



NITRATE REMOVAL FROM AQUEOUS SOLUTIONS WITH SOME ECO-FRIENDLY ADSORBENT MATERIALS

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ABSTRACT

In the current study, Eco-friendly adsorption materials were used for nitrate removal from aqueous solutions, user-friendly adsorption media for nitrate removal from water, surfactant modified was evaluated as a potential adsorption media for this purpose. The study used batch experiments at laboratory of Faculty of Agricultural Environmental Science Arish University. Therefore, residual olive wood-derived biochars (BI), Olive pomace (BW) and modified zeolite (ZE), were used as adsorbent substances. The effect of different nitrate concentrations (1.0, 1.5 and 2.0 mM⁻¹) at contact time intervals (30, 60, 90, 180, 360 and 1440 min) and pH values (4.0, 7.0 and 10.0) on the removal of nitrate was investigated in batch experiment. Residual olive wood-derived biochars (BI) recorded the highest removal percentages values of nitrate (98.9%) at initial concentrate 2.0mM⁻¹, compare with two studied prepared, followed by (BW) 93.9% and (ZE) 89.5%. Nitrate removal rate were superior under high nitrate concentration pH value 4 with all studied adsorbent substances at different magnitudes recorded the highest removal percentages values of nitrate (BI) 99.9%, (BW) 94.8% and (ZE) 90.4 %, respectively. At the same time, nitrate removal percentage rates were low during the initial period of the experiment then the rate was high. This study suggests that could nitrate and any pollution elements be removed from water using biochars. The pollution water elements concentration, contact time and pH are impact on the efficacy of such adsorbents as removal pollution elements from water.



INTRODUCTION

Pollution issue has become one of the most important public awareness issues, the excessive use of the pesticides and fertilizers in agriculture with the threat of these chemicals in crops and water. Water pollutants represent one of a serious problem for both humans (Ward *et al.*, 2018; Sorour *et al.*, 2021) and the environment. Nitrate (NO₃⁻) is an ion formed by the oxidation of nitrogen and is considered as one of the world's major surface and groundwater pollutants along with nitrite (NO₂⁻) (Revilla *et al.*, 2020) and it is the most widely used criteria for judging the quality of drinking water

(Bujnovský *et al.*, 2022). This can be explained by the anionic nature of nitrate ion, it readily leaches from the soil in addition to highly water solubility and accordingly, nitrates represent a globally widespread water pollutant (Moloantoa *et al.*, 2022). (Wray-McCann, 2022); such as the excessive use of fertilizers as well as wastes driven by animal and human (Abo-Alenen *et al.*, 2018). The total acceptable daily intake (ADI) of nitrate from all sources, including drinking water, vegetables, and food, is 3.7 mg/kg/day (Kiani *et al.*, 2022). Many studies have been aimed towards natural, environmentally friendly, low-cost materials that could be used as filters in water purification (Power and

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Schepers, 1989; Almasri, 2007) because the US environmental protection Agency (US. EPA) has set 10 mg L^{-1} as a nitrate standard level for drinking water (Development, 2011). The conventional processes used to eliminate nitrate from water are ion exchange, reverse osmosis and electro-dialysis. The utility of these processes has been limited due to their low efficient, expensive and/or subsequent disposal problem of the generated nitrate waste brine (Shrimali and Singh, 2001). The adsorption method is more preferred as filters in water purification of nitrate because its lower expenses as well as simple equipment requirement (Meftah and Zerafat, 2016). Zeolites are synthetic or naturally occurring hydrated aluminosilicates with a cage-like structure, high external cationic exchange capacity and high surface area (Moshoeshe et al., 2017). The structural framework of zeolite is negatively charged due to isomorphic substitution of aluminum for silicon. Thus, loading anionic contaminants like nitrates onto its surface is only negligibly possible, due to columbic repulsive forces (Dionisiou and Matsi, 2016). Therefore, to render the zeolite suitable for anionic contaminant removal from water such as nitrate, it is need to modify the zeolite surface to hold anions (Onyango et al., 2010; Onyango and Wanyoike, 2014). The charge properties of modified zeolites depend both on the kind of the modifier and conditions of preparation (Mahmoodi et al., 2019).

Biochar is a carbon-rich solid obtained by heating biomass, such as wood, manure with little or no oxygen which called pyrolysis or charring. The specific properties of biochars including large specific surface area, porous structure, enriched surface functional groups and mineral components make it possible to be used as proper adsorbent to remove pollutants from aqueous solutions (Tan et al., 2015). As an adsorbent, Biochars has

porous structure similar to activated carbon, which is the most commonly employed and efficient sorbent for the removal of diverse pollutants from water throughout the world (Faria et al., 2004). Compared with activated carbon, Biochars appears to be a new potential low-cost (which mainly obtained from agricultural biomass and solid waste), effective adsorbent and cheaper with lower energy requirements (Mai et al., 2013). In addition, converting invasive plant into biochars can improve the invasive plant management and protect the environment (Wang et al., 2013). Therefore, the conversion of biomass into biochars as a sorbent is a “win-win” solution for both improving waste management and protecting the environment (Ward et al., 2018).

The present study aims to investigate the nitrate removal from water with both modified zeolite and two locals prepared biochars. Also, to study the effect of initial water nitrate concentration, contact time and pH on the efficacy of such adsorbents as nitrate removal materials from water.

MATERIALS AND METHODS

Nitrate removal by prepared two biochars surfaces and modified zeolite were studied using batch experiments. Experiments were conducted in a set of 50 mL Falcon tube (fig.1) after the addition of nitrate solution to 0.333 g of different three adsorbent. The adsorbent materials were 1) Olive trees residues (B1), has been collected from Experimental Farm of Arish University, 2) Olive solid wastes (BW) has been used to produce biochars at pyrolysis temperature and time of $350 \text{ }^{\circ}\text{C}$ and 60 min, respectively, and 3) Modified Zeolite (ZE) has been provided by University of Nottingham, Faculty of Science, UK. Samples were then shaken at 120 rpm at laboratory temperature $20 \pm \text{ }^{\circ}\text{C}$ for different stilling contact times.



Fig. 1. Falcon tube

Nitrate Stock Solution Preparation

Nitrate stock solution of ($4 \text{ mM L}^{-1} \text{ NO}_3^-$) concentration was prepared by dissolving 0.404 g potassium nitrate (KNO_3) in 100 mL distilled water and then diluted to 1000 mL. From this solution different concentrations of (1, 1.5 and 2 mM L^{-1}) were prepared by dilution in 30 mL of each of these solutions was used in every experiment.

Effect of Contact Time

To study the kinetics of nitrate ion adsorption process, nitrate solutions were added to 0.333g of three studied adsorbent materials at laboratory conditions with contact time intervals of (30, 60, 90, 180, 360 and 1440) min.

Effect of Initial Nitrate Ion Concentration

Nitrate solutions with different concentrations ($1, 1.5$ and 2 mM L^{-1}) were added to three adsorbent materials (0.333g). The suspensions were then shaken for 30, 60, 90, 180, 360 and 1440 min. Initial and final nitrate concentrations were measured immediately. The percent nitrate removal was calculated as:

$$R = \frac{C_0 - C_f}{C_f} \times 100$$

Where, C_0 and C_f are the initial and final concentrations of nitrates (mM L^{-1}) in the aqueous solution.

Effect of pH

The pH of the solutions was traded as - acidic (4.0) basic (10.0) and neutral (7.0) using 0.1 molar hydrochloric acid and 0.1 molar sodium hydroxide solutions. At the end of the contact time, the samples were filtered and analysis for nitrate concentration.

Nitrate Determination in Aqueous Solution

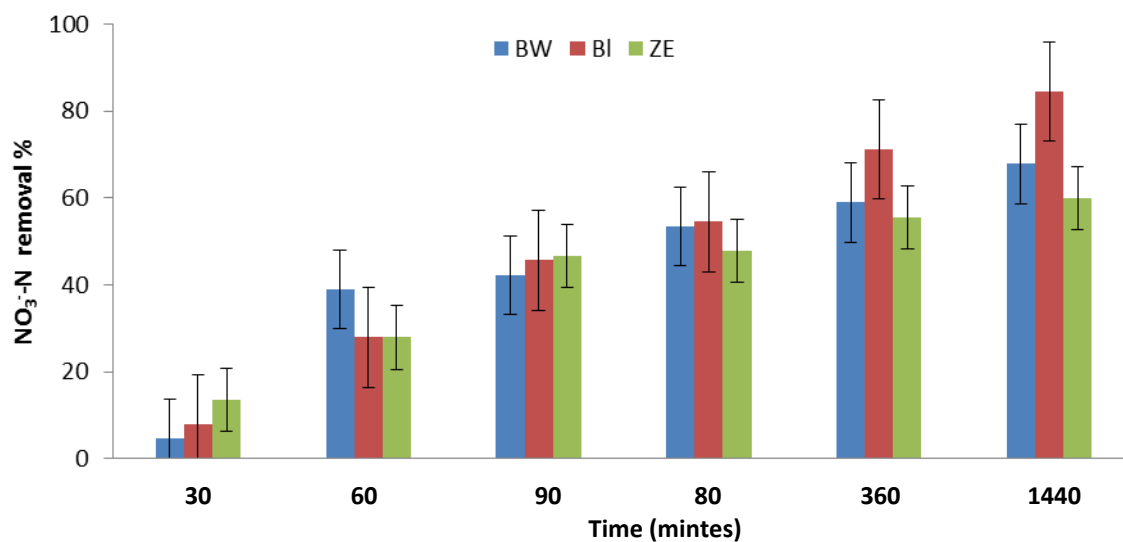
Spectrophotometry was chosen and preferred to many other methods. That is due to its low pollution effects, simplicity, speed and suitability to indicate the kinetic change of the nitrate concentration. Nitrate was determined according to **Singh (1988)**.

RESULTS AND DISCUSSIONS

Adsorption Kinetic is an important characteristic in order to evaluating the efficiency of adsorption processor. The Kinetic behavior of the three studied adsorbent materials was studied at native pH value of the prepared nitrate aqueous solution, obtained results in Table 1 and Figs. 2.1 to 2.4.

Table 1. Effect of initial concentration mMl^{-1} and contact time min, on the removal of nitrate (%), from aqueous solution using studied adsorbent materials

Adsorbent material	NO_3^- -N mMl^{-1}	Contact time, min					
		30	60	90	180	360	1440
Nitrate removal, percent (%)							
BI	1.0	7.9	27.9	45.6	54.5	71.1	84.5
	1.5	60.0	68.9	72.3	74.1	83.9	91.1
	2.0	12.3	23.4	41.7	52.8	87.2	98.9
BW	1.0	4.5	39.0	42.3	53.4	58.9	67.8
	1.5	61.6	73.8	80.8	82.8	86.5	88.2
	2.0	23.4	36.7	45.6	73.4	80.0	93.9
ZE	1.0	13.4	27.9	46.7	47.8	55.6	60.1
	1.5	14.9	65.1	77.1	80.6	83.0	85.2
	2.0	10.6	27.3	37.3	75.0	81.1	89.5

**Fig. 2.1. NO_3^- -N removal percent by ZE, BW and BI at (initial conc: 1.0 mMl^{-1} during contact time of (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g /30 mL) and error bars represent.**

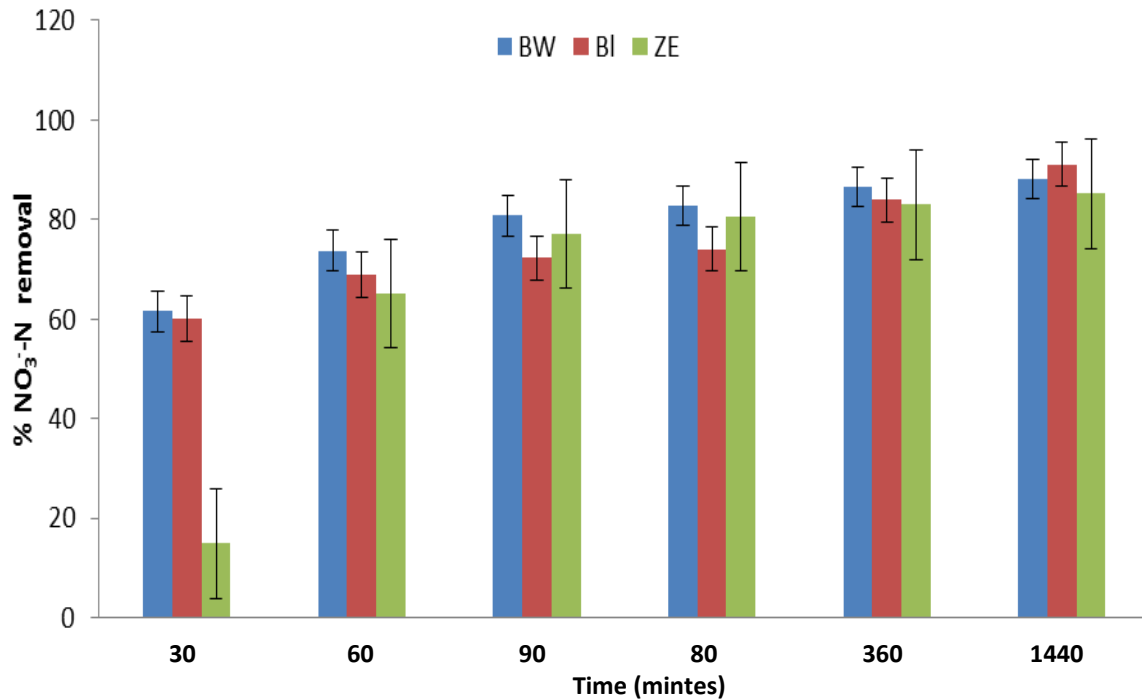


Fig. 2.2. ($\text{NO}_3\text{-N}$) removal percent by ZE, BW and BI at (initial conc: 1.5 mM l^{-1} during contact time of (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g /30 mL) and error bars represent.

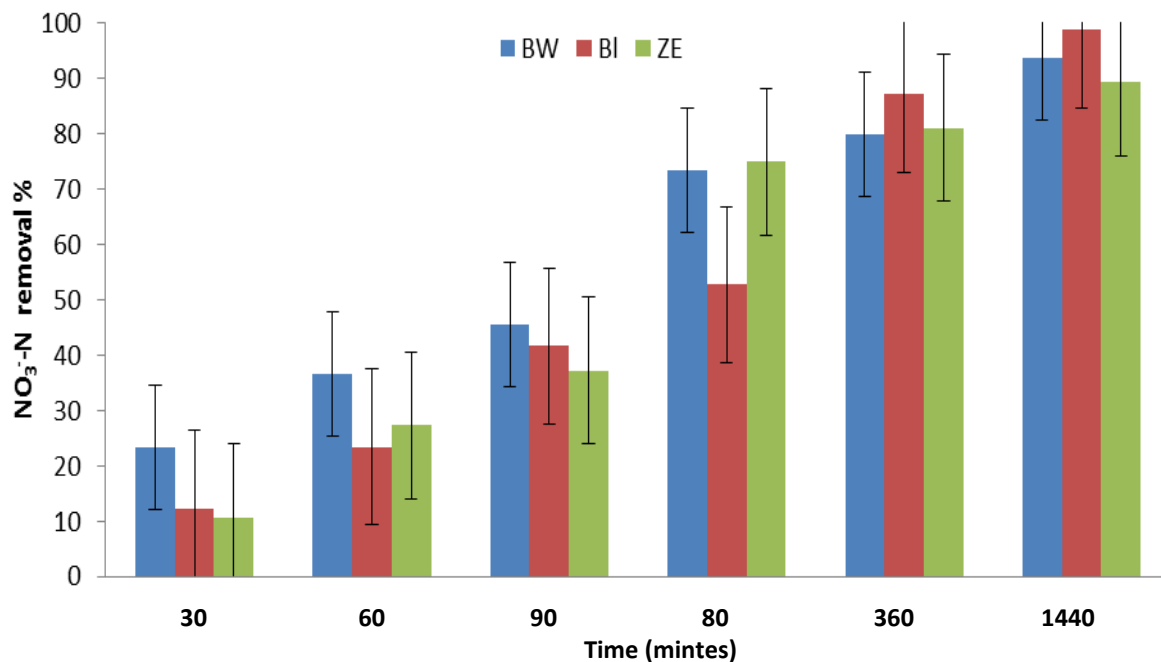


Fig. 2.3. ($\text{NO}_3\text{-N}$) removal percent by ZE, BW and BI at (initial conc: 2 mM l^{-1} during contact time of (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g /30 mL) and error bars represent.

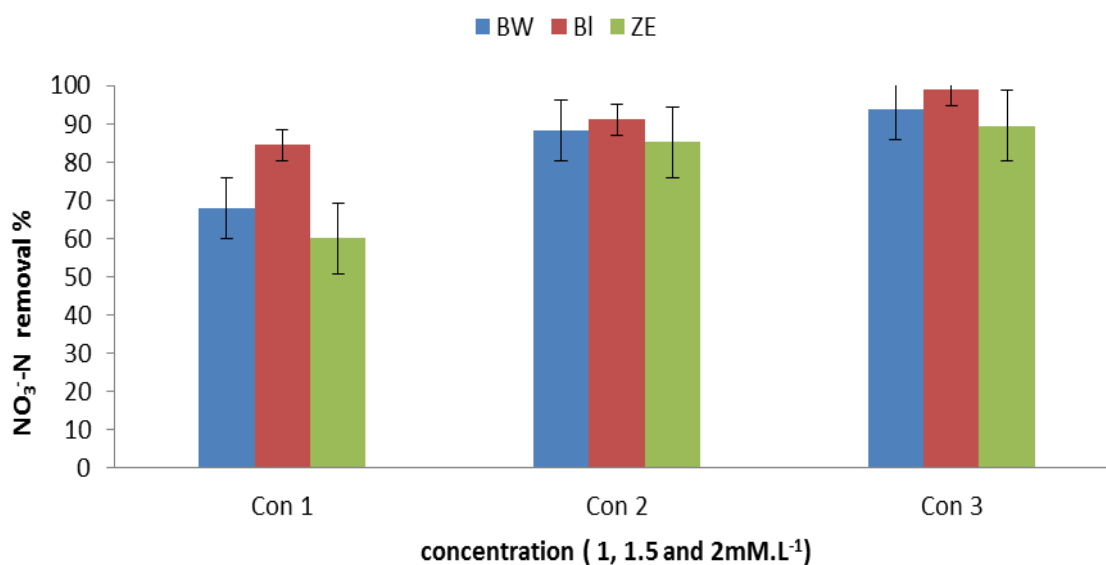


Fig. 2.4. (NO_3^- -N) removal percent by ZE, BW and BI at (initial conc: 1, 1.5 and 2 mM.L^{-1} , contact time (1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent.

Effect of Adsorbent Type

Results presented in Table 1 and Fig 2.1 to 2.3 shows that BI has a greater NO_3^- removal percentage than that of BW and ZE. It exhibited a removal percentage highly during increasing time period of the experiment. Nitrate removal percentage by BI, BW and ZE increased with increasing contact time. The percentages of nitrate removal by BI, BW, and ZE were 98.9%, 93.9%, and 89.5%, respectively. Furthermore, BI had the highest capability of removing NO_3^- ions, followed by BW and ZE. These results in general were in agreement with **Shartooh *et al.*, 2014**. Such fixations can be explained by the fact that there was high surface sites available for nitrate adsorption on BI, BW, and ZE. These findings were in agreement with **Brumagne *et al.* (2004)**, **Osma *et al.* (2012)**, **Habib *et al.* (2014)** and **Shartooh *et al.* (2014)**.

Effect of Contact Time

Effect of contact time on removal of nitrate (NO_3^- -N) by BI and BW, ZE at (initial con: 1, 1.5 and 2 mM.L^{-1} , contact

time (30, 60, 90, 180, 360 and 1440 min) and solid/liquid ratio 0.333 g/30 mL) is shown in Fig. 2.1 to 2.3. Obtained results cleared that the kinetic of nitrate adsorption under using of the three studied adsorbent materials, consists generally of two phases, An initial phase with a slower process while the second one is very fast phase when equilibrium state was nearly reached. The high rate of nitrate removal is probably due to the greater availability of surface binding sites with the passage of contact time. Adsorbent materials with different adsorption equilibrium state was reached after a contact time of about 180, 360 and 1440 min for such obtained results may be indicated to almost all covering of available adsorption sites or reaching to equilibrium state which adsorption process equal to desorption from the surfaces of three, studied adsorbent materials. The high of nitrate removal rate might possibly be due to ion exchange followed by a slow chemical reaction of the ions with active groups on the sample (**Saeed *et al.*, 2005**).

Effect of Nitrate Concentration

The initial nitrate concentration in solution was varied from 1, 1.5 and 2 mM^{-1} . Obtained results showed that the percentage removal of nitrate decreased in three studied adsorbent materials for initial nitrate concentration (such effects could be due to the limitation of adsorption sites on all studied adsorbent material with different magnitudes in first times (30, 60 and 90 min) (Vimonses *et al.*, 2009) (Moussavi and Khosravi, 2011). (Islam and Patel, 2011) indicated that with increase in initial nitrate concentration the amount of adsorptive species in the soluble increases, but the amount of adsorbent remains constant. Thus, the percentage removal increases with increase in concentration and *vice versa*. The higher adsorption of high concentrations may be due to the higher availability of more active sites on adsorbent materials.

The results for the effect of concentration on adsorption of the nitrate ion Fig. 2.4 show that adsorption of $\text{NO}_3\text{-N}$ increased as their concentrations increase. The variations in removal percentage of the nitrate with different concentrations showed a regular trend, this result agrees with (Padmapriya *et al.*, 2012). Such effects can be explained by the fact that for small particles a large external surface area results in a powerful driving force per unit surface area for mass transfer (Mustaqeem *et al.*, 2013).

Effect of pH

Obtained results in Table 2 and Fig. 3.1 to 3.3 clear that, removal by BI was the highest and each of BW and ZE was the lowest with NO_3^- concentrations, nitrate removal, obtained results showed that under pH 4 removal nitrate percent after 1440 min were 85.3, 92.0 and 99.9 with initial NO_3^- of concentration of 1.0, 1.5 and 2.0 mM^{-1} , respectively. The corresponding removal percentage values for BW and ZE adsorbent materials were 68.5, 89.0, 94.8

and 60.6, 86.0, 90.4, respectively. By increasing pH values, to 7 NO_3^- removal percentages with 1.0, 1.5 and 2.0 mM^{-1} initial concentration with BI recorded 80.7, 82.0 and 94.4%, respectively. The corresponding values for BW and ZE adsorbent materials were 64.8, 79.3, 89.7 and 57.3, 76.7 and 85.4%. On the other hand, increasing pH value to 10 resulted in NO_3^- removal percentages of 76.0, 82.0 and 89.0 with BI adsorbent material. The corresponding values with BW and ZE were 61.0, 79.3, 84.5 and 54.0, 76.7 and 80.5, respectively.

In general, nitrate removal percentages were higher under lower pH values (pH 4) compared with the other two studied pH values 7 and 10. It is worth noting that generally, the BI adsorbent material has highly and more efficiency in nitrate removal percentages, the higher pH value of an aqueous solution, and the lower removal percentages of NO_3^- . Such effects were found true under all studied adsorbent materials. With the increase in pH values, the surfaces of adsorbent materials (two biochars and zeolite) decrease in the extent of positive charging and become negative under modified zeolite at a pH of about 6 (the isoelectric point of clay is at 5.6) and at pH 7 for two used biochars. Thus the high adsorption capacity under low pH is mainly due to the strong electrostatic between the positively charged sites of adsorbent (high presence of H^+) and the nitrate negatively charged anion. However, lower sorption of nitrate ions at high pH alkaline conditions could be attributed to the abundance of OH^- ions which will compete with the negatively charged nitrate ion pollutant for the same sorption sites (Almubarak *et al.*, 2015). The decrease in nitrate adsorption with the increase in pH could be due to interaction during passive transport in the pores and competition between OH^- and NO_3^- anions for active sites one disadvantage of using adsorption. A process in anions removal contaminates from water

Table 2. Effect of pH values under different initial concentration (mMI⁻¹) on the removal of nitrate, percent from water using studied adsorbent materials

Adsorbent material	pH	NO ₃ ⁻ -N mMI ⁻¹	Contact time, min					
			30	60	90	180	360	1440
Nitrate removal, percent (%)								
BI	4	1.0	8.0	28.1	46.1	55.0	71.9	85.3
		1.5	60.6	69.6	73.0	74.8	84.9	92.0
		2.0	12.4	23.6	42.1	53.4	88.1	99.9
	7	1.0	7.5	26.6	43.6	52.0	67.9	80.7
		1.5	54.0	62.0	65.0	66.7	75.8	82.0
		2.0	11.8	22.4	39.8	50.4	83.3	94.4
	10	1.0	7.1	25.1	41.1	49.0	64.0	76.0
		1.5	54.0	62.0	65.0	66.7	75.5	82.0
		2.0	11.1	21.1	37.6	47.5	78.5	89.0
BW	4	1.0	4.6	39.3	42.7	53.9	59.5	68.5
		1.5	62.2	74.5	81.6	83.7	87.4	89.0
		2.0	23.6	37.1	46.1	74.1	80.8	94.8
	7	1.0	4.3	37.2	40.4	51.0	56.3	64.8
		1.5	55.4	66.4	72.7	74.5	77.9	79.3
		2.0	22.4	35.1	43.6	70.1	76.4	89.7
	10	1.0	4.1	35.1	38.1	48.0	53.0	61.0
		1.5	55.4	66.4	72.6	74.5	77.9	79.3
		2.0	21.1	33.1	41.1	66.0	72.0	84.5
ZE	4	1.0	13.6	28.1	47.1	48.3	56.2	60.6
		1.5	15.1	65.7	77.8	81.4	83.8	86.1
		2.0	10.8	27.6	36.7	75.8	81.9	90.4
	7	1.0	12.8	26.6	44.6	45.7	53.1	57.3
		1.5	13.4	58.7	69.4	72.6	74.7	76.7
		2.0	10.2	26.1	35.6	71.7	77.5	85.9
	10	1.0	12.1	25.1	42.1	43.1	50.0	54.0
		1.5	13.4	58.6	69.4	72.6	74.7	76.7
		2.0	9.6	24.6	33.6	67.5	73.0	80.5

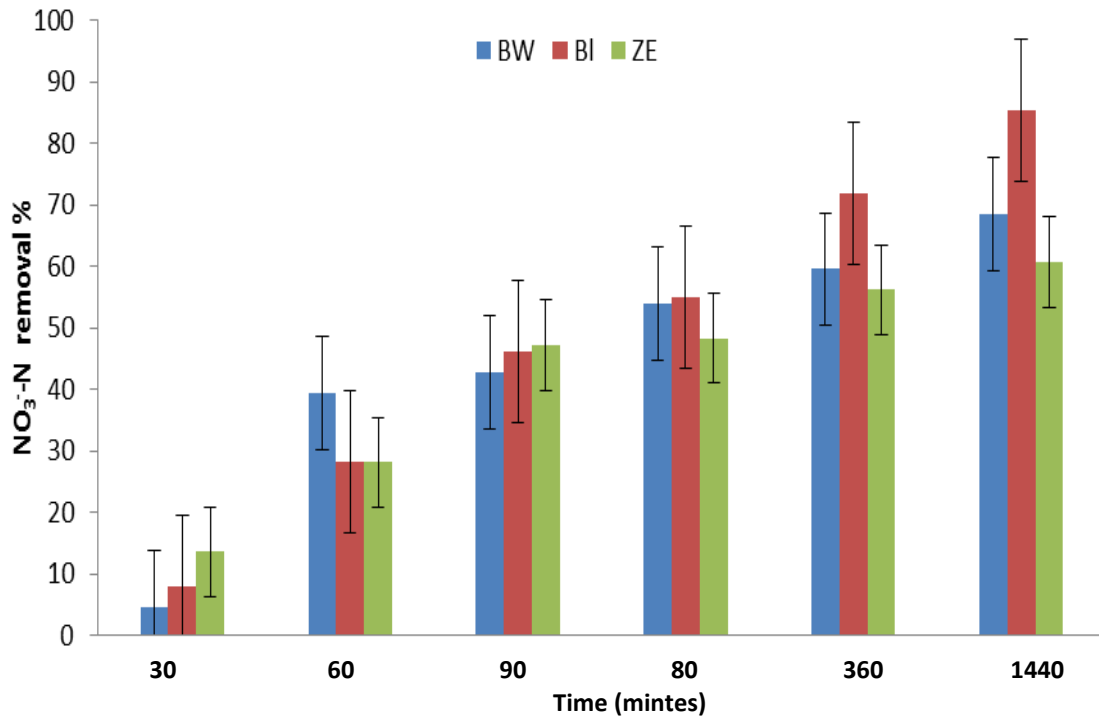


Fig. 3.1. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 1.0 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent

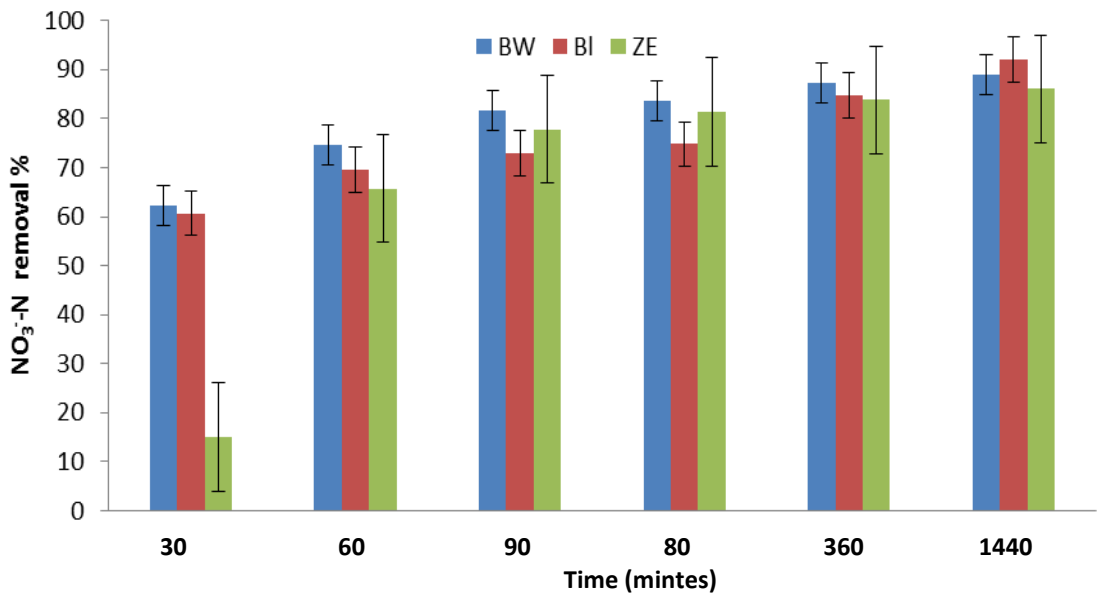


Fig. 3.2. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 1.5 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent

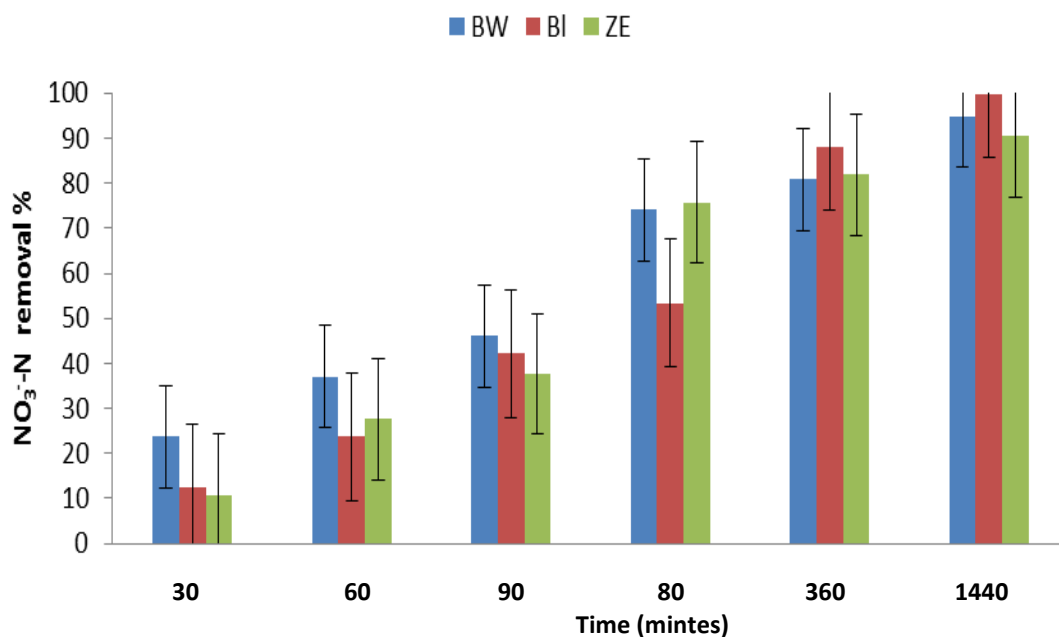


Fig. 3.3. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 2 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent.

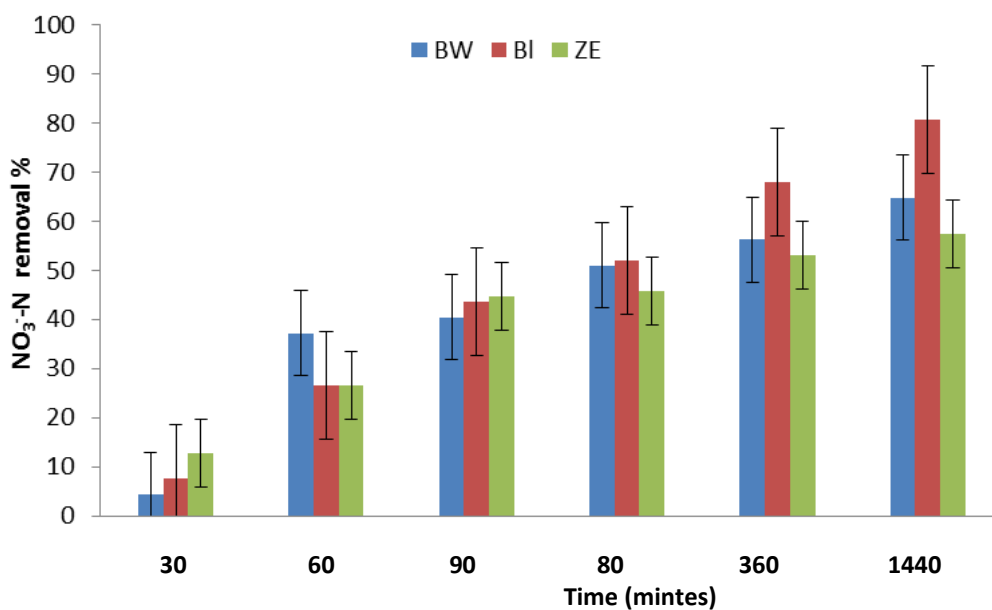


Fig. 3.4. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 1.0 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent.

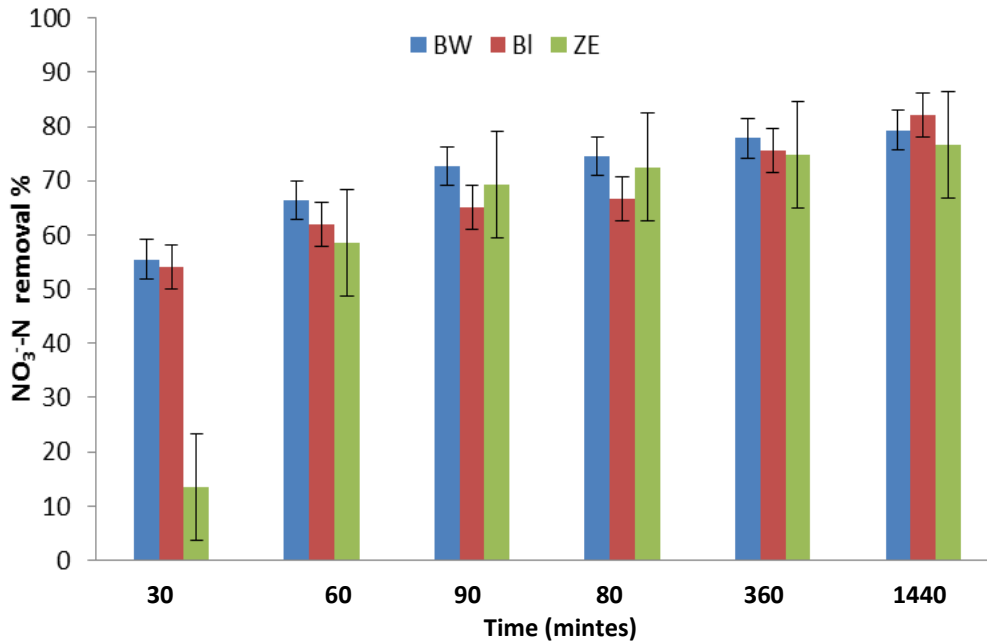


Fig. 3.5. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 1.5 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent

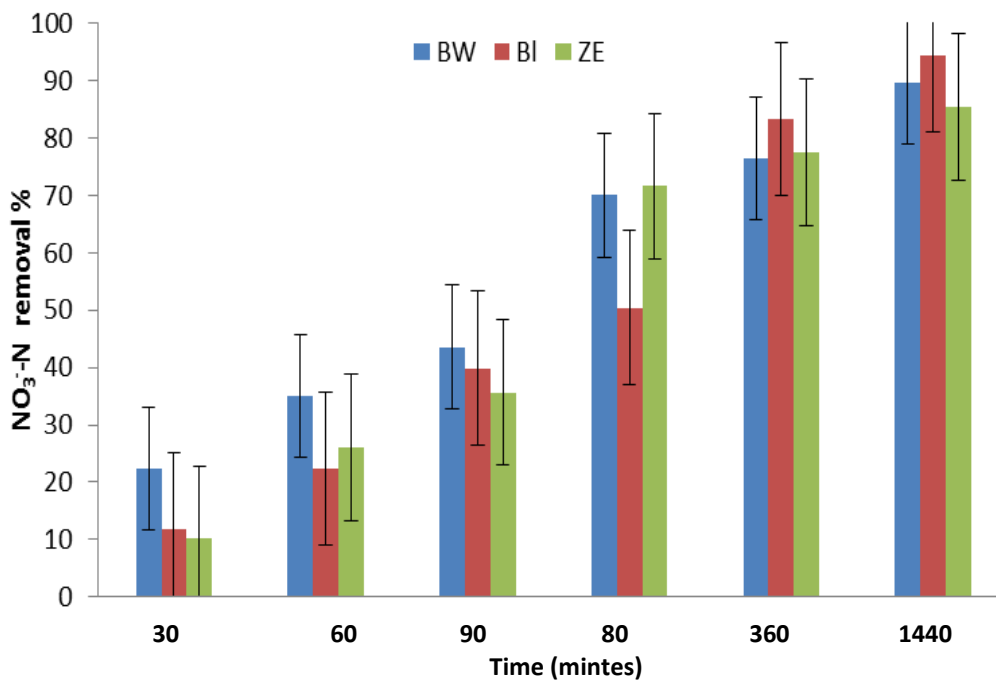


Fig. 3.6. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 2 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent

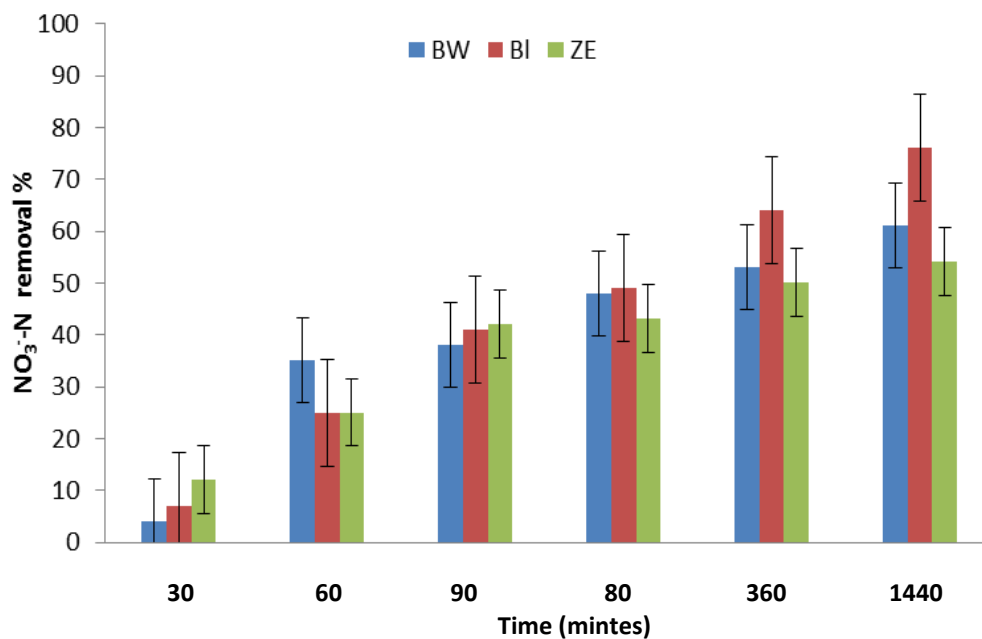


Fig. 3.7. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 1.0 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent.

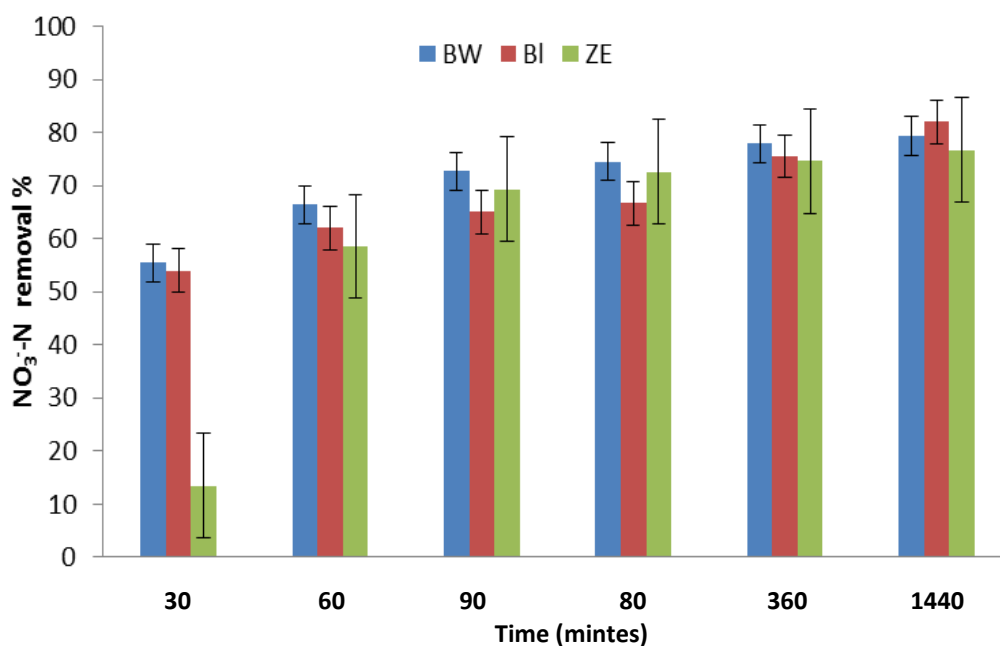


Fig. 3.8. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 1.5 mM l^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent.

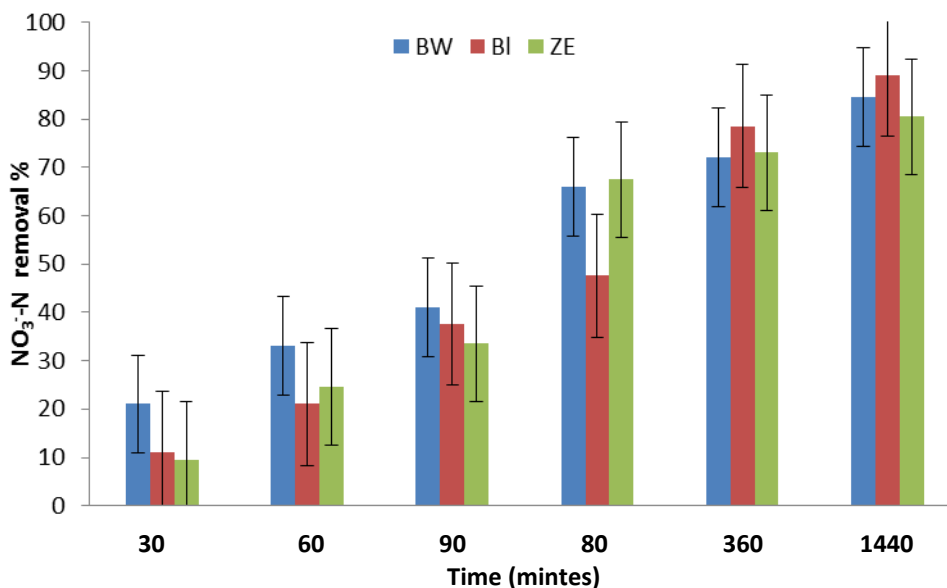


Fig. 3.9. (NO_3^- -N) removal percent by (ZE, BI and BW at initial conc: 2 mMl^{-1} , contact time (30, 60, 90, 180, 360 and 1440 min) and solid /liquid ratio 0.333 g/30 mL) and error bars represent

In here's in most sorption media's inability To perform efficiently at higher pH, concerning nitrate removal, significant reduction in media performance at higher neutral and alkaline pH (6.5- 8.5) which is typical of natural systems such as groundwater (Chatterjee and Woo, 2008; Chatterjee *et al.*, 2009). A media such as three studied adsorbent material that's their performance of nitrate removal does not deteriorate with pH increase can be effectively used with different magnitudes among them. Meanwhile, the pH of the studied system (solution/Solid) after sorption was found to increase when the initial pH was acidic and *vice versa*. Such effects could be due to the buffering action of the three studied adsorbent materials indicating that serpens normally shift solution pH towards their initially naturally pH or their point of zero charge (pH_{zc}).

Conclusion

Olive wood- derived biochar (BI) used under the current batch experiment conditions reveals high efficient capability with respect to nitrate removal from aqueous solutions. On the other hand,

pyrolysis of local olive wastes fewer than 350 and 60 min. with oxygen limited conditions resulted in height removal of nitrate compare to modified zeolite. Hence, the obtained results indicate that pre-pyrolysis of such local olive residues must be impregnation to produce modified biochars with a dual capability of removing nitrate ions from aqueous solutions.

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المخلص العربي

ازالة النتترات من المحاليل المائية باستخدام بعض المواد الممتزة الصديقة للبيئة

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تهدف الدراسة الحالية إلى إزالة النتترات من المحاليل المائية بمواد امتصاص صديقة للبيئة لإزالة النتترات من المحاليل المائية، بواسطة استخدام مواد سهلة الامتزاز لإزالة النتترات من الماء، وتم تقييم المواد المستخدمة كوسيلة امتزاز محتملة لهذا الغرض وتمت دراستها باستخدام تجارب دفعية في معمل كلية العلوم الزراعية البيئية - جامعة العريش. لذلك، تم استخدام الفحم الحيوي الناتج من كلا من مخلفات اشجار الزيتون (BI) وكذلك تفل الزيتون (BW) والزيوليت المعدل (ZE) كمواو ممتزة. تمت دراسة تأثير تركيزات النتترات الأولية (1، 1.5، و2 ملي مول / لتر) في أوقات التلامس (30، 60، 90، 180، 360 و1440 دقيقة ودرجة الحموضة (4، 7 و10) على إزالة النتترات في التجربة المعملية. سجلت النتائج ان الفحم المشتق من بقايا اشجار الزيتون (BI) اعطي أعلى نسب إزالة للنتترات 98.9% عند التركيز 2.0 ملي مولار/ لتر، مقارنة مع الاخرين (BW) و (ZE) المحضرين في التجربة الاولى. بينما في تجربة الحموضة الثانية الأس الهيدروجيني 4 لتركيز النتترات العالي سجلت جميع المواد الممتزة المدروسة بمقادير مختلفة أعلى نسب إزالة للنتترات (99.9% BI و 94.8% BW و 90.4% ZE). في نفس الوقت. كانت معدلات إزالة النتترات منخفضة خلال الفترة الأولية للتجربة ثم كان المعدل أعلى. تقترح هذه الدراسة أنه يمكن إزالة النتترات من الماء باستخدام الفحم الحيوي. ، يؤثر تركيز نتترات الماء الأولي ووقت التلامس ودرجة الحموضة على فعالية المواد الماصة مثل مواد إزالة النتترات من الماء.

الكلمات الإسترشادية: الزيوليت، البيوتشار، ازالة النتترات، المائي.

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