



Atrazine removal in aqueous solutions using activated carbon from peach stone

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ABSTRACT

Activated carbon is commonly used as a material for contaminant-adsorption processes in aqueous systems. However, its use is more restricted to charcoal than to coal, for the most part, in view of the fact of the higher cost (~ 40%) if the mineral is a fossil fuel which needs to be extracted from the earth by mining. For this reason, the peach stone that comes from alimentary industrial tailings can be a good choice for the separation of pollutants from aqueous suspensions and other soluble substances. The purpose of this research was the development of a low-cost filter, using stones to remove atrazine from water. Appraisal and characterization studies were performed along with batch experiments to investigate dosing effects of the activated carbon, atrazine concentration, contact time, and adsorption pH on removal procedures. From the results of the experiment, an excellent removal of the analyte in question was observed under conditions that can be considered as close as possible to the environment, such as pH = 6.5, room temperature and 10 minutes of agitation time, always choosing the best alternative with the lowest cost of energy and time. Batch system application has been recommended as versatile for utilization in seasonal problems such as pesticide contamination.

Keywords: adsorption, biomass, pesticide, water treatment.

Remoção de atrazina em meio aquoso usando carvão de caroço de pêssigo

RESUMO

Carvão ativado é um material que desponta para o uso da adsorção de contaminantes em águas, no entanto seu uso é mais restrito ao carvão vegetal do que ao mineral, principalmente devido ao seu maior custo (~ 40%), uma vez que é um combustível fóssil extraído da terra através da mineração. Assim, o caroço de pêssigo, proveniente de rejeitos de indústrias alimentícias, pode ser uma boa alternativa para a remoção de contaminantes em meio aquoso. O objetivo deste estudo foi o desenvolvimento de um carvão ativado de baixo custo proveniente de caroços de pêssigo para a remoção de atrazina em meio aquoso. Estudos de caracterização e experimentos em batelada foram realizados para investigar os efeitos de dosagem do carvão, concentração de atrazina,



tempo de contato e pH do meio de adsorção, na remoção de atrazina. A partir dos resultados do delineamento experimental, observou-se excelente remoção do analito em condições próximas a do meio ambiente, como pH = 6,5, temperatura ambiente e tempo de agitação de 10 minutos, buscando sempre um menor custo de energia e tempo. A aplicação por sistema de batelada foi sugerida como versátil para aplicação em problemas sazonais, como a contaminação por pesticidas.

Palavras-chave: adsorção, biomassa, pesticide, tratamento de água.

1. INTRODUCTION

Since consumption and demand for food have increased, the use of pesticides increased in agriculture so that production could be improved. Also, agrochemical application has generated major environmental complications, such as contamination of the public water supply. Consequently, inappropriate disposal of these organic compounds in nature tends to increase their already existing concentrations in soil and in aqueous media, making them toxic to all living beings with whom they come in contact (Ghosh and Philip, 2006). In this context, Brazil is one of the nations use pesticides the most (Fao, 2014), as a result of agricultural expansion. Currently, the atrazine is one of the most commonly used agrochemicals in the country.

Atrazine (ATZ) is among the most widely used herbicides in the world, and it has been detected in high concentrations on the surface and water table. ATZ is characterized by its wide application, high persistence in different media, capability to produce effects in the neuroendocrine and reproductive systems. Some scientific studies have begun to investigate the genetic mechanisms of toxicity; however, studies have also demonstrated that epigenetic mechanisms are limited (Wirbisky *et al.*, 2016; Martins-Santos *et al.*, 2018).

Atrazine is a suspected EDC (endocrine-disrupting chemical), and its intensive use and low biodegradability has led to the accumulation of this compound in the environment, contaminating surface water and groundwater (Schleder *et al.*, 2017). There are several methods that can be used in water/effluent treatments containing pesticides, such as, adsorption in activated carbons, biological treatment, oxidation processes (Ozone, Hydrogen Peroxide, Chlorine, Ultraviolet Irradiation) and advanced oxidative processes (Photocatalysis, Fenton Reagent, Combined $O_3/H_2O_2/UV$ Systems) (Mudhoo and Garg, 2011).

The adsorption of organic compounds using activated carbon (AC) is one of the most important technologies indicated for the industrial effluent treatment. AC is a porous adsorber that can be obtained from different carbonaceous raw materials. In addition, it has a large surface area containing a variety of functional groups, responsible for its adsorption power (Lima *et al.*, 2014; Mandal and Singh, 2017; González-García, 2018).

AC is typically used to adsorb taste and smell from organic compounds, in addition to synthetic chemicals in potable water treatment procedures (USEPA, 2011). The two main types of activated carbon used in the purification processes are the GAC and PAC (respectively granular and powdered carbon). The principal attribute that distinguishes the GAC from PAC is the size of its particle. The GAC normally has its diameter varying from 1.2 mm to 1.6 mm. In Brazil, the treatment plants are not structured for the continuous use of powdered carbon; therefore, even when the waters are treated, they exude a characteristic ground smell and release taste; also, treatment facilities make use of pulverized carbon as a method to remediate the problem.

There are many public health problems that can be caused by the presence of ATZ in the potable water; besides that, conventional water treatment is limited and is not capable of the removal of these kinds of contaminants. Thus, the objective of this work was to evaluate the ability to reduce the concentration of ATZ in aqueous solutions by using an alternative activated

carbon. The AC was produced from peach stone coming from food industries, where they were discarded on a large scale.

2. MATERIALS AND METHODS

2.1. Adsorbent Material, equipment and reagents

The raw material used was obtained from the carbonization, activation and pulverization of the peach stone *in natura*. The carbonization processes were performed using controlled burning in conventional refractory furnaces, air convection (oxygen as an oxidant agent) and high temperatures (from 260°C to 432°C). The carbonized peach stone activation was made by using steam and air (oxygen) in a refractory furnace at high temperatures (above 600°C), in a reaction time of about 4 hours.

This material was kindly provided by AlphaCarbo Ltda. The quantification of ATZ was made by using spectrophotometry in the ultraviolet region at 222 nm (Spectrum Meter, Model SP-2000 UV) with a 10 mm optical path quartz cuvette (ASTM, 2014). Adsorption studies were performed using a shaker table (Tecnal TE-420). The analytical standard of ATZ from Sigma-Aldrich was also used. Natural water samples were collected and filtered by 0.45 µm membrane to remove suspended particulate matter, then different concentrations of ATZ were added for the evaluation of the adsorptive capacity of the peach stone activated carbon- PSAC.

2.2. Characterization of peach stone activated carbon

The peach stone activated carbon characterization was succeeded by the analysis of some physical and chemical properties, such as: porosity (NBR 9165, ABNT, 1985), volatiles content (Adad, 1982), iodine number (D 4607-94, ASTM, 1999), caramel adsorption (JIS K 1474, JSA, 2007) and fixed carbon (Adad, 1982). The PSAC morphology was investigated by a scanning electron microscopy (SEM) using the Hitachi High Tech TM 3000 device attached to an EDS Swif ED 3000 at 15Kv.

2.3. Evaluation of ATZ removal efficiency in aqueous medium

Studies have been developed involving pH, temperature, contact time, mass and adsorptive capacity of PSAC in order to obtain the best condition for the removal of ATZ in aqueous medium. In order to evaluate the required mass of the PSAC to remove ATZ from the water solutions, numerous experiments were performed (D3860-98, ASTM, 2014). Different dosages of PSAC (0.001 to 0.005 g) were added in water samples (50 mL) with varied concentrations of ATZ (200 to 600 µg L⁻¹) in contact with different masses of PSAC (0.001 to 0.005 g), and it was maintained under constant stirring for 2 hours. The adsorptive capacity of the ATZ by the PSAC was calculated according to Equation 1.

$$q = (C_o - C_e) \frac{V}{M} \quad (1)$$

Where:

q = carbon adsorptive capacity (µg ATZ g⁻¹ carbon);

C_o = initial concentration (µg L⁻¹);

C_e = concentration at equilibrium (µg L⁻¹);

V = volume (L);

m = activated carbon mass (g).

The best conditions for the adsorption of ATZ (400 µg L⁻¹) by PSAC (m=0.0025 g) were investigated by using an experiment design 2³, which was organized through the variables of pH, temperature and stirring time, in three levels (lower, upper and central) as per Table 1.

Table 1. Results obtained from experimental design 2³.

Experiment	Experimental conditions
1 st	pH = 8.00; 50°C; 60 min
2 nd	pH = 4.00; 50°C; 60 min
3 rd	pH = 8.00; 25°C; 60 min
4 th	pH = 4.00; 25°C; 60 min
5 th	pH = 8.00; 50°C; 10 min
6 th	pH = 4.00; 50°C; 10 min
7 th	pH = 8.00; 25°C; 10 min
8 th	pH = 4.00; 25°C; 10 min
9 th	pH = 6.00; 35°C; 30 min
10 th	pH = 6.00; 35°C; 30 min

Adsorption isotherms were plotted according to Freundlich and Langmuir equations to obtain the information about the adsorption capacity of PSAC and adsorbate/adsorbent affinity.

3. RESULTS AND DISCUSSION

3.1. PSAC characterization

It is extremely important to know the chemical and physical properties of the adsorbent materials, since they will indicate for which purposes they can be applied according to their structure and interactions. Figure 1 shows images of peach stone in pieces, powdered PSAC and a scanning electron microscope (SEM) image of the PSAC. Table 2 presents the results of the physicochemical parameters from the PSAC and also of a commercial activated carbon (PAC), which was used for comparison with the proposed alternative material. The SEM image suggests a porous surface of PSAC (Figure 1C), which is in agreement with measured parameters of iodine index (487.89 mg g⁻¹) and caramel discoloration (29.44%) of PSAC, indicating the possibility of the product application. Silva *et al.* (2019), described the total BET surface area of PSAC as 500.70 m² g⁻¹ and also that 64% of the total pore volume consisted of micropores.

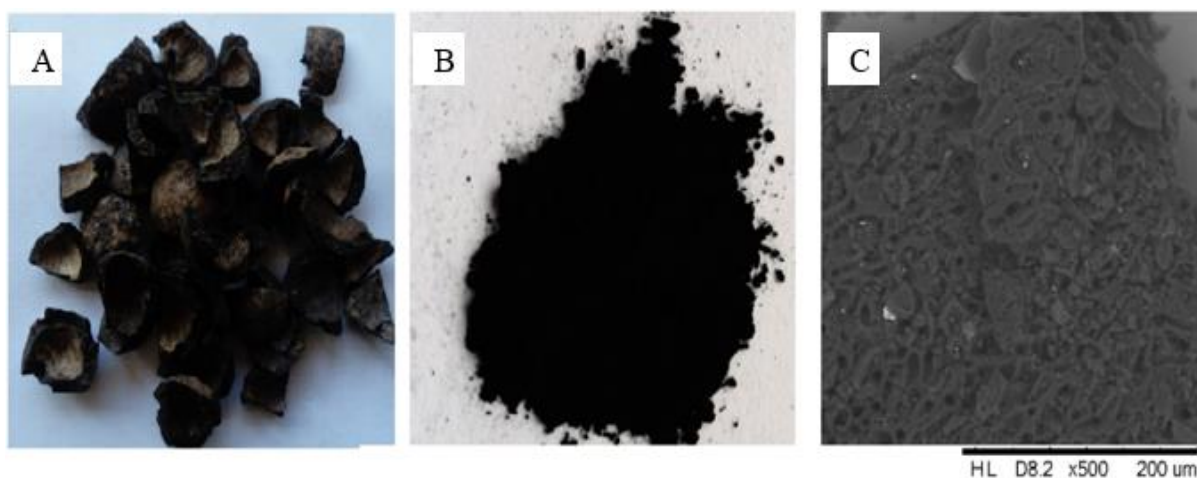


Figure 1. Images of peach stone in pieces (A), powdered activated carbon of the peach stone (B), and enlarged image of the PSAC (C).

Source: author.

Table 2. Physicochemical parameters of PSAC and commercial activated carbon (PAC).

Parameters	PSAC	PAC
% Porosity	0.31	0.74
% Volatiles contente	16.04	22.20
% Fixed carbono content	74.18	61.83
Iodine number (mg g ⁻¹)	487.79	767.16
% Caramel adsorption capacity	29.44	77.23

It was observed that the PAC used commercially presented higher values in four of the five evaluated parameters. Volatile material content, defined as the gases which are released during the burning of carbon, depends on the chemical composition of the raw material used, indicating that the two carbons have different chemical composition (Carmo, 1988). The PSAC's lower volatile percentage corroborates and is in agreement with the result of porosity (0.31%), which was about 50% lower than the commercial PAC, because the smaller the pore quantity, the smaller the volatile quantity. Furthermore, the composition of the vegetable residual product has a strong influence on the porosity of the obtained activated carbon, and that high lignin amount has a great potential to produce structure of macropores while cellulose produces predominantly microporous materials (Cabal *et al.*, 2009).

In the chemical activation the oxidant agent and the carbonization temperature are factors that can contribute to the carbon porosity, as they influence the area and porosity characteristics of the activated carbon produced (Tay *et al.*, 2009).

The adsorption characteristics of the AC are determined by the pore structure (magnitude and pore volume distribution) and functional groups that provide the acid-base characteristics surface of the activated carbon (Ahmedna *et al.*, 2009; Aworn *et al.*, 2008). Raw materials from fruit residues, such as seeds and hazelnut shells, when subjected to activation at high temperatures present vast surface area and high development of micropores (Tay *et al.*, 2009). The existence of micropores is favorable for adsorption in gaseous states, as well as meso and macropores are indicated for liquid state adsorption (Teng and Lin, 2002).

Iodine adsorption capacity and caramel discoloration can indicate the size of the pores in activated carbon. High values of the two parameters indicate that the material can adsorb molecules of small molecular weight (by iodine number) and heavy molecular weight (by caramel discoloration). Other studies have already evidenced such results, the number of iodine is related to the adsorption of molecules of small molecular weight (Di Bernardo and Dantas, 2005), being used as representative index of the amount of micropores present in activated carbon sample (Brandão and Silva, 2006). The PAC showed high values for both parameters, indicating that it is a carbon that presents micro- and macropores in its structure, whereas the PSAC suggests to present intermediate pores (mesopores). According to the study (Donati *et al.*, 1994), in which eight samples of PAC that were analyzed, it was verified that the carbons produced from wood present the largest volume of micropores and mesopores.

Trugilho and Silva (2001) has studied a carbon produced from eucalyptus, and reported a low quantity of volatile materials, high levels of fixed carbon and increased calorific value in the material. The PSAC showed a value of calorific power higher than the commercial material PAC, being able to present a great energetic and industrial potential of the carbon derived from this raw material (Trugilho and Silva, 2001). It is estimated from the result obtained from the fixed carbon amount for PSAC (74.18%), that it has high levels of lignin and holocellulose (Santos *et al.*, 2016).

3.2. Evaluation of PSAC adsorption efficiency to ATZ removal

Different masses of PSAC were placed in contact (2 h) with solutions containing different dosages of ATZ. Table 3 presents the results of this evaluation. It was observed that the higher the PSAC mass the greater the ATZ removal, and that from the mass of 0.00750 g, all ATZ was removed regardless of its concentration (200 a 600 $\mu\text{g L}^{-1}$).

The adsorptive capacity of the PSAC was calculated after further tests were carried out by setting the PSAC mass to 0.00250 g and varying the ATZ concentrations from 200 to 600 $\mu\text{g L}^{-1}$. In addition to the adsorptive capacity of the PSAC (Table 4), the adsorption isotherm of the ATZ in PSAC was also constructed (Figure 2). The curve presented a characteristic of the isotherm model described by class H, suggesting that the adsorbate has a great affinity for the adsorbent (Giles *et al.*, 1960). The isotherm configuration is a strong indicative to explain the adsorption phenomenon. Activated carbons that have large adsorptive capacity shows concave isotherms. It means that a large amount of ATZ can be adsorbed per unit of activated carbon mass (Moreno-Castilla, 2004).

Table 3. Study of the variation of PSAC mass as a function of ATZ concentration.

PSAC (g)	Concentration of ATZ ($\mu\text{g/L}$) removed by PSAC				
	1	2	3	4	5
0.00050	108.20	92.96	88.00	80.95	67.78
0.00075	114.75	107.25	106.67	104.76	100.42
0.00100	137.70	111.97	117.33	114.29	110.46
0.00250	180.33	135.21	133.33	130.95	115.48
0.00500	186.89	300.00	400.00	485.71	600.00
0.00750	190.16	300.00	400.00	500.00	600.00
0.01000	196.72	300.00	400.00	500.00	600.00

Initial ATZ concentration: (1) 200; (2): 300; (3): 400; (4): 500; (5): 600 $\mu\text{g/L}$.

Table 4. Study of PSCA adsorption capacity ($m= 0.00250$ g).

ATZ initial concentration ($\mu\text{g/L}$)	ATZ residual concentration ($\mu\text{g/L}$)	% Removal	q
200	6.06	97	0.0038
300	41.7	86	0.0051
400	102.9	74	0.0059
500	123.6	75	0.0075
600	208.0	65	0.0078

q = adsorptive capacity of carbon.

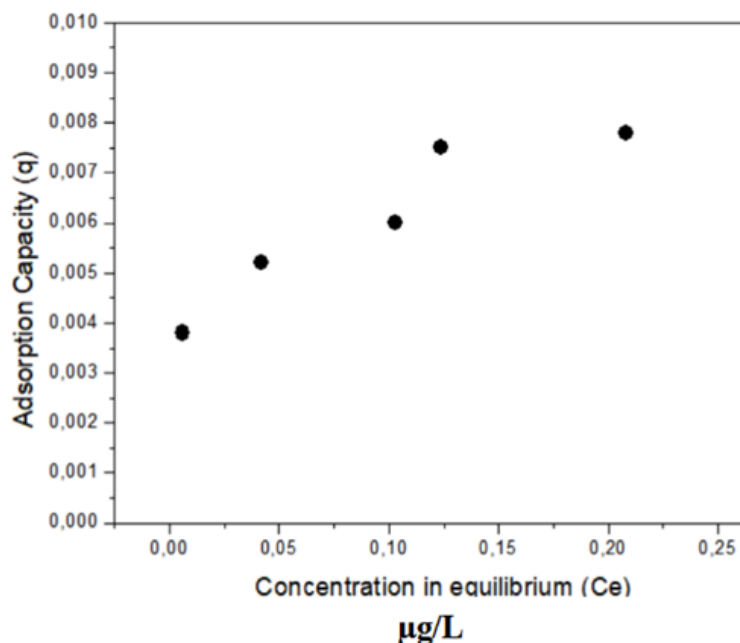


Figure 2. ATZ adsorption isotherm in PSAC.

Several parameters were varied simultaneously through experimental design to evaluate the best ATZ adsorption medium by PSAC. Figure 3 shows the Pareto graph indicating that none of the factors applied to planning 2^3 were significant, so univariate studies can be done. No factor as well as its correlations were important in ATZ sorption by PSAC.

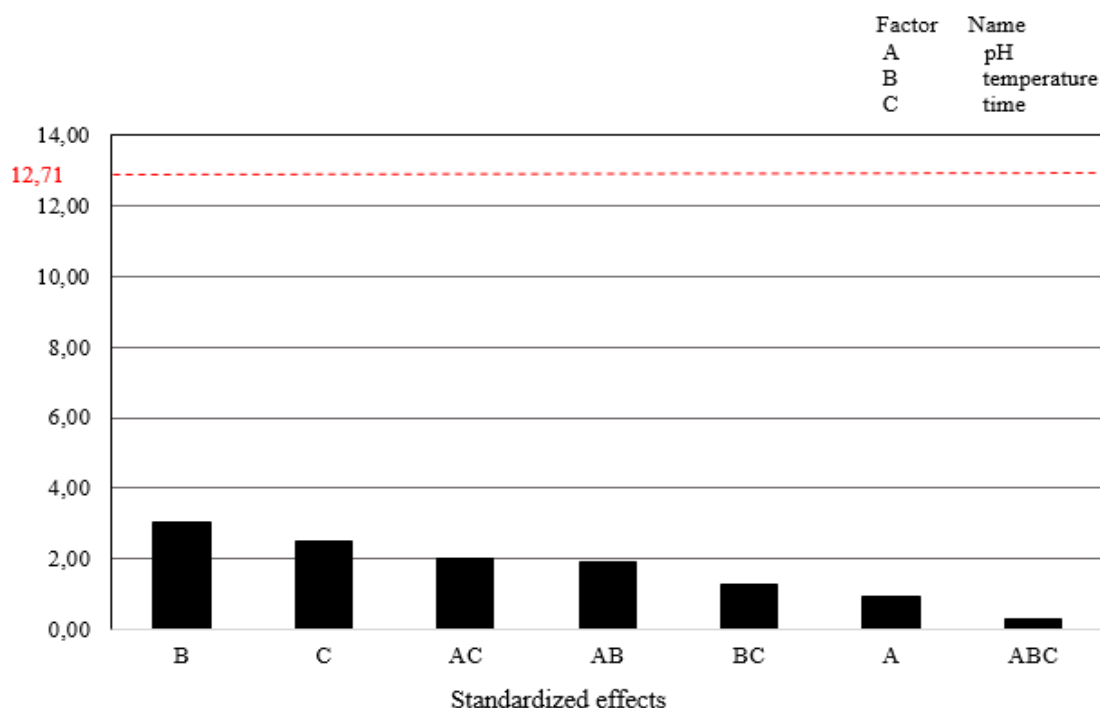


Figure 3. Pareto graph obtained from the results of factorial design 2^3 .

From the results obtained in the experimental design, an adjustment was made for conditions close to the environment, such as pH = 6.5, ambient temperature and the stirring time of 10 minutes, always seeking a lower cost of energy and time. Lima et al described that the PSAC zeta potential is 7.11, so in the adsorption pH suggested in this work (6.5), the surface of the carbon showed positive charges, positively influencing the adsorption of ATZ (Lima *et al.*, 2014).

3.3. PSAC application in natural water sample

In order to evaluate the adsorption study of ATZ in PSAC, it was applied in real samples of lake water doped with ATZ ($400 \mu\text{g L}^{-1}$) to verify the adsorption efficiency of the ATZ in the presence of competitors dissolved in the sample, such as metallic ions, humic and fulvic acids. The PSAC dosage used was 0.00250 g in order to evaluate the factors involved during the ATZ removal process, because in the presence of activated carbon heavy masses, all ATZ would be sorbed and it would be difficult to calculate the sorption capacity in view of the natural interference in the sample. Figure 4 shows the ATZ contents removed in ultrapure water and in natural water samples. When ATZ was present in ultrapure water, 35% of it was removed by the PSAC, and when present in the natural water sample was 25%, indicating that there is, undoubtedly, competition between other natural compounds present in the sample for PSAC active sites. However, it should be noted that the adsorbent mass used in this evaluation was very small and that larger masses of carbon could certainly remove all of the ATZ from the medium, as may already be observed from Table 2 data for PSAC masses equal to or greater than 0.0075 g.

Coelho *et al.* (2012), also described competition between ATZ and organic matter in adsorption studies using pulverized coconut shell activated carbon. They observed that the Adsorption Capacity Constant (Kf) value, in effluent water, suffered a reduction of 29%, demonstrating interference from other adsorbates in the TOC/ATZ relation. Zadaka *et al.* (2009) removed ATZ from water using polycations pre-adsorbed on montmorillonite. Batch experiments demonstrated that the most suitable composite poly (4-vinylpyridine-co-styrene)-montmorillonite removed 90–99% of atrazine within 20–40 min and the filter was only slightly influenced by organic material. Torrellas *et al.* (2015) evaluated activated charcoal from peach stones for adsorption of emerging contaminants (caffeine, diclofenac and carbamazepine) in ultrapure water. Adsorbent material was chemically activated by $\text{H}_3\text{PO}_4(\text{s})$. Carbamazepine adsorption capacity was higher than caffeine and diclofenac. However, this activated carbon was not applied to actual samples to assess competition between other components present in natural waters.

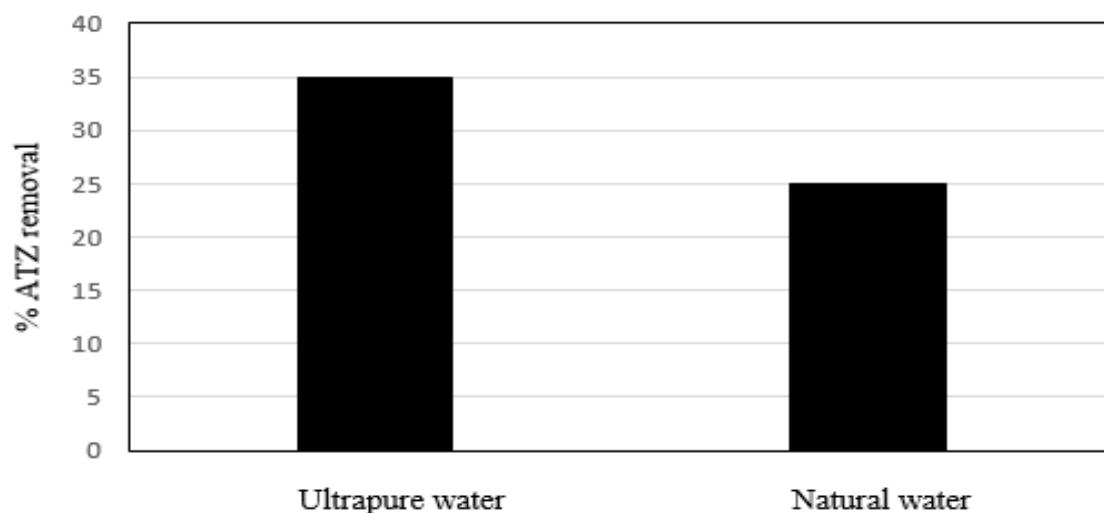


Figure 4. Removal of ATZ ($400 \mu\text{g L}^{-1}$) in solution using PSAC (0.0025 g).

4. CONCLUSION

Considering the effects of ATZ in the medium and its health impacts, its removal by the adsorption mechanism with the use of peach stone activated carbon becomes a viable alternative as ATZ removal technology.

The suggested experimental adsorption conditions, such as pH close to neutral, ambient temperature and short contact time (10 min) are favorable, since they do not demand a high energetic cost.

According to the physicochemical characteristics of peach stone activated carbon, porosity, surface area and iodine number, its indication as adsorbent material is suggested.

The application by batch system was suggested because it's versatility for application in seasonal problems, such as pesticide contamination, as well as the low cost.

5. ACKNOWLEDGEMENTS

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