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ORIGINAL ARTICLE

Evaluation and improvement of the WWTP performance of an agricultural cooperative by adsorption on inert biomaterial: case of orthophosphate, nitrate and sulfate ions

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Abstract

The study is carried out at the wastewater treatment plant (WWTP) of an agricultural cooperative that operates according to the activated sludge process. Dairy industry is enlisted as one of the top-most industries in the food industry. Dairy wastewater treatment is a big issue as dairy wastewater releases a high amount of chemical oxygen demand, inorganic and organic particles, biological oxygen demand, and nutrients. But, these processes partly degrade wastewater containing fats and nutrients as dairy wastewater. The aim of this study was to evaluate the purification performance of this treatment process. The qualitative analysis of decanted raw wastewater (DRWW) and purified wastewater (PWW) shows that the concentration of orthophosphate, nitrate and sulfate ions is slightly higher. Such contaminated water if not handled appropriately, it pollutes water bodies and largely affects our ecosystem and biodiversity. Hence, our proposal is to improve the WWTP performances by using the adsorption process onto dried *Carpobrotus edulis* as an inert biomaterial. This adsorption process is recognized as one of the best water treatment techniques, more and more works are oriented towards the search for new materials, cheaper and having a good adsorbent potential. This study opens the path for the use of natural and abundant local material to remove orthophosphate, nitrate and sulfate ions using the *C. edulis* plant particles shred. The surface micromorphology of the biomaterial was investigated using a scanning electron microscope; while the qualitative element composition was analyzed using energy dispersive X-ray and infrared spectroscopies. The found results of DRWW was about 57% for orthophosphates, 67% for sulfates and 73% for nitrates ions. For PWW, the percentage removal was found to be 62%, 73% and 84% for orthophosphates, sulfates and nitrates respectively. These results indicate that dried *C. edulis* plant, as an environmentally friendly adsorbent could be recommended for the removal of mineral pollutants. In conclusion, the *C. edulis* adsorbent can be integrated into the activated sludge process for wastewater treatment after identifying the optimal hydraulic loads, associated sizes, and shapes in continuous operations.

Keywords Wastewater · Activated sludge · Adsorption · *Carpobrotus edulis* · Orthophosphates · Nitrates · Sulfates

List of symbols

WWTP	Wastewater treatment plant
RWW	Raw wastewater
DRWW	Decanted raw wastewater
PWW	Purified wastewater
COD	Chemical oxygen demand
BOD_5	Biochemical oxygen demand
TSS	Total suspended solids
C. edulis	*Carpobrotus edulis* Plant

Introduction

In the world, the role played by agro-industries is important for the food security of populations that are growing exponentially. However, the damage caused by their liquid discharges is a problem for the preservation of the environment (Noukeu et al. 2016).

Many agri-food industries produce a very high pollution load (Wu et al. 2011). This high pollution often leads to the

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death of marine organisms and the depletion of dissolved oxygen (Li et al. 2011).

The region of Souss Massa (South Morocco) has recently experienced a notable industrial development. This industrial sector has become diverse in the region; it is not dominated by the agro-industry anymore. This sector relies heavily on the groundwater for the water supply. Hence, the protection of these vital water resources has become a necessity given the increase of the untreated wastewater.

The agricultural cooperative in Taroudant (South Morocco) is active in several fields starting from agricultural and dairy production to processing these products. According to previous studies, the manufacture of dairy products generates a large amount of wastewater (Birwal et al. 2017).

In order to protect the environment, the company has built its own WWTP, which aims to produce clean water for irrigation. The purification process adopted in this industrial unit is based on an activated sludge system. This system is recommended to treat Dairy wastewater using biological methods (Lateef et al. 2013).

The parameters that were the subject of this study are the major parameters of pollution (COD, BOD_5, TSS, nitrates, sulfates, and orthophosphates) and the parameters measured on the spot, namely: air temperature; water temperature; pH; electrical conductivity and dissolved oxygen.

From the characterization of the studied wastewater, it appears that the organic matter and pollutants studied must be scrupulously removed from the wastewater before it is discharged into the natural environment in order to avoid eutrophication and the release of H_2S (Ge et al. 2010; Afridi et al. 2019). Thus, suitable treatments for wastewater are required to use effective disposal methods.

Our work has a double objective. On the one hand, it consists in evaluating the purification performances of this station, and on the other hand, to proceed to the evaluation of the potential of dried *Carpobrotus edulis* plant to eliminate the studied pollutants. Because the purified water from the WWTP is no longer useful as it has a very high amount of contaminants, which make it non-recyclable.

Preferably, a new low-cost solution is needed so that the activated sludge process can be improved to effectively remove the pollutants being studied. In Morocco, many studies have addressed this issue. However, a few studies have been carried out with the aim of improving the purification performance of WWTP in Morocco using the adsorption technique (Abali et al. 2018).

However, adsorption efficiency and interaction mechanisms are dependent on the nature of the adsorbent used, and on the process conditions (Paunovic et al. 2019). This method is normally treated as an alternative approach to the conventional wastewater treatment (Anastopoulos et al. 2017).

The process described in this work is simple, inexpensive and easy to extrapolate to a larger scale for practical application to wastewater treatment. It consists in using the microparticles obtained from dried *C. edulis* plant which are brought in contact with studied anions.

Materials and methods

Description of the WWTP

The purification process adopted in the industrial unit is based on an activated sludge system. The raw wastewater is first subjected to a pre-treatment carried out by a rotary screen, followed by a primary treatment by coagulation-flocculation and flotation. The secondary treatment is carried out by the activated sludge system rich in bacteria.

Sampling

The sampling method adopted is a composite type of 24 h during the period of activity of the industrial unit (J.O N°. 1607-06, 2006). The sampling points are located at the inlet and the outlet of the WWTP in order to evaluate the purification efficiency.

For the adsorption study, the raw wastewater was decanted for 2 h, followed by filtration to remove impurities and suspended solids. In the remainder of this work, raw wastewater is marked "RWW", decanted raw wastewater "DRWW" and purified wastewater "PWW". The samples were collected in polyethylene bottles (Rodier et al. 2009).

Wastewater characterization parameters studied

The temperature of the air is measured by a thermometer of the Alla France type and the temperature of the water and the pH by a pH-meter type WTW pH 3310 SET (Rodier et al. 2009). Electrical conductivity is determined by a conductivity meter type WTW Cond 3310 SET (Rodier et al. 2009). Dissolved oxygen is followed by an Oximeter type WTW Oxi 3310 SET (Rodier et al. 2009). Chemical oxygen demand (COD) is determined by the colorimetric method) (CEAEQ, MA. 315 - COD 1.1). Biochemical Oxygen Demand DBO_5 by the electrometric method (CEAEQ, MA. 315 - DBO 1.1.) and Total suspended solids TSS by the gravimetric method (CEAEQ, MA 104 – S.S 1.1). Nitrate, Sulfate and Orthophosphate concentrations are measured by the cadmium reduction method (Rodier et al. 2009), the nephelometric method (Turbidimeter type IR Orbeco-Hellige) (Rodier et al. 2009) and by the spectrometric method of molecular absorption (Rodier et al. 2009).

Preparation of the natural adsorbent

The adsorbent used is based on the plant *C. edulis* which belongs to the family Aizoaceaes. *C. edulis* plant was collected from the semi-arid zones of Morocco (Agadir zone). The different parts of this plant (leaves and stems) are dried, crushed and sieved. Before use, samples were cleaned several times with water to remove sand, mud and other particles. Afterward, they were air-dried for 2–3 days and then ground and sieved using 500 µm sieves and used as such without any pre-treatment. The picture of the plant is shown in the figure below (Fig. 1). The adsorption study of orthophosphate, sulfate and nitrate ions was carried out under static conditions.

Batch adsorption studies

The study of the batch adsorption consists in introducing a mass m of the adsorbent into a volume V of the wastewater at an initial concentration of ion C_i. The whole solution is stirred at a constant temperature of 25 °C by a thermostat. Samples were taken at different contact times and then centrifuged at 500 rpm and analyzed. The ratio $R = \text{mass biomaterial/Volume of the solution}$ ($R = m/V$) used in our study corresponds to the smallest mass of biomaterial which leads to a maximum adsorption rate. In the batch adsorption experiments, 40 mL of wastewater solution with a given ions concentration, C_i, was mixed with 1 g of biomaterial without any pre-treatment (Chiban et al. 2011).

The concentrations retained C_r (mg/L) by the equilibrium biomaterial and the quantities of adsorption Q_{ads} (mg/g) are given by the following relationships:

$$C_r(\text{mg/L}) = C_i - C_{eq}, \quad Q_{ads}(\text{mg/g}) = \left(C_i - C_{eq}\right) \times \frac{V}{m}$$

Where m: mass of biomaterial in g, V: total volume in (L), C_i: initial concentration (mg/L), C_{eq}: equilibrium concentration in (mg/L), Q_{ads}: amount adsorbed per 1 g of biomaterial (mg/g).

The percentage removal X (%) was calculated as follows (Nehra et al. 2018):

$$\%\text{Removal} = \left[(C_i - C_{eq})/C_i\right] \times 100$$

Determination of pH_{pzc} of C. edulis

The point of zero charge (PZC) was determined using the solid addition method (Banerjee et al. 2013), viz: For the determination of pH_{pzc} of the adsorbent, 50 mL of 0.01 M NaCl solutions were taken in different Erlenmeyer flasks of 250 mL and 0.5 g of adsorbent was introduced in each of them. Now, pH values of these solutions were adjusted in 2–12 range by 0.1 M HCl/NaOH solutions. These flasks were kept for 48 h, and the final pH of the solutions was measured. Graphs were plotted between pH_{final} versus $pH_{initial}$. The point of intersection of the curve of pH_{final} versus $pH_{initial}$ was recorded as pH_{pzc} of *C. edulis*.

Adsorbent characterization

The surface morphology of *C. edulis* particles was studied by scanning electron microscopy (SEM, HITACHI S-4500). The elemental analysis was analyzed by SEM coupled with energy dispersive X-ray analysis (SEM/EDX, LEICA-S-260). The surface functional groups were detected by Fourier transforms infrared (FT-IR) spectrometer (Spectrum 100 Perkin Elmer). A KBr pellet containing the sample was used for the FT-IR spectroscopic



Fig. 1 Picture of *C. edulis* plants

measurements, and the spectra were collected in the scan range between 500 and 4000 cm^{-1}.

Results and discussion

In the first item, the results of the evaluation of the purification performance of the WWTP are presented with an inherent discussion. In the second item, a special application of this biomaterial in the adsorption of the studied anions contained in real wastewater.

Characterization of the adsorbent

The electron micrograph and EDX spectra of *C. edulis* particles, which were used as an adsorbent, are shown in Figs. 2 and 3. This micrograph reveals clearly the appearance of grains and organic fibers on the surface of *C. edulis* particles. It has been shown that the ground material is very porous with adversity in the size and texture of the grains. So, based on the morphology, as well as on the presence of high amounts of amino acids and tannins (Chiban et al. 2011), can be concluded that this material presents an adequate morphological profile to retain the anions and metal ions. EDX spectra showed the presence of O, C, Ca, Mg, Au, Cl, K, and Mo in the adsorbent. These elements are the main constituents of proteins, polypeptides, phenolic compounds, alkaloids, including functional groups (–COOH, –NH, –OH), which are totally or partially involved in ion retention (Kurniawan et al. 2006).

Moreover, Infrared spectroscopy provides information on the chemical structure of the adsorbent material (Fig. 4). Figure 4 shows typical absorption bands of the biomolecules. Hydroxyl groups (OH) are represented by a broadband at 3400 cm^{-1} and absorption of these groups indicates an attribution to the hydrogen bonds. Stretching vibration corresponds to C–H is characterized by absorption in the region of 2900 cm^{-1}. The absorption bands in the region of 1600 and 1700 cm^{-1} correspond to carbonyl groups stretching vibrations. The absorption bands in the region of 1300 cm^{-1} and 1060 cm^{-1} correspond to amines vibration and R–OH. The adsorption by *C. edulis* particles might be attributed mainly to their surface, which contains anion binding functional groups such as carboxyl, hydroxyl and amino groups.

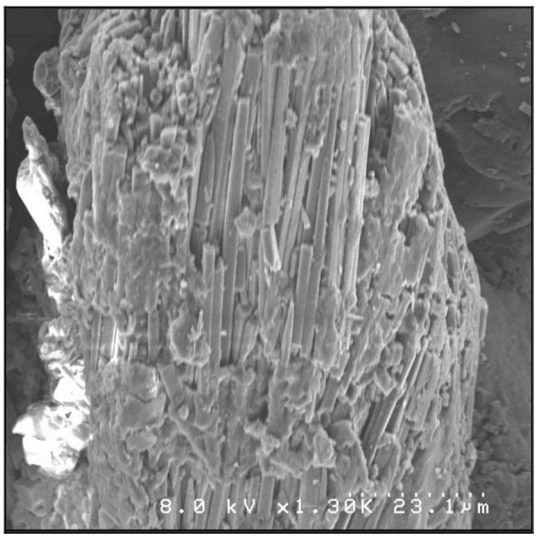


Fig. 2 SEM image of *C. edulis* particles

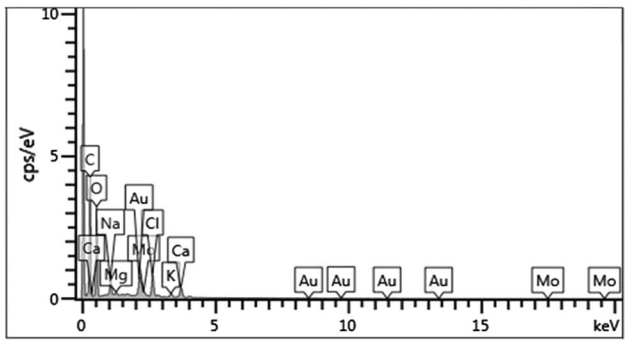


Fig. 3 EDX spectrum of *C. edulis* particles

Characterization of the wastewater studied

In order to compare the purification performance amelioration of the solid inert biomaterial with the activated sludge process adopted. A study of the polluting load of wastewater used was carried out during 2018, the table below (Table 1) shows the average values of physico-chemical parameters of the raw wastewater (RWW), raw wastewater decanted (RWWD), and purified wastewater (PWW) of the WWTP. Its results are reproducible and verifiable.

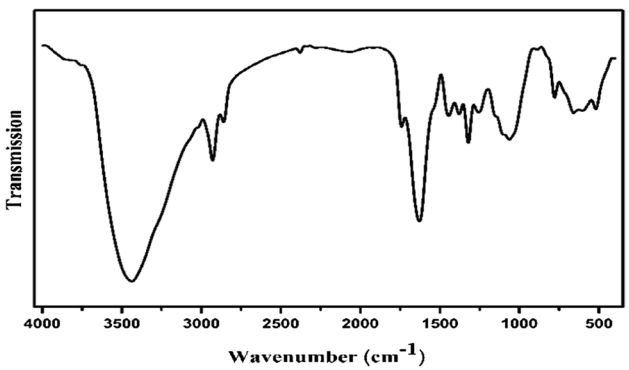


Fig. 4 Infrared spectrum of *C. edulis* particles in KBr

Table 1 Pollution load of wastewater used from WWTP and Moroccan standards in PWW

Parameters	Water type	Values	Moroccan standard
T (°C) of water	RWW	20.5	35 (J.O N° 1276-01, 2002)
	PWW	21.6	
pH	RWW	11,05	6.5–8.4 (J.O N° 1276-01, 2002)
	PWW	7.75	
Electrical conductivity (µs/cm) at 20 °C	RWW	3630	2700
	PWW	2530	
Dissolved oxygen (mg O_2/L)	RWW	0	–
	PWW	4.3	
TSS (mg/L)	RWW	940	150 (J.O N° 1607-06, 2006)
	PWW	118	
BOD_5 (mg O_2/L)	RWW	3995	120 (J.O N° 1607-06, 2006)
	PWW	110	
COD (mg O_2/L)	RWW	6686	250 (J.O N° 1607-06, 2006)
	PWW	429	
PO_4^{3-} (mg P/L)	DRWW	38	10 (J.O N° 1276-01, 2002)
	PWW	16	
NO_3^- (mg N/L)	DRWW	7.7	30 (J.O N° 1276–01, 2002)
	PWW	51	
SO_4^{2-} (mg/L)	DRWW	384	250 (J.O N° 1276-01, 2002)
	PWW	333	

For purified wastewater, the temperature is an important biologically significant factor, which plays important role in the metabolic activities of the organism. The maximum value found for purified wastewater is about 21.6 °C. This value respects the limit value of the standard. The average pH value of the purified wastewater is 7.75 and was within the limit prescribed by the standard for the quality of water intended for irrigation. On the other hand, the electrical conductivity, reflects the overall degree of mineralization, and provides information on the salinity level. According to the results obtained, the average value of the electrical conductivity of the treated wastewater is about 2530 µs/cm.

For dissolved oxygen, concentration increases from the inlet to the outlet of the WWTP. During the study period, the dissolved oxygen concentration did not exceed 4.3 mg/L. The qualitative analysis of raw wastewater (RWW) shows a very high organic load with COD, BOD_5 and TSS values equal to 6686 mg O_2/L, 3995 mg O_2/L and 940 mg/L, respectively. Similarly, previous studies have confirmed the results found (Şentürk et al. 2010; Qasim et al. 2013). In addition, the dairy industry produces organic wastewater characterized by both biochemical oxygen demand (BOD_5) and COD values of up to tens of thousands of mg/L (Loperena et al. 2006; Ardley et al. 2019; Shams et al. 2010). These high concentrations of organic matter have a significant impact on receiving environments (Magdalena et al. 2019). Furthermore, the BOD value in the wastewater was lower than the COD value indicating the presence of biodegradable organic compounds. For the purified wastewater (PWW), the BOD_5 and TSS contents are in conformity with the standard of discharge in the receiving environment (J.O N° 1607-06, 2006). However, the COD value is slightly higher.

The mineral ions analyzed (PO_4^{3-}, NO_3^- and SO_4^{2-}) exceed the values set by Moroccan standards (J.O N°1607-06, 2006; J.O N°1276-01, 2002). The presence of these ions was confirmed because of the use of sanitizers, detergents, or some kind of cleaning agents for the cleaning of machinery or equipment. According to previous studies, nutrient removal by the activated sludge system was less important (Kaur 2021). The residual concentrations of these mineral ions could favor the eutrophication phenomenon and the transformation of SO_4^{2-} to H_2S ions, which have a negative impact on the surrounding population and the environment (Hao et al. 2014; Ren et al. 2014).

Point of zero charge of C. edulis

The study of the influence of pH on adsorption is very important because it affects the surface charge of adsorbent and the degree of ionisation of the adsorbate (Vishwakarma et al. 2018).

The point of zero charge of *C. edulis* was determined by solid addition method. In this study, the pH_{pzc} of *C. edulis* was found to be 7.4 (Fig. 5).

At a pH of the solution below $pH_{pzc,}$ the surface of *C. edulis* is positively charged and can attract anions from the solution. When the solution pH is greater than $pH_{pzc,}$ the

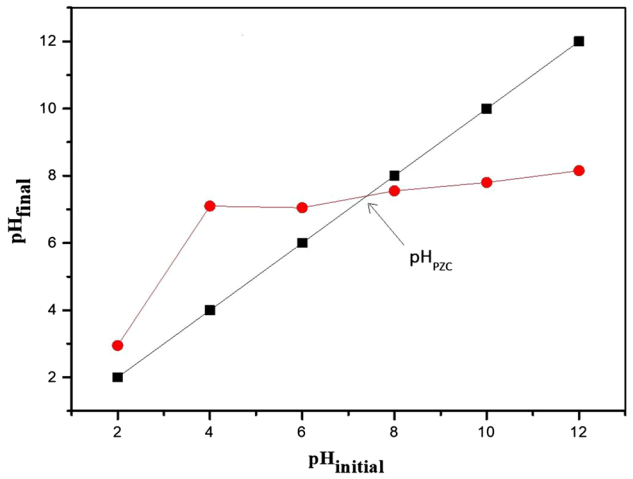


Fig. 5 pH at the point of zero charge (pH_{pzc}) for *C. edulis*

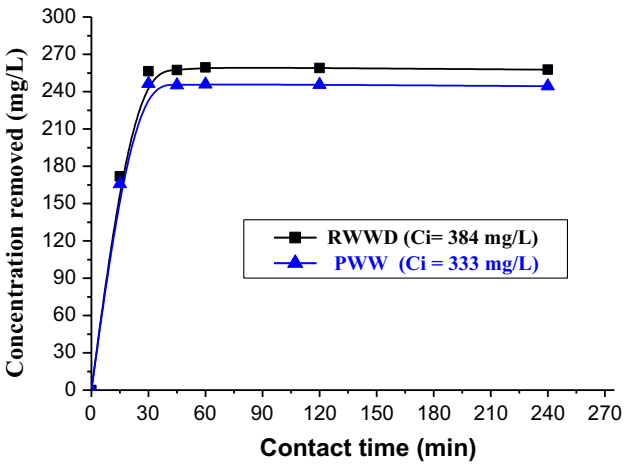


Fig. 6 Effect of contact time of sulfate ions removal by *C. Edulis* for DRWW and PWW

surface is predominated by negative charges and can attract cations contained in wastewater.

Thus, at pH < 7.4, the surface has a high positive charge density, so the uptake of negatively charged *C. edulis* would be high (Shi et al. 2019). For *C. edulis*, pH below pH_{pzc} would be beneficial for the absorption of the anions studied. Several studies have reportedly shown that electrostatic attraction may play some role in the adsorption of anions (Fu et al. 2018).

However, when pH > 7.4, the negative charge of the adsorbent increased, and the adsorption of anions began to weaken due to the electrostatic repulsion. Additionally, with the increase of pH, the hydroxyl group competed with anions for active sites on the surface of *C. edulis*. At high pH values, the concentration of OH- far exceeds that of the mineral ions.

In addition, the pH of PWW is slightly low (pH = 7.75) compared to the pH of RWWD (pH = 11.05), which implies that the adsorbent surface in the first case will be less charged to OH- ions, which can compete with anions due to the effect of the negative charge.

Adsorption study of orthophosphate, nitrate and sulphate ions

In order to improve the purification efficiencies of WWTP, we have studied the removal of mineral ions orthophosphates (PO_4^{3-}), nitrates (NO_3^-), and sulfates (SO_4^{2-}) by adsorption on a solid inert biomaterial, Plant origin: the crushed plant *C. edulis*. This study concerns decanted raw wastewater (DRWW) and purified wastewater (PWW). Figures 6, 7 and 8 show the variation, as a function of the contact time (R = 25 g/L, T = 25 °C), of the concentration retained by the *C. edulis* plant, respectively in orthophosphate ions (PO_4^{3-}), nitrates (NO_3^-) and sulfates (SO_4^{2-}).

The analysis of these curves (Figs. 6, 7, 8) shows that the concentration retained increases with the contact time to reach the saturation stage corresponding to the adsorption equilibrium. The results of the adsorption study of the studied anions (PO_4^{3-}, SO_4^{2-} and NO_3^-) are the average of twelve successive reproducible and verifiable tests.

According to Figs. 6 and 7, the adsorption of sulfate and nitrate ions is relatively rapid: the adsorption equilibrium is reached after a contact time of about 30 min for the sulphate ions and 45 min for the nitrate ions. Moreover, the shape of the curves does not make it possible to distinguish two distinct stages of adsorption; everything happens as if the adsorption takes place in a single very rapid step from the very first minutes of contact. This step corresponds to the retention of ions located near the surface of the mill

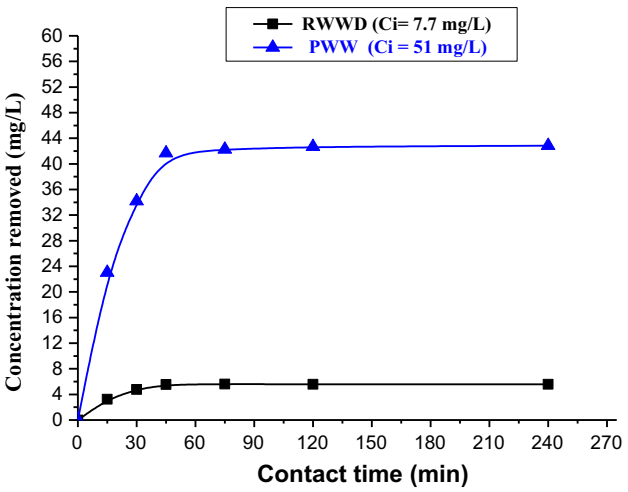


Fig. 7 Effect of contact time of nitrate ions removal by *C. Edulis* for DRWW and PWW

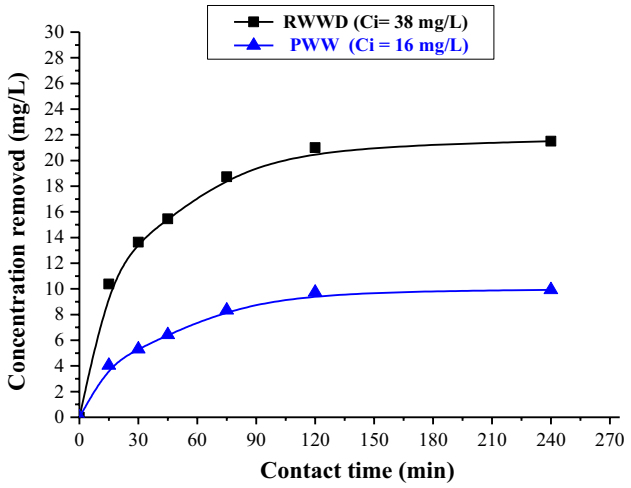


Fig. 8 Effect of contact time of orthophosphate ions removal by *C. edulis* for DRWW and PWW

particles, and consequently to a rapid diffusion of sulfate and nitrate anions to the external sites of the adsorbent surface.

For purified wastewater, it appears that the retained C_r concentration of sulfate and nitrate ions increases with the contact time. Like the raw wastewater adsorption study, equilibrium is quickly reached after about 30 min for sulfate ions and 45 min for nitrate ions, respectively.

On the other hand, the adsorption of orthophosphate ions is relatively slow; the adsorption equilibrium is reached after about 2 h. The analysis of the figure (Fig. 8) reveals that the retained concentration C_r of phosphate ions contained in the raw and purified wastewater does not make it possible to distinguish two distinct stages of adsorption, everything happens as if the adsorption takes place in a single stage. This adsorption could be explained by the rapid diffusion of phosphate ions to the external sites on the surface of the adsorbent. After the external surface reaches the saturation, phosphate ions start to penetrate into the pores of adsorbent and the internal adsorption occurs, and finally, the adsorption reaches the equilibrium. This result can be explained by the specificity of *C. edulis* particles – anion interactions and by the form of the studied anions such as: PO_4^{3-} has tetrahedral form.

For all three curves, the additional increase in time did not substantially affect the concentration of anions at equilibrium.

The quantitative results of the adsorption are given in Table 2, which shows the initial concentrations before adsorption (C_i) and the concentrations and adsorbed quantities, expressed in C_r and Q_{ads} ($Q_{ads} = C_r.\ V/m = C_r/25$).

In addition, this table (Table 2) also presents the removal percentages of the studied ions contained in the raw settled wastewater (RWWD) and the purified wastewater (PWW).

Table 2 Percentage removal of studied ions at adsorption equilibrium in RWWD and PWW

			PO_4^{3-}	SO_4^{2-}	NO_3^-
DRWW	Before adsorption	C_i (mg/L)	38	384	7.7
	After adsorption	C_r (mg/L)	21.51	257.65	5.59
		% Removal	56	67	72
		Q_{ads} (mg/g)	0.86	10.30	0.22
PWW	Before adsorption	C_i (mg/L)	16	333	51
	After adsorption	C_r (mg/L)	9.92	244.4	42.84
		% Removal	62	73	84
		Q_{ads} (mg/g)	0.4	9.78	1.71

For WWTP, the removed concentration (C_r) of the orthophosphate, sulfate and nitrate ions are respectively 21.51 mg/L, 257.65 mg/L and 5.59 mg/L corresponding to percentages of elimination at the equilibrium of about 56%. 67% and 72%. With regard to purified wastewater (PWW). The improvement yields of biomaterials are of the order of 62%. 73% and 84%, respectively for the orthophosphate, sulfate and nitrate ions.

The difference in the retained quantities (Q_{ads}) for each of these ions may be due to the difference in initial concentration and to the specificity of physicochemical interactions between each ion and the adsorption sites of biomaterial.

Generally, wastewater is composed of several coexisting components, such as anions, heavy metals, and organic compounds, which may influence the adsorption process. In addition, the direct competition between the studied anions and metal anions for adsorption sites reduced the adsorption efficiency. Furthermore, metal anions exert a stronger inhibition effect than metal cations (Lee et al. 2017).

Conclusion

Aerobic treatment is most widely used for treating dairy industry wastewaters; since they can treat large volumes of influents in a relatively short period. Nevertheless, these processes partly degrade wastewater containing fats and nutrients as dairy wastewater. Therefore, further treatment is essential for anaerobically treated dairy wastewater.

The current study reveals that the treatment process discussed can play a very pronounced role in the treatment of nutrient anions contained in agri-food wastewater.

The purification yields recorded by the WWTP studied in terms of the major parameters of pollution are satisfactory; and the contents of mineral ions. Namely PO_4^{3-}, SO_4^{2-} and NO_3^-. Exceed the limit values of the releases in the receiving environment. For this purpose, the residual concentrations remain very high in the treated effluent and could

constitute a great risk of eutrophication for releases in the natural environment.

This experimental study using a real wastewater showed that the removal of mineral ions orthophosphates (PO_4^{3-}), nitrates (NO_3^-) and sulfates (SO_4^{2-}) by adsorption on the ground material of the *C. edulis* plant enables to improve the purification yields in a significant way. The elimination percentages are ordered as 62%, 73% and 84%, respectively for the orthophosphate, sulphate and nitrate ions at the outlet of the WWTP. Given its low cost and high retention rate. The biomaterial used could constitute a potential adsorbent for the mineral pollutants studied in the wastewater treatment industry. Moreover, the fast adsorption kinetics observed for *C. edulis* waste represents a key advantage for the design of a complementary wastewater treatment system.

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Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

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