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Removal of Amoxicillin from Water by Adsorption on Water Treatment Residues

Mahmood M. Barbooti*¹

Sarah H. Zahraw²

^{1,2}Chemistry Division, Applied Science Department, University of Technology, Baghdad, Iraq.

* Corresponding author: brbt2m@gmail.com,

*ORCID ID: <http://orcid.org/0000-0002-9793-7400>

²sarahhadi204@gmail.com

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Abstract:

The presence of residual antibiotics in water results in the development of antibiotics resistant genes. The available wastewater treatment systems are not capable of removing such antibiotics from sewage. Thus, antibiotics need to be removed before the discharge of wastewater. Adsorption is among the promising techniques for the wastewater treatment to aid the removal of a wide range of organic and inorganic pollutants. The present work is a contribution to the search for an economical method for the removal of low concentrations of amoxicillin (AMX) from water by adsorption on water treatment residue, WTR, taken from a local drinking water facility. The chemical composition and the adsorptive characteristics of the material were first evaluated using energy dispersive spectroscopy, EDS, and sorption of methylene blue, respectively. The porous character of the sorbent was modified by ignition. The application of the WTR for the adsorption of AMX was studied under various operating conditions including sorbent dosage, 2-20 g/L at room temperature; contact time 30-240 min.; and initial concentration range of the antibiotic, 0.00004-0.00012 M. facility. To aid the experimental work, statistical software was employed to design the experiments and evaluation of the results. Graphical and mathematical relationships have been established for the adsorption efficiency with the operating conditions. The adsorption capacity was calculated from the plot of the adsorbed drug against the sorbent content and found to be 19.966 $\mu\text{mol/g}$ WTR. The sorption efficiency depends on the initial concentration and being better at low concentration (0.00004 M) and equilibrium time (within 100 mins.). The optimum conditions of the adsorption are: AMX Concentration, 0.00004 M; Contact time, 90 min., and WTR content of 15.5 g/L to give removal efficiency of 89.2%.

Key words: Adsorption, Amoxicillin, Antibiotic removal, Experimental design, Methylene blue Water treatment residue.

Introduction:

Antibiotics are uniquely effective and selective on pathogenic bacteria, in both human and veterinary medicine for therapeutic treatment of infections related diseases, (1). The antibiotic usage increased rapidly and concern focused on their residues in the environment as it can induce antibiotic resistant genes from extended exposure at relatively low concentrations (2). The residues of human and veterinary antibiotics have been detected in environmental samples from soil and more specifically in water (3, 4). The exposure to antibiotics at the parts per

million levels may be risky on aquatic organism's survival, body weight and growth (5).

Urban wastewater treatment plants are likely to be hotspots for the release of antibiotics in the natural environment (6). Thus, antibiotics are recognized as emerging environment contaminants (7), hence antibiotics are regarded as toxic and hazardous chemicals (8). Partial removal of antibiotics may be performed by adsorption on river sediments and the process is influenced by their iron and organic contents (9). It is important, therefore, to remove antibiotic residues before discharging wastewater

into the environment but the process is usually costly. Case studies that involved the search for providing cheap and efficient methods and materials to affect the cost of antibiotic removal are urgently needed (10, 11). Processes are advanced oxidation such as catalytic ozonation (TiO_2/O_3 in dark), photocatalytic oxidation ($\text{TiO}_2/\text{UVA}/\text{O}_2$), and photocatalytic ozonation ($\text{TiO}_2/\text{UVA}/\text{O}_3$). The methods can convert antibiotic molecules into simple compounds or even mineralize them completely. However, the relative costs and outcomes of these processes are very expensive (12, 13). Even though, they are difficult to maintain regarding the total removal of compounds including antibiotics at industrial scale. Thus, physicochemical methods are proving to be highly suitable treatment option for organic contaminants (14, 15). Adsorption process is very efficient, simple to be designed and operated; and it is relatively inexpensive (16 - 18). The efficiency of adsorption processes is highly affected by the type of adsorbent, adsorbate properties and the compositions of waste stream (19). Clay minerals, e.g. bentonite, montmorillonite, and kaolinite have a high surface area and pore volume and may act as adsorbent for the remediation of antibiotics from water and wastewater. Montmorillonite proved an excellent adsorbent for the removal of oxtetracycline from water (17). Activated carbon, AC, gave better adsorption efficiency than bentonite regarding amoxicillin removal from real wastewater. However, neither of the two adsorbents could completely remove amoxicillin due to the sorption competition from other substances which were also present in the wastewater (19). Balarak, et al., (20) used palm bark as adsorbent for AMX from aqueous solutions and reported an adsorption capacity of 35 mg/g. Tovar, et al., (21) used AC impregnated with ZnCl_2 as an adsorbent for the removal of amoxicillin from aqueous solution at a removal rate of 77.4%. The efficiency of the removal was 60-70% for a 200 mg/L solution of amoxicillin using a dose of 600 mg/L adsorbent. Both oxidation and adsorption techniques have their advantages and limitations. Boukhelkhal, et al., (22) utilized wheat grains as adsorbent to remove amoxicillin antibiotic from wastewater for a maximum removal percentage of 84 % for amoxicillin on wheat grain treated with 20% tartaric acid. Different clay materials were evaluated by Maia, et al., (23) for the removal of amoxicillin and other medicines like caffeine, propranolol, and diclofenac sodium from aqueous solutions and gave removal efficiency varied between 23-98 % for amoxicillin. Song, (24) succeeded in the removal of amoxicillin on novel

nano (mesoporous silica supported polymers), which was characterized by large surface area and amendable surface characteristics. Graphene oxide (GO) and magnetite GO were also employed for the adsorption of amoxicillin by Moradi, (25).

Many authors used cheap adsorbents to ensure the economy of the process like slag (magnetite) and bentonite clay (Maichin, et al., (26), sludge (De Gisi, et al, (27), and aluminum-based WTR (Lee et al, (28). Zhou and Haynes, (29) reviewed the effect of working parameters on the sorption efficiency of heavy metal ions on WTR. Punimaya, et al. (30), evaluated the arsenite As (III) and arsenate [As(V)] sorption by Fe- and Al-based WTR as a function of the particle size at different pH values. Suda et al., (31) used WTR to stabilize arsenic in flooded soils and attenuate arsenic uptake by rice (*Oryza sativa*L.) plants. Drinking water treatment residual was characterized by Zhao, et al., (32) to adsorb chlorpyrifos. The use of Ca-WTR was studied as a cost-effective remediation method for the Cu-contaminated soils without affecting citrus production (33). Barbooti, et al., (34) utilized the WTR in the control of phosphate levels in irrigation water.

The aim of the present work is the evaluation of WTR as a sorbent with the aid of methylene blue, MB, adsorption as a model compound. The WTR will be then utilized for the removal of AMX from water under various operating conditions like initial concentration, contact time and the amount of the adsorbent. Experimental design program was employed for the design and evaluation of results.

Materials and Methods:

The AMX (molecular mass 365.4 g/mol) was 98% pure materials supplied from The State Drug Industries State Company, Samarra, Iraq and used without further purification. The MB was analytical reagent from Riedel de Haen, Germany. The water treatment residue, WTR was obtained from a local drinking water facility (Wathba Station, Utafiyah, Baghdad) during the maintenance period. The composition will be studied in details after some preparation. It was naturally dried and ground in an agate mortar to pass 0.06 mm mesh. The material was further activated by thermal treatment in a muffle furnace at 650° C and the

Apparatus

The concentration of methylene blue and AMX solutions were measured by spectral absorption on EMC Lab, GMBH, Germany, (UV-1100) spectrophotometer. The full spectra were recorded on a Shimadzu, UV/Vis spectrometer. Energy dispersive spectral, EDS, measurements

were made on EDS instrument from FEI company, Netherland. The sorption experiments involved the shaking of the samples with Genex, shaker, Florida, USA.

Procedures

Calibration graphs

The calibration graph for the spectral determination of MB was prepared by serial dilution of the stock solution within the range of 0.000004.0 – 0.000006 M. The absorbance was measured at 229 nm as the wavelength of maximum absorption over a range of concentration 0.00004-0.00012 M. This corresponds to 14.6-36.5 mg/L.

2.3.2. Preparation and evaluation of WTR

The WTR sample was air-dried for 72 h at room temperature, dried at 105° C. It was then homogenized, grinded and sieved to end up with three portions of 1.18, 0.075, and 0.07 mm particle sizes. The pH was measured for the 1:2.5 w/v (WTR: water) suspension for 1 h. To decompose the organic matter (OM), a known weight of WTR was placed in porcelain crucible and ignited to 650°C by laboratory Furnace. The composition of WTR was evaluated before and after ignition using the EDS. The adsorption properties of WTR were evaluated by studying the removal of methylene blue (MB) from water. In four plastic containers, 20-ml aliquots of MB solutions of 0.0002M were placed together with four different WTR amounts in plastic containers, capped, sealed with Parafilm and shaken for two hours at a speed of 250 rpm. The residual MB was estimated by referring to the calibration graph.

The Cation Exchange Capacity, Determination:

Dye cations like MB will mainly be adsorbed by cation exchange (35). Thus, the MB adsorption depends on the exchangeable cations of the sorbent. The Cation Exchange Capacity, CEC, was determined using the procedure used by Barbooti, et al., (34) employing MB adsorption as indicator material.

Surface Area Determination:

The specific surface area can also be calculated with MB adsorption when the sorbent surface area per charge corresponds with the area of the methylene blue cation of 130 Å² (35). The details of the procedure were given in a recent application for the removal of phosphate from water (34)

Experimental Design:

Experimental design (Statistica) was used to study the effects of experimental variables (operating parameters), at various levels

simultaneously on the adsorption of the selected antibiotics on the basis of statistical treatment to determine the optimum conditions (36). The details of the approach were recently described regarding the removal of nickel ions from water by adsorption on certain composite (37). It saves time and efforts and at the same time measures the precision of the process. The response can be estimated at any set of conditions when the selected model fits with the practical results. The operating conditions for AMX adsorption on WTR are given in Table 1.

Table 1. Real experimental parameters conditions for adsorption of AMX on WTR.

No.	Contact Time, min	Sorbent Dose, g/L	Amoxicillin Concentration, M
1	105	6.5	0.00006
2	105	6.5	0.0001
3	105	15.5	0.00006
4	105	15.5	0.0001
5	195	6.5	0.00006
6	195	6.5	0.0001
7	195	15.5	0.00006
8	195	15.5	0.00008
9	60	11	0.00008
10	240	11	0.00008
11	150	2	0.00004
12	150	20	0.00012
13	150	11	0.00008
14	150	11	0.00008
15	150	11	0.00008
16	150	11	0.00008
17	150	11	0.00008
18	150	11	0.00008

Preliminary experiments

A set of adsorption experiments was conducted in duplicate on a fixed AMX concentration solution (0.00006 M) with varying amounts of WTR contents (0.2, 0.35, 0.4, 0.55, 0.7, 0.85 and 1.0 g) placed in 60-mL capacity container and 1.0 mL of KCl solution to ensure the necessary ionic strength in the solution. The total volume was made up to 50 mL by deionized water. The containers were capped and further sealed by Parafilm. The containers were shaken for 180 min at 250 rpm. The residual concentrations were analyzed by UV absorption at 229 nm. The absorbance measurements were checked against a blank to ensure freedom from interferences.

Results and Discussion

Evaluation of the WTR

The ignition of the WTR resulted in a loss of 16.56%, which is associated with the thermal decomposition of the organic matter content and the possible decomposition of some carbonates. The

color of the material changed from light grey to pink. The chemical composition of the material before and after the ignition confirms such suggestion. The changes in the elemental contents are listed in Table 2. A clear increase in the calcium content and a decrease of the oxygen could be observed and disappearance of the carbon signal. Carbon is related to the calcium carbonate and the organic matter content of the WTR. The composition of WTR comprised a mixture of various inorganic elements. The Ca and Al contents were reported by Ippolito et al. (2003) (39) to be present in Al-WTR, whereas the presence of Al, Fe, P, Si, Ca, and Na in Al- and Fe-WTR as given by Makris et al. (40). The thermal activation results in enhancement of the power of the adsorptive materials and gives the porous structure due to interlayer spaces collations (41). Moreover, some chemical activation of adsorptive materials generated a more porous structure as well as more oxygenated functional groups on their surfaces than thermal activation only (41).

Table 2. Elemental composition of the water treatment residue before and after the ignition.

Element	Weight Percentage	
	Before burning	After burning
O	48.49	41.50
Si	15.73	21.51
Ca	11.38	13.25
C	7.27	-----
Al	5.66	7.65
Fe	3.23	5.63
Mg	2.27	3.98
K	1.58	1.12
Na	0.10	0.16

The CEC value of the WTR was determined by plotting the adsorbed amount of MB (319.85 g/mole) against the corresponding WTR content as shown in Fig. 1. From the slope of the plot, the CEC value was calculated and found to be 20.63 $\mu\text{mole/g}$. This corresponds to 6.6 mg/g. Sorbents with smaller particle size gave higher CEC values (30).

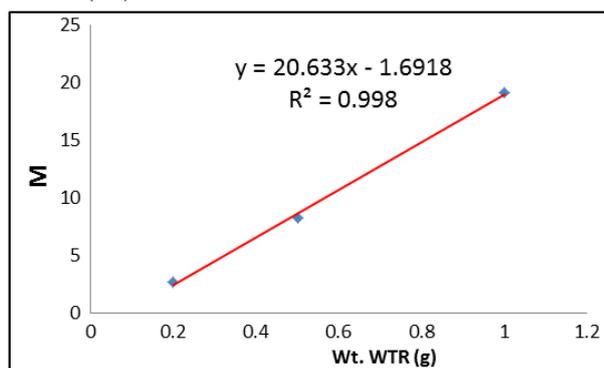


Figure 1. Cation exchange capacity plot for the water treatment residue.

Preliminary experiments

A sets of sorption experiments were carried using a range of WTR amounts 2-20 g/L) in contact with the same aliquot of AMX (0.00006 M) and shaken for 120 mins. The adsorption capacity was estimated over a range of WTR contents by plotting the adsorbed concentration against quantity of the WTR (Fig. 2). The slope of the plot represents the average adsorption capacity. The estimated capacity for AMX was 19.966 $\mu\text{mol AMX/g WTR}$.

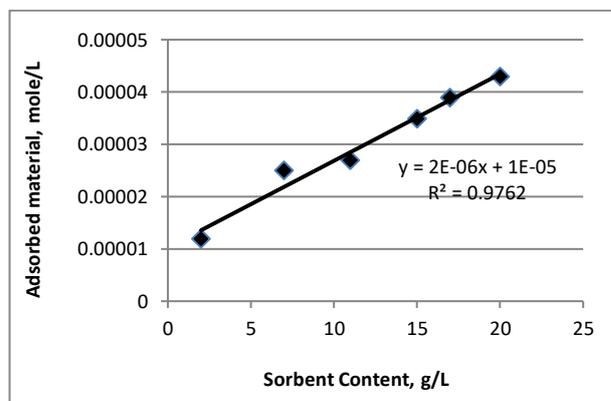


Figure 2. Determination of adsorption capacity of the amoxicillin.

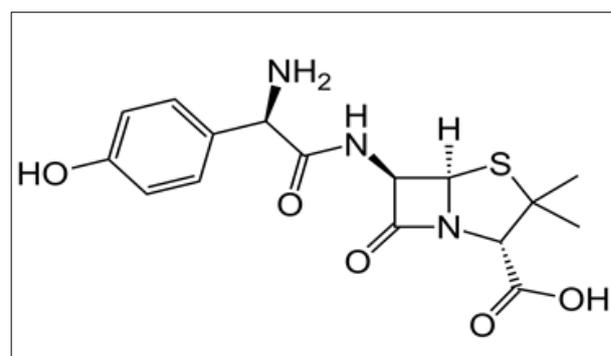


Figure 3. The chemical composition of the amoxicillin.

The chemical composition of AMX (Fig. 3) indicates the existence of two OH and two NH₂ groups. Thus, the interaction was mostly through lone pairs of electrons in the two materials.

Amoxicillin Adsorption on WTR

Experimental design results

The adsorption efficiency values were calculated using eq. 1

$$\text{Eff\%} = (C_0 - C_i) / C_0 \dots\dots\dots(1)$$

Where, C₀ is the initial AMX concentration and C_i is the AMX equilibrium concentration. The results of the adsorption efficiency of AMX on the WTR were calculated at various equilibrium times, initial AMX concentration and WTR contents, were

the input. The experimental design program used the data of Table 1 together with the corresponding sorption efficiency to estimate the coefficients of the response equation (2) relating the efficiency with the experimental parameters:

$$\text{Eff\%} = a_0 + a_1 * X_1 + a_2 * X_2 + a_3 * X_3 + a_4 * X_1 * X_2 + a_5 * X_1 * X_3 + a_6 * X_2 * X_3 + a_7 * X_1^2 + a_8 * X_2^2 + a_9 * X_3^2 \dots \dots (2),$$

where X_1 , X_2 , and X_3 represent the equilibration time (min), the sorbent content (g/L) and initial AMX concentration (M), respectively. The coefficients (a 's) were taken directly from the program output. The numerical values of the coefficients represent a direct proof on the dependence of the sorption efficiency on the specific parameters (Table 3).

Table 3. the coefficients of equation (2).

a_0	a_1	a_2	a_3	a_4
-6.7	0.5	7.2	90416	0
a_5	a_6	a_7	a_8	a_9
537.4	-29366	0	-0.2	0.1

The zero value of the a_4 reflects the negligible interaction between equilibrium times with WTR dosage. The negative value of (a_6) coefficient gave a clear evidence that concentration has a negative interaction with the sorption efficiency. Further, the zero value of the (a_7) indicates the independence of sorption efficiency with the square of the equilibrium time and thus only linear correlation exists with time. After the AMX solution was equilibrated with the WTR powder for the predetermined time, the residual antibiotic concentration was determined by spectral absorption at 229 nm. The adsorption efficiency was calculated for each specimen using the relation (Equation 3):

$$\text{Eff\%} = -6.7 + 0.5 * X_1 + 7.2 * X_2 + 90416.4 * X_3 + 537.4 * X_1 * X_3 - 29366.2 * X_2 * X_3 - 0.2 * X_2^2 + 0.1 * X_3^2 \dots (3)$$

Effect of sorbent content

Figure 4 shows the effect of the sorbent content on the sorption efficiency, at fixed AMX concentration of 0.00008 M after various contact times (30 - 120 minutes). The sorption efficiency showed an increase as the sorbent content increase up to a value of 11.0 g/L. Also, longer equilibrium time results in higher sorption efficiency.

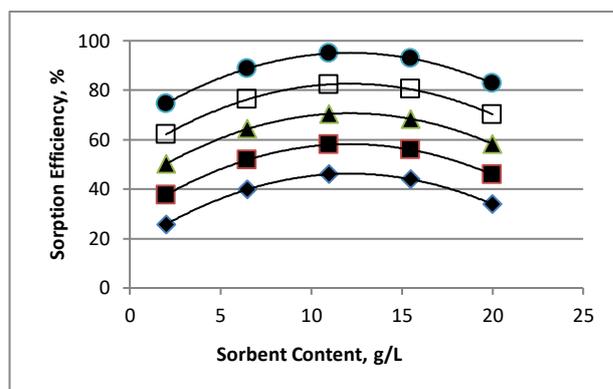


Figure 4. Effect of the WTR content on the sorption efficiency of 0.00008 M AMX after 60 (◆), 75 (■), 90 (▲), 105 (□) and 120 minutes (●) contact time.

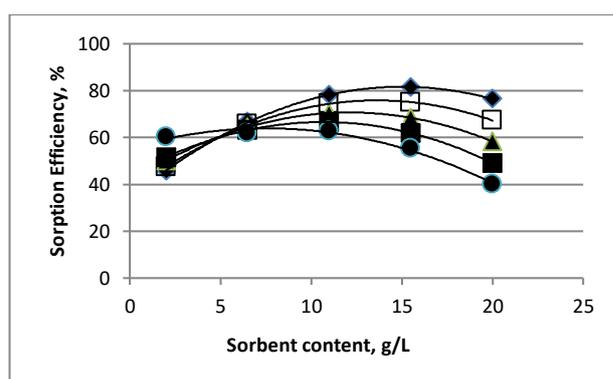


Figure 5. Effect of WTR content on the sorption efficiency after 90 min contact time and for: AMX initial concentrations. 0.00004 (●); 0.00006 (■); 0.00008 (▲); 0.00010 (□), and 0.00012 M (◆).

Figure 5 shows the effect of sorbent content on the sorption efficiency at a fixed equilibration time for 90 min and various AMX concentrations. The sorption efficiency exhibited a significant increase as the sorbent content increases until a maximum value is obtained at 15.5 g/L. However, the sorption process decreases significantly with the increase of the concentration. Higher concentration resulted in a competition between the AMX molecules towards the active sites of the sorbent (24).

Effect of Equilibrium time

Figure 6 shows the effect of equilibrium time on the sorption efficiency at a fixed AMX concentration of 0.00008 M and various sorbent contents. The sorption efficiency linearly increases as the equilibrium time increases at all sorbent content values. Again the sorbent content of 11-15.5 g/L gave the highest response (92-94%). For the highest sorbent content, the efficiency dropped down to 82.7%.

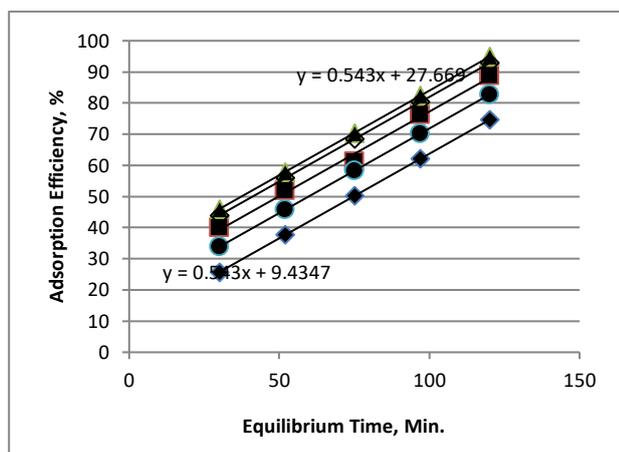


Figure 6. Effect of equilibrium time on sorption efficiency of 0.00008 M AMX concentration on 2 (◆), 6.5 (□), 11 (▲), 15.5 (◇) and 20 g/L WTR content (●).

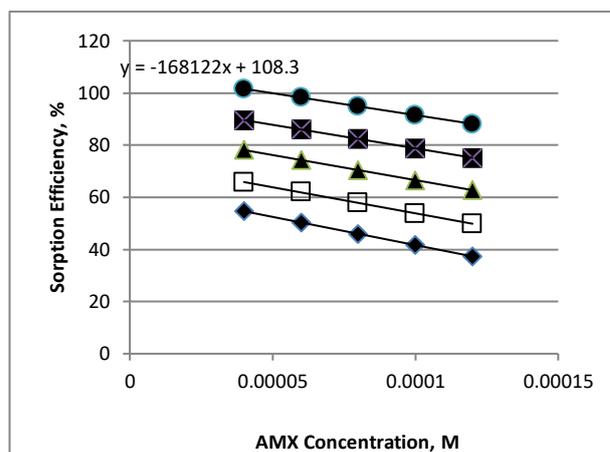


Figure 8. Effect of AMX concentration of the sorption efficiency on 11 g/L sorbent after 60 (◆); 75 (□); 90 (▲); 105 (●) and 120 min (■).

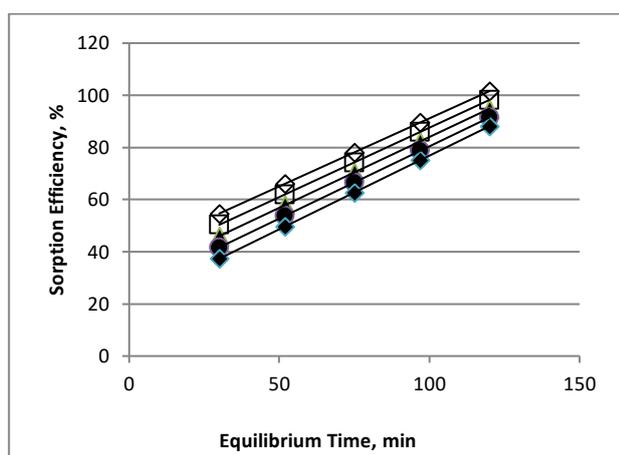


Figure 7. Effect of equilibrium time on AMX sorption efficiency on 11 g/L WTR for initial concentration of 0.00004 (◇); 0.00006 (□); 0.00008 (▲); 0.0001 (●), and 0.00012 M (◆).

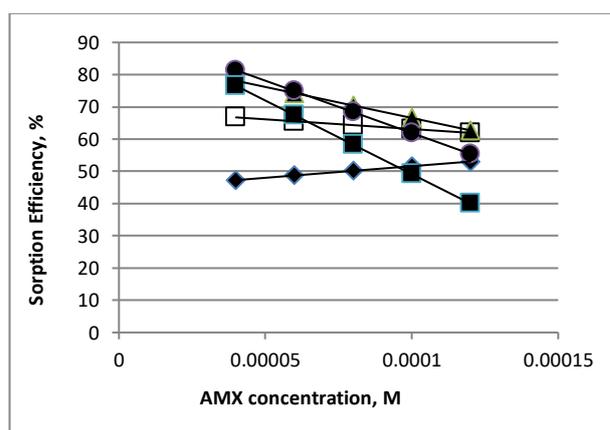


Figure 9. The effect of AMX concentration of the sorption efficiency after 90 min contact time using WTR contents of 2 (◆); 6.5 (□); 11 (▲); 15.5 (●) and 20 g/L (■).

Figure 7 and 8 shows the effect of equilibrium time on the sorption efficiency at a fixed sorbent content of 11 g/L and various AMX concentrations. The sorption efficiency linearly increases as the contact time between the AMX molecules and the WTR sorbent for various AMX concentration levels. Although the 120 minutes gave a sorption efficiency as high as 100%; the 90 minutes will be taken as the optimum contact time as the efficiency rapidly drops beyond 120 minutes. The results are more stable and repeatable at 90-100 minutes time. This is in agreement with the results of Balarak, et al., (20) who used adsorption onto palm bark biomass. The significance of adsorption time duration is not yet solved. Some authors (17) reported an overnight contact between sorbent with solution (17). Others used a 2-6 hours contact time and reported that longer time may cause desorption of the pollutant (39).

Figure 9 shows the effect of AMX concentration of the sorption efficiency at a fixed equilibrium time of 90 min. and various WTR contents. The WTR content is very important to determine the sorption efficiency as a function of concentrations. Only a slight increase in efficiency from 54 up to 62% could be established at 2 g/L WTR content with the triplication of initial concentration from 0.00004 to 0.00012 M. The concentration has no significant effect at lower WTR content while the combination of low concentration with high WTR content gave the highest sorption values (85%) but sharply decreases down to less than 50% with the increase in concentration up to 0.00012 M. Pachauri, et al., (13) found that the removal of amoxicillin dropped from 80% down to 24.2% when the initial concentration was increased from 200 mg/L up to 1000 mg/L through GAC column.

Conclusion:

Water treatment residue proved successful and rather cheap sorbent with cation exchange capacity of 20.63 $\mu\text{mole/g}$ as been determined using MB adsorption. The material could be used successfully for the removal of AMX from wastewater. The sorption efficiency of the process is significantly affected by the initial antibiotic concentration being better for lowering the AMX concentration. The optimum WTR content was 15.5 g.L^{-1} . The process is relatively fast and 90 minutes contact time was found adequate for the removal of 90% of the antibiotic from water using a sorbent content of about 15.5 g/L .

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Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Technology.

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إزالة الاموكسيسيلين من الماء بالامتزاز على مخلفات معالجة المياه

سارة هادي زهراو

محمود مهدي بربوتي

قسم العلوم التطبيقية، الجامعة التكنولوجية، بغداد، العراق.

الخلاصة:

يؤدي وجود مخلفات المضادات الحيوية في المياه الى تطور جينات مقاومة لتأثيرها. ويذكر أن نظم معالجة المياه الحالية تعجز عن التخلص من هذه المخلفات من مياه المجاري. ولهذا ينبغي ازالة بقايا المضادات الحيوية من المياه المصرفة قبل ضخها الى مسارات المياه السطحية. وتعد طريقة الامتزاز من الوسائل الناجحة لازالة العديد من الملوثات العضوية وغير العضوية من الماء. البحث الحالي يقدم طريقة لازالة بقايا المضاد الحيوي (الاموكسيسيلين) من الماء وبتركيز واطنة نسبيا وذلك بالامتزاز على مخلفات معالجة المياه المأخوذة من محطة لاسالة مياه الشرب من منطقة بغداد. تم ايجاد التركيب الكيميائي لهذه المخلفات بتقنية مطيافية الانتشار الالكتروني وكذلك تحديد مواصفاتها الامتزازية باستخدام امتزاز صبغة الميثيلين الزرقاء. تم تحسين الخاصية المسامية للمادة بالحرق. تم تطبيق الطريقة لازالة الاموكسيسيلين من الماء عند ظروف مختبرية مختلفة. تم العمل بأسلوب الوجبات باستخدام زمن امتزاز ما بين 30-240 دق، تركيز ابتدائي ما بين **0.00004** مولاري، وكمية المادة الممتزة ما بين 2-20 غم/لتر. استخدمنا برنامج احصائي لتصميم التجارب وتحليل النتائج وتثبيت الظروف المثلى للعمل. تم الحصول على علاقات رياضية وبيانية ما بين ظروف العمل وكفاءة الامتزاز، فالتركيز الابتدائي عامل مؤثر جدا في تحديد الكفاءة لانه يرتبط عكسيا معها بينما تزداد الكفاءة بزيادة الزمن وكمية المادة الممتزة. وكانت الظروف العملية المثلى لامتزاز الاموكسيسيلين على مخلفات المعالجة التركيز: **0.00004** مولاري، الزمن: 90 دق واخيرا كمية المادة الممتزة: **15.5** غم/لتر. وتعتمد كفاءة الامتزاز على التركيز الابتدائي للاموكسيسيلين بدرجة كبيرة حيث تحسن كلما كان التركيز أوطأ (**0.00004 M**) وكذلك على زمن التماس مع المادة الممتزة (100 دقيقة).

الكلمات المفتاحية: الامتزاز، الاموكسيسيلين، ازالة المضادات الحيوية، التصميم التجريبي، بقايا معالجة المياه الزرقاء بالميثيلين.