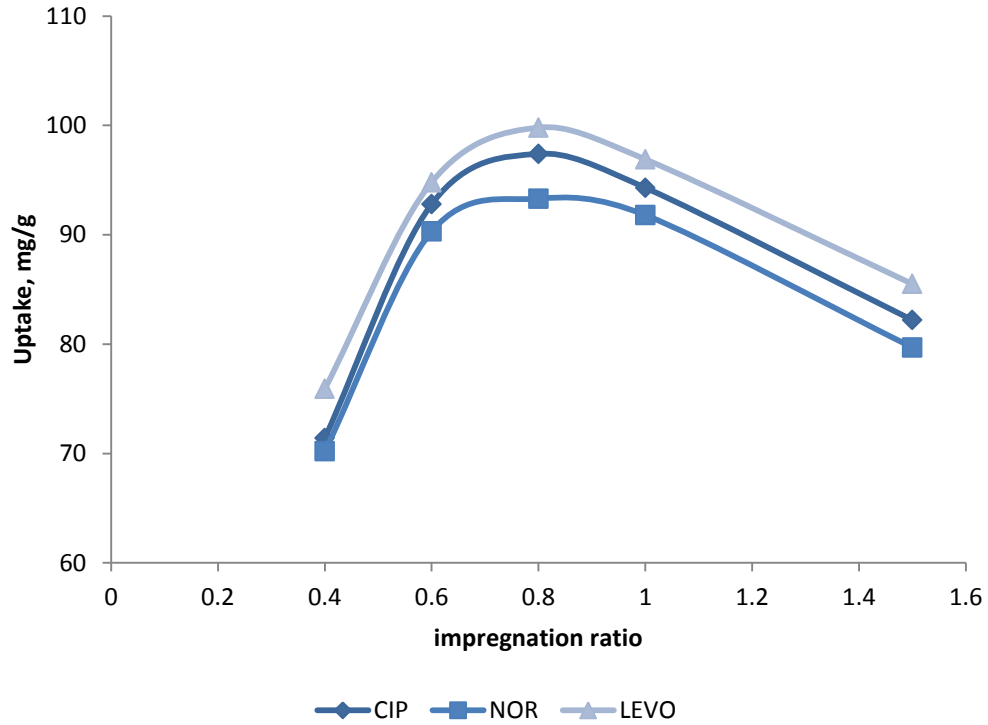


Figure 12. Effect of impregnation ratio on the uptake of antibiotics.

Table 1. Characterization of activated carbon.

Characteristics	AC-K ₂ CO ₃
Surface area, m ² /g	852
Pore volume, cm ³ /g	0.671
Ash content, %	3.88
Moisture content, %	4.9
Bulk density, g/ml	0.352