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Article

Removal of Diuron and Hexazinone from Public Water Supply Using a Filter System

Remoção de Diuron e Hexazinone em Água de Abastecimento Público Utilizando Um Sistema de Filtros

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ABSTRACT **-** The intensive use of pesticides, such as the herbicides diuron and hexazinone, leads to a concern about the preservation of the water quality of the water resources destined for human consumption. Currently, conventional water treatment techniques do not have high capacity to remove herbicides; therefore, the need to study other techniques arises. The present study aimed to evaluate the efficiency of the removal of diuron and hexazinone herbicides present in water through a commercial filtration system. The study was carried out in a filtration system composed of the following sequence of filters: sand filter and coconut shell activated granular carbon, polypropylene filter 20 mm, activated carbon filter with silver salts, polypropylene filter 5 mm and reverse osmosis. The affluent contained an initial concentration of 12.06 mg mL^{-1} of hexazinone and 37.70 mg mL^{-1} of diuron. The samples were collected in all filters and subjected to the analysis of turbidity and presence of herbicides by high performance liquid chromatography. The filter set removed 99.31; 99.84 and 99.99% of water turbidity, hexazinone and diuron respectively at the end of the system. After the filtration process, the treated water presented adequate quality for public supply and human consumption. The technique employed in this study is easy to operate and has a low acquisition and maintenance cost.

Keywords: activated granular coal, reverse osmosis, herbicide removal, water treatment.

RESUMO - O uso intensivo de pesticidas, como os herbicidas diuron e hexazinone, levam a uma preocupação quanto à preservação da qualidade da água dos recursos hídricos destinados ao consumo humano. Atualmente, as técnicas convencionais de tratamento de água não possuem alta capacidade de remover os herbicidas; portanto, surge a necessidade de estudar outras técnicas. O presente estudo teve por objetivo avaliar a eficiência de remoção dos herbicidas diuron e hexazinone presentes na água, através de um sistema de filtração comercial. O estudo foi realizado em um sistema de filtração composto pela seguinte sequência de filtros: filtro de areia e carvão granular ativado de casca de coco, filtro polipropileno de 20 mm, filtro de carvão ativado com sais de prata, filtro de polipropileno de 5 mm e osmose reversa. O afluente continha uma concentração inicial de 12,06 mg mL^{-1} de hexazinone e 37,70 mg mL^{-1} de diuron. As amostras foram coletadas em todos os filtros e submetidas à análise de turbidez e presença dos herbicidas por cromatografia líquida de alta eficiência. O conjunto de filtros removeu 99,31, 99,84 e 99,99% da turbidez da água, hexazinone e diuron, respectivamente, ao final do sistema. Após o processo de filtração, a água tratada apresentou qualidade adequada para o abastecimento público e consumo humano. A técnica empregada neste estudo é de fácil operação e possui baixo custo de aquisição e manutenção.

Palavras-chave: carvão granular ativado, osmose reversa, remoção de herbicidas, tratamento de água.

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INTRODUCTION

The use of combinations of different herbicides has been a common practice in agriculture, due to the increase in the action spectrum to control weeds and the reduction of recommended doses (Oliveira Jr., 2011). Thus, the commercial mix of herbicides diuron + hexazinone is frequently recommended to control weeds in sugarcane crops, applied in pre-emergence directly on the soil (Ross and Fillols, 2017).

Hexazinone is highly soluble in water (33,000 mg L^{-1} at 25 °C), with moderately long residual activity (half-life in the field of 115 days under aerobic conditions). It is highly mobile in the soil (Celis et al., 2002; Mendes et al., 2013), and it may contaminate ground waters. Diuron also shows a moderately long residual activity (half-life between 102 and 134 days), however, it has low solubility in water (42 mg L^{-1} at 25 °C) and low mobility in the soil, remaining only on the surface layer, up to 10 cm (Mendes et al., 2016). However, diuron may suffer surface runoff and contaminate surface waters (Prichard et al., 2005).

A major concern are the risks and effects of environmental contamination by herbicides, which may reach the water resources (Rao and Hornsby, 1999; Kogan and Pérez, 2003) and cause deleterious effects to human and animal health, due to the accumulation of these toxic substances in the adipose tissue (Marchis et al., 2012). Hexazinone and diuron are often found as microcontaminants of the soil and water resources near agricultural locations, where they are constantly used (Hussain et al., 2015, Villaverde et al., 2017). In addition, the concentrations of both herbicides found in water resources vary from 15 ng L^{-1} up to 408.0 μg L^{-1} (Davis et al., 2008; Paschoalato et al., 2009; Ferreira et al., 2016; Metcalfe et al., 2016). Diuron and hexazinone were detected in rivers and wells of cities near Piracicaba (sugarcane-producing region), whose waters are partially destined to human consumption (Paschoalato et al., 2009; Santos et al., 2015).

The conventional techniques used on water treatment systems are not as efficient to remove organic microcontaminants, such as herbicides, therefore, it is necessary to add adequate pre- or post-treatments to remove these unwanted compounds (Voltan et al., 2016). Due to that fact, environmentally and economically feasible technologies to remove such microcontaminants have been currently searched for.

Methods such as filtration in sand filters and adsorption in activated carbon were shown to be efficient in the removal of organic microcontaminants (Frank et al., 2015). It is noteworthy that filtration is a physical-chemical process, whose efficiency depends on the composition material of the filter. Among the different materials available, the polypropylene filters have been widely used in water treatment to retain particulates in suspension. The structure of polypropylene makes it highly versatile, showing a wide variation in pore sizes, significant durability, inert material, easy installation and great filtration capacity (Liu et al., 2006). Combined to the treatment process mentioned, there is also the activated carbon adsorption technique, which is an efficient non-specific adsorbent substance, with an extremally porous superficial area (Ilomuanya et al., 2017), allowing the removal of organic compounds, such as herbicides, in addition to inorganic compounds, color and flavor of the water (Hamdaoui and Naffrechoux, 2007).

To obtain high-quality water, membrane technologies are used, including reverse osmosis (ANA, 2007). This technique is used for the desalination and demineralization of waters (Bitaw et al., 2016), and its main principle is to apply a force greater than the osmotic pressure to the compartment with the concentrated solution, causing the inversion of the flow, forcing the passage of the solvent and retaining the solute (Gouvêa et al., 2012). Reverse osmosis has been widely used as an important option to recover residual waters, since it may be highly efficient to remove microorganisms, colloidal matter, dissolved solids, organic and inorganic materials in the water (Garud et al., 2011).

Within this context, it is considered that a filtration set used as post-treatment stage for contaminated water may be efficient to remove herbicides. Thus, the objective of this research was to evaluate the efficiency to remove diuron and hexazinone in the public supply water, through a filtration system.

MATERIAL AND METHODS

Treatment of fortified water with herbicides

To conduct the study, a mix of hexazinone and diuron was prepared with water from the conventional public treatment system of the “Luiz de Queiroz” School of Agriculture, from the University of São Paulo, which collects water from the Piracicaba river. A previous chromatographic analysis of the water was used and the contamination by both herbicides was not found. The contamination concentration of the products was fixed at 12.06 µg mL^{-1} and 37.70 µg mL^{-1} for hexazinone and diuron, respectively, based on the doses used for both herbicides on a study conducted by Dantas et al. (2011). The affluent showed pH 7.0, therefore, it was not necessary to correct the pH.

The mix was filtered on a pilot installation (Figure 1), where the water fortified with herbicides was stored in a 500 L tank (Gratt®); then, it was subjected to filtration in a sand and carbon column filter, constituted by three layers of quartz and one layer of coconut shell activated vegetable carbon, with the capacity to operate at a 500 L h^{-1} flow (Teixeira Tozzi®).

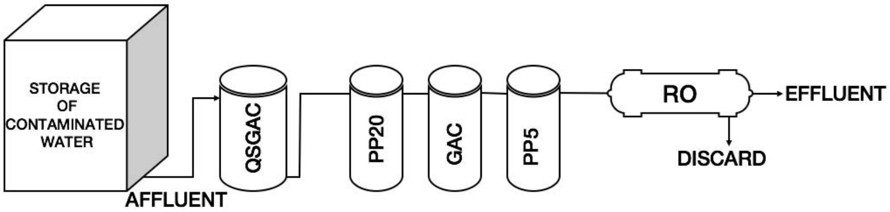


Figure 1 **-** Illustrative layout of the test bench for the treatment of contaminated water (QSGAC - Quartz Sand and Granular Activated Carbon filter, PP20 - Polypropylene filter 20 µm, GAC - Granular Activated Carbon filter, PP5 - Polypropylene filter 5 µm, and RO - Reverse Osmosis).

Then, the pre-purified water was treated in a filtration set (considering the treatments), constituted by 9 3/4" column filters, in the following sequence: polypropylene 20 µm (EcoFi®), granular activated carbon filter with colloidal silver, covered with two layers of internal and external covering to retain particles suspended in water (Carbon Block - Acquabios®) and polypropylene filter 5 µm (EcoFi®). After this stage, the water was subjected to reverse osmosis using a Dry Reverse Osmosis membrane (Tw-1812-100D Hydron®). Two By-pass pumps were used, with pressure of 861.8 kPa (Pump-24D-CE - Diaphragm Booster®), one after the storage tank and the other between the 5 µm polypropylene filter and the reverse osmosis filter, with a 9.33 L h^{-1} flow, measured with a beaker.

At each water treatment stage, samples of the treated water were collected for further chromatographic analysis. The water samples were collected by simple sampling in triplicates, using 1 L amber flasks, and refrigerated for two days, until the analysis. The monitored parameters were: turbidity (turbidimeter TB-1000 - Tecnal®) and the concentrations of both herbicides by high-performance liquid chromatography.

Chemical products

The analytical standards for hexazinone (99.9% of purity) and diuron (99.6% of purity) acquired from Sigma Aldrich® (St. Louis, MO, USA) were used to prepare a stock solution of 1,000 µg mL^{-1} in acetonitrile. The stock solution was used to elaborate the calibration curve for the chromatographic analyses.

Chromatographic analysis of the herbicides

The composition and concentration of the herbicides on the water samples from the public supply were determined using a high-performance liquid chromatographer (HPLC), Agilent Tecnologies® model 1200, equipped with a UV-Vis detector and C18 column (3.5 x 4.6 x 150 mm, Kromasi®).

The chromatographic conditions were: water (with orthophosphoric acid added to correct the pH to 2.2) and acetonitrile as the mobile phase (40:60, v v^{-1}) under a flow of 1.0 mL min^{-1}; injection volume of 10 µL; column temperature of 35 °C; and readings on wave lengths 242 and 235 nm. The analyses were made in triplicates, and the Chemstation® software was used to analyze the data.

Both herbicides were identified comparing the retention times, and they were quantified by comparing the areas on the chromatograms of the water analyses obtained with the standard through an external calibration method. The chromatographic method used in the study was modified and adapted from Mendes et al. (2016), followed by a validation. The results were expressed as a percentage of the herbicides found in each segment of the filtering system, in relation to the initially applied dose.

Statistical analysis of the data

The data was analyzed from the descriptive statistics, represented by the average (n = 3) and the standard deviation. The figures were plotted with the assistance of software Sigma Plot (version 10.0 for Windows, Systat Software Inc., Point Richmond, CA, EUA).

RESULTS AND DISCUSSION

Validation of the chromatographic method

The chromatographic analyses of the herbicides to validate the method are shown on Table 1. The linearities were obtained for each herbicide at the following concentrations: 0.5, 1.0, 2.0, 3.0, 4.0 and 5.0 µg mL^{-1}, showing high determination coefficient values (R^2 = 0.999) for hexazinone and diuron. These R^2 values were higher than 0.99, which, according to the literature, is the minimal acceptable linearity value for analytical methods (ANVISA, 2003), making the suggested method reliable.

The recovery of the method varied from 97.69-99.40% and 96.87-98.80% for hexazinone and diuron, respectively, both at a concentration of 5.0 µg mL^{-1} and with three injections of each replicate. The repeatability (coefficient of variation - CV) was ≤0.12% for both herbicides (Table 1). According to Anvisa (2003), an acceptable recovery varies between 70% and 120%, with CV below 20%.

For hexazinone, the limit of detection (LOD) and the limit of quantification (LOQ) were 0.005 and 0.016 µg mL^{-1}, respectively (Table 1). These values are similar to those found by Ferracini et al. (2005), who, when using HPLC to analyze hexazinone, obtained LOD of 0.005 µg mL^{-1} and LOQ of 0.018 µg mL^{-1}, due to the signal/noise of the equipment.

The LOD for diuron was 0.004 µg mL^{-1}, while LOQ was 0.013 µg mL^{-1} (Table 1). Similar values were also obtained by Ramli et al. (2012) and Felicio et al. (2016), who also used HPLC and

Table 1 **-** Values of retention time, residual standard deviation, limit of detection, limit of quantification, equation of linearity curve, coefficient of determination, recovery percentage, standard deviation, and coefficient of variation of the proposed method for hexazinone and diuron

Herbicide	RT (min)	RSD	LOD (µg mL^{-1})	LOQ (µg mL^{-1})	Equation of linearity curve	R^2
Hexazinone	1.632	0.226	0.005	0.016	Y= 30.70022x + 0.0222839	0.999
Diuron	2.327	0.167	0.004	0.013	Y= 29.30563x + 0.0169527	0.999

Herbicide	Sample (3 injections of each replicate)								
	1st			2nd			3rd		
	R(%)	±SD	CV(%)	R(%)	±SD	CV(%)	R(%)	±SD	CV(%)
Hexazinone	97.69	0.01	0.10	99.07	0.01	0.12	99.40	0.00	0.00
Diuron	96.87	0.01	0.12	97.87	0.01	0.12	98.80	0.00	0.00

RT: Retention Time; RSD: Residual Standard Deviation; LOD: limit of Detection; LOQ: Limit of Quantification; R(%): recovery percentage, SD: standard deviation, CV: coefficient of variation.

obtained LOD of 0.02 µg mL^{-1} and 0.018 µg mL^{-1}, respectively, and LOQ of 0.05 µg mL^{-1} and 0.058 µg mL^{-1}, respectively. However, both authors did not evaluate the detection of the diuron and hexazinone mix.

The chromatographic system used in the study was able to adequately separate analytes and provide a clear detection within only 6 min.; the detection of both herbicides occurred in less than 3 min (Figure 2B), and as described previously, the water used showed no residues of both herbicides (Figure 2A). Some authors, such as Ferracini et al. (2005), Ramli et al. (2012) and Felicio et al. (2016), found a total running time of 15 min., with hexazinone retention time of 10 min., and 10 and 8.5 min., respectively for diuron. These results indicate that the method was sensitive to the detection of the herbicides at low concentration levels and with a fast analysis time.

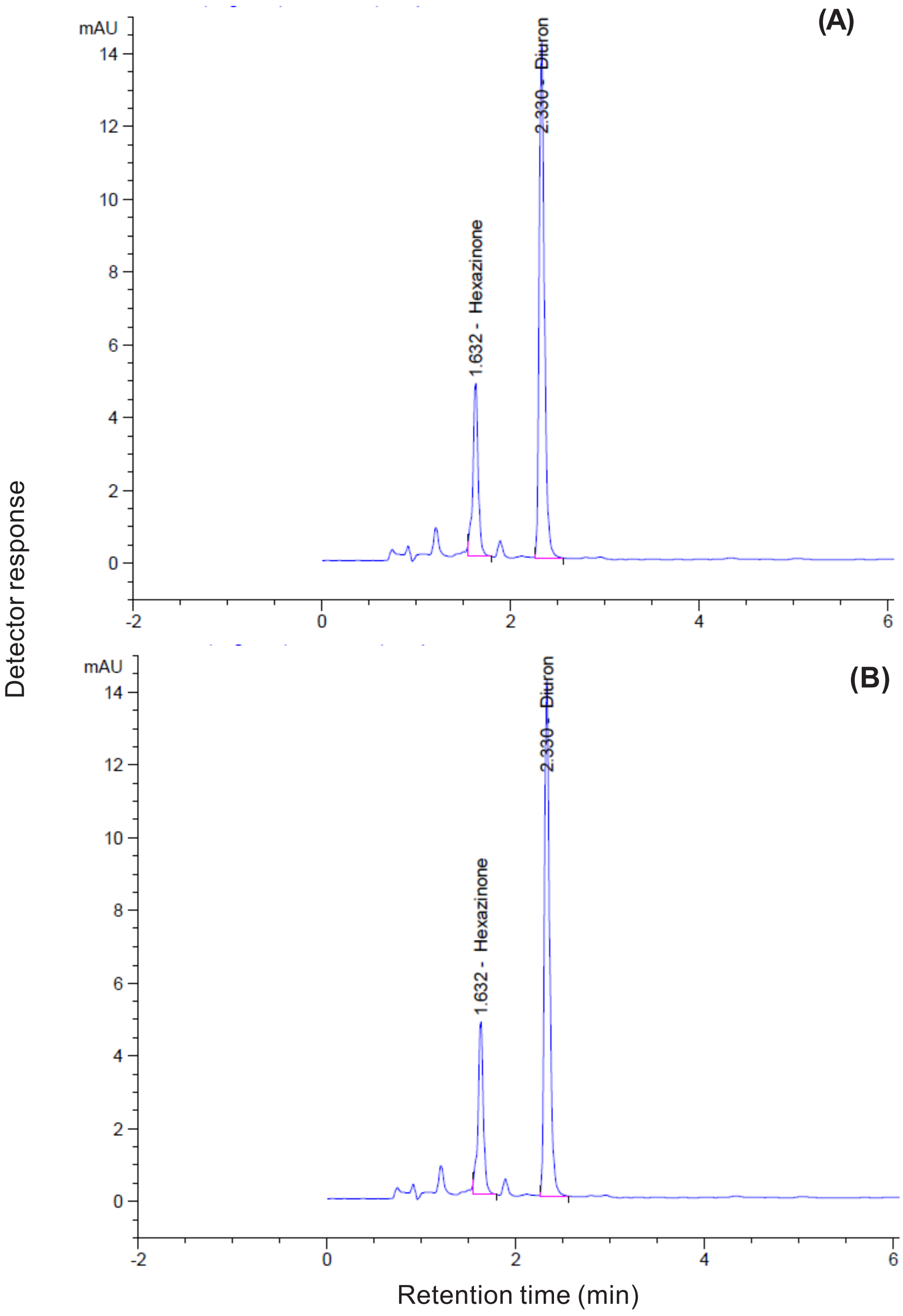


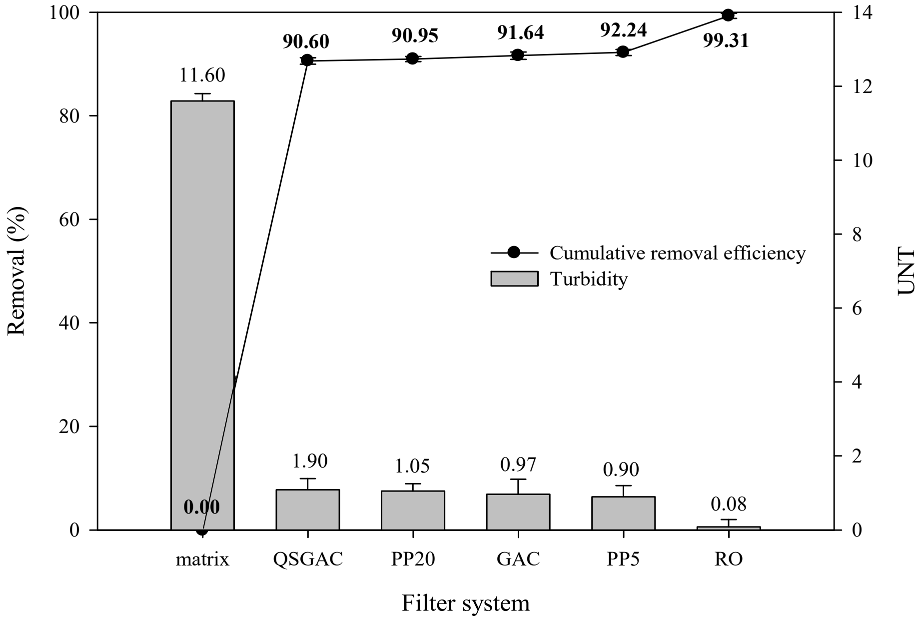
Figure 2 - Chromatogram of hexazinone and diuron obtained by HPLC in matrix without both herbicides (A) and with 12.06 and 37.70 µg mL^{-1} for hexazinone and diuron, respectively, in matrix contaminated water (B) used in the study.

Removal of the water turbidity

The system was highly efficient to remove the turbidity of the contaminated water. The initial turbidity of the water was 11.6 UNT, and the first quartz sand and granulated activated carbon filter (QSGAC) was responsible for reducing 90.60% of such turbidity (Figure 3). The filtration set was not as efficiency to remove turbidity, since it was able to remove less than 2% of the turbidity of the affluent. However, the last reverse osmosis filter (RO) showing a cumulative removal efficiency of 99.31% of turbidity, resulting in an effluent with turbidity of only 0.08 UNT (Figure 3).

It is important to point out that, when it occurs naturally, the turbidity of the water may be related to rock, clay, and silt particles or to microorganisms; however, when it occurs due to anthropogenic reasons, it may be related to the contamination by toxic compounds and pathogenic organisms (Binotto, 2012; Calegari et al., 2015). Therefore, in this study, the turbidity of the filtered water occurs due to natural and anthropogenic reasons.

To meet the potability standards, according to Decree number 2,914/11 from the Ministry of Health (Brasil, 2011), the water should not exceed 0.5 UNT. In this study, such requirement was met after the RO filter. Therefore, the system used was highly efficient to remove turbidity and meet the national potability standards.



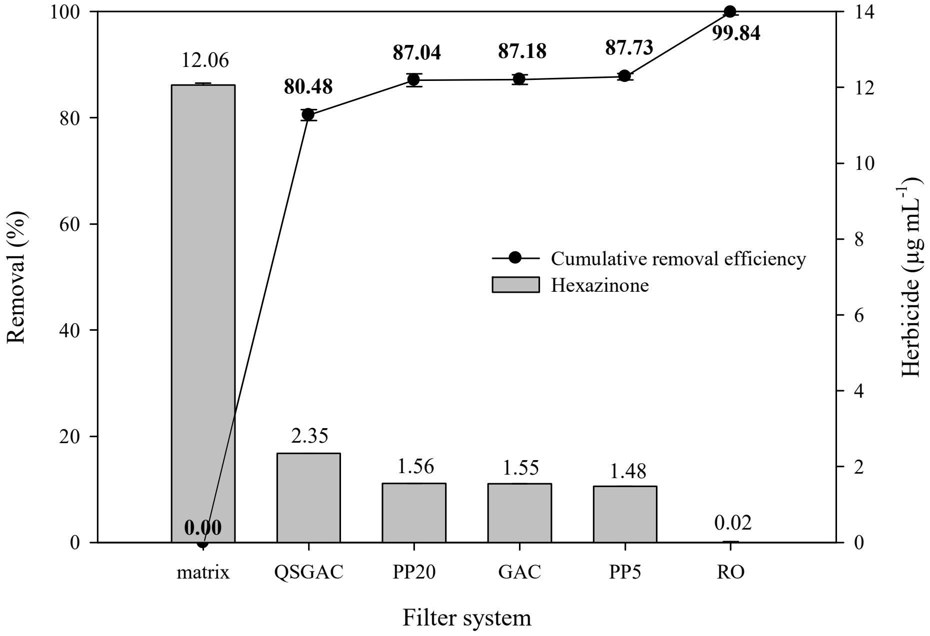
The vertical bars associated with each column or point represent the standard deviation (±SD) of each mean value (n = 3). (QSGAC - Quartz Sand and Granular Activated Carbon filter, PP20 - Polypropylene filter 20 µm, GAC - Granular Activated Carbon filter, PP5 - Polypropylene filter 5 µm, and RO - Reverse Osmosis).

Figure 3 **-** Removal of turbidity from public water supply in a filter system.

Removal of hexazinone from the water

The filtration system was able to remove 99.84% of the initial hexazinone concentration (Figure 4), which was 12.06 µg mL^{-1}. The QSGAC filter removed most of the hexazinone in the contaminated water (80.48%); in addition, after the reverse osmosis, 99.84% of the initial concentration was reduced, showing a concentration of only 0.02 µg mL^{-1} of hexazinone residues on the final effluent. This concentration meets the potability standard of the USA and Australia, whose maximum limit is 0.4 and 0.3 µg mL^{-1}, respectively (Hamilton et al., 2003).

In Brazil, Decree number 2,1914/2011 from the Ministry of Health (Brasil, 2011), which establishes the potability standards for the water for human consumption, indicates the maximal values of some herbicides, such as heptachlor, glyphosate, atrazine and diuron, however, it does not establish the maximal limits for hexazinone.



The vertical bars associated with each column or point represent the standard deviation (±SD) of each mean value (n = 3). (QSGAC - Quartz Sand and Granular Activated Carbon filter, PP20 - Polypropylene filter 20 µm, GAC - Granular Activated Carbon filter, PP5 - Polypropylene filter 5 µm, and RO - Reverse Osmosis).

Figure 4 - Removal of hexazinone from public water supply in a filter system.

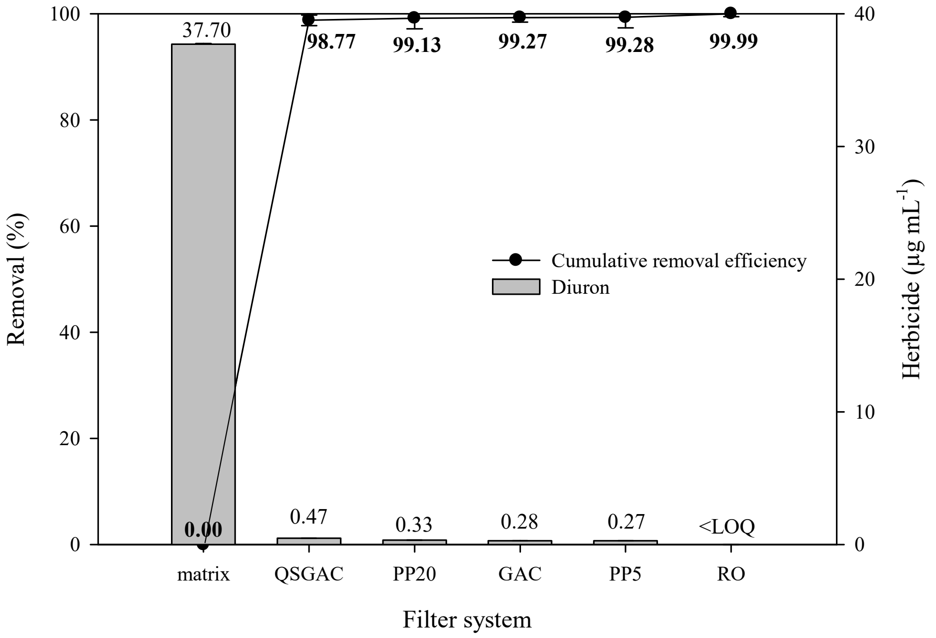
On the other hand, according to Kawamoto and Urano (1989), the higher the solubility of the herbicides, the lower the affinity with the organic matter. This characteristic of some herbicides may present a disadvantage in their removal efficiency in a filtration system that uses activated carbon. However, although hexazinone is highly soluble, the filters were able to remove them satisfactorily. This study also points out to the importance of removing hexazinone from the water efficiently, considering that this herbicide is highly soluble and may easily reach the water table.

Considering that most of the Brazilian households in rural areas (65.5%) collect water from wells, fountains or untreated water courses (IBGE, 2015), the filtration system suggested in this study may be used in rural households in regions where hexazinone is intensively applied, with the aim of reducing the risks to human health related to the possible ingestion of residues of this herbicide in the water.

Removal of diuron from the water

The QSGAC filter removed most of the diuron in the contaminated water (98.77%), resulting in an effluent with a concentration of only 0.47 µg mL^{-1} of the herbicide (Figure 5). Concomitantly, this filter was able to more efficiently remove the diuron in relation to hexazinone. It is assumed that the diuron molecule may bind more strongly to the organic carbon (CO) in the filter, considering that this herbicide shows a non-ionic (neutral) characteristic (PPDB, 2017). Rocha et al. (2013), when evaluation the diuron sorption in the soils, observed that, the larger the CO contents, the greater the sorption of the herbicide in the soil.

The same was described by Martinez et al. (2011), who, when evaluating the *babaçu* carbon for the sorption of diuron and hexazinone, observed a greater affinity of the carbon with diuron, in comparison to hexazinone. The authors also state that the pH of the water has a major influence in the adsorption of diuron by the activated carbon, since the neutral forms are more strongly adsorbed, when compared to hexazinone, which has a basic nature. Since the pH of the water treated in this study was neutral (7.0), this may explain the greater adsorption of diuron by QSGAC in relation to hexazinone.



The vertical bars associated with each column or point represent the standard deviation (±SD) of each mean value (n = 3). (QSGAC - Quartz Sand and Granular Activated Carbon filter, PP20 - Polypropylene filter 20 µm, GAC - Granular Activated Carbon filter, PP5 - Polypropylene filter 5 µm, and RO - Reverse Osmosis). <LOQ (below the limit of quantification).

***Figure 5* -** Removal of diuron from public water supply in a filter system.

After the QSGAC filter, it was observed that the filter set with polypropylene 20 µm + granular activated carbon + polypropylene 5 µm (PP20 + GAC + PP5) was responsible for a low removal of diuron, as observed in the removal of the turbidity and hexazinone, making them unnecessary for this filtering system. Then, after the RO filter, an effluent with only traces (<LOD) of the diuron concentration was obtained, which, as observed in the removal of hexazinone, also meets the potability standards of the USA, Australia and Canada, which established a maximal limit for the referred herbicide of 0.01, 0.03 and 0.15 µg mL^{-1}, respectively (Hamilton et al., 2003). In Brazil, Decree number 2,914/2011 (Brasil, 2011) establishes the maximal allowed value of 0.09 µg mL^{-1} of diuron for potable water. Since the referred decree is less restrictive than the potability standards of the USA and Australia, the RO filter effluent also meets the water potability standards in Brazil.

The QSGAC filter was efficient to remove both studies herbicides and also the water turbidity, coupled to the RO filter, allowing a high-quality water purification, compatible with the requirements of developed countries. The QSGAC and RO filters are widely used in purification and desalinization processes of waters for human consumption (van der Bruggen and Vandecasteele, 2002), due to their low cost and ease of use (Khawaji et al., 2008). Thus, the QSGAC and RO filter combination may be a feasible alternative to purify the water for human consumption, due to its low acquisition and maintenance cost, in addition to not demanding major technical expertise to be used and due to its high efficiency to remove herbicide residues.

Concomitantly, this filter system may be used in remote communities of potable water supply public systems, where the application of herbicides is intensive, such as in regions near sugarcane plantation areas.

It is concluded that the QSGAC filter was responsible for the greater removal of hexazinone and diuron, in addition to the water turbidity, while the filter set PP20, GAC and PP5 was not efficient to remove both herbicides in its affluent. However, after the last filter (RO), a removal of 99.99; 99.84 and 99.31% of diuron, hexazinone and contaminated water turbidity was observed, respectively. The water treated by the filtration system showed an adequate quality for public supply and human consumption. It is also pointed out that the technique used in the study is easy to operate and has a low acquisition and maintenance cost.

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