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Evaluation of two bioadsorbents for removing paracetamol from aqueous media

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Abstract The frequent contamination of water resources with drugs comprises one the most important environmental problems. In order to avoid serious disturbances for aquatic life, efficient and economically viable procedures should be developed for removing common pollutants, as paracetamol. From these considerations, the present work evaluated the efficiency of sugar cane bagasse (SCB) and vegetable sponge (VS), two natural adsorbents commonly found in Brazil, for retaining paracetamol molecules dispersed in aqueous solutions. Thus, systems composed of glass columns and peristaltic pumps were optimized and, for pH, the best value was 7.0. After optimisation, adsorption isotherms were built and it was possible to calculate the MAC_F values for SCB (120.5 µg/g) and VS (37.5 µg/g). Additionally, real matrices of pretreated water, from a municipal plant for water catchment, were enriched with paracetamol at 5 µM and passed through glass columns packed with SCB, VS and activated carbon (AC). The results showed that SCB was more attractive than AC in terms of price and efficiency (60% against 45% adsorption, respectively), while VS was responsible for removing 40% of paracetamol dissolved in the enriched water samples. Thus, the proposed natural adsorbents can be classified as viable materials to remove paracetamol from water used for human consumption.

Keywords: adsorption, bioadsorbent, paracetamol, water

INTRODUCTION

The lack of appropriate treatment systems is responsible for introducing medicines into diverse aquatic ecosystems, thus creating serious environmental problems throughout the world. Pharmaceutical drug consumption has increased exponentially; three thousand different active ingredients are released for human and veterinarian use every year (Nebot et al. 2007). This fact has occasioned the constant involvement of aqueous residues contaminated with the referred pollutants in several aquatic ecosystems (Radjenovic et al. 2007; Carrara et al. 2008; Song et al. 2008; Verlicchi et al. 2010), including public water stations (Williams et al. 2006).

The frequent contamination of water resources with antibiotics (Sanderson et al. 2004; Managaki et al. 2007), hormones (Carballa et al. 2008), analgesics (Reif et al. 2008), psychotropics (Carballa et al. 2008), antipyretics (Suntisukaseam et al. 2007) and anti-inflammatories (Hilton and Thomas, 2003) has attracted enormous attention because of the environmental impacts (Rabiet et al. 2006) and potential adverse effects on human health (Thomas et al. 2007; Sammartino et al. 2008), since chronic

exposure to pharmaceutical drugs, even below toxic levels, may lead to unexpected side effects (Hari et al. 2005).

Most pharmaceutical compounds are introduced into water bodies through domestic sewage (Nebot et al. 2007; Reif et al. 2008) or industrial wastewater (Stackelberg et al. 2004), hospitals (Focazio et al. 2008; Verlicchi et al. 2010) and health centres (Feldmann et al. 2008). Usually, these drugs are found in the environment in concentrations in the μg L^{-1} or ng L^{-1} range (Hilton and Thomas, 2003). The presence of these compounds in water for domestic consumption is constant because of the lack of efficient procedures to remove them (Roberts and Bersuder, 2006; Radjenovic et al. 2007).

Among the drugs frequently used by the Brazilian population, analgesics and anti-inflammatories are the most common, and are widely consumed with or without medical prescription (Bertoldi et al. 2004). Among those, paracetamol stands out for being highly toxic to the liver (Rubenstein and Laine, 2004; Kim et al. 2007), with a potential risk of hepatitis development (Larrey, 2009).

The increasing preoccupation with the impact of pharmaceutical drugs on the environment has lead Brazilian government regulators to recommend ways to handle and dispose wastes from the health service. However, domestic sewage usually does not get an adequate treatment for pharmaceutical drug elimination (Reif et al. 2008), so it is mandatory to search for efficient and economically feasible procedures to remove this kind of pollutant (Bila and Dezotti, 2003; Bila and Dezotti, 2007).

Among the most promising decontamination materials for aquatic environments, the use of bioadsorbents such as sawdust (Wan Ngah and Hanafiah, 2008), sugar cane bagasse (Raymundo et al. 2010), corncob (Garg et al. 2007), aquatic plants (Baral et al. 2009), vegetable sponge (Demir et al. 2008), loofa sponge (Nabizadeh et al. 2008), coconut mesocarp (Sousa et al. 2007), banana peel (Wan Ngah and Hanafiah, 2008), *Caladium bicolor* (Horsfall and Spiff, 2005), maize husk (Igwe and Abia, 2007), and seaweed (Antunes et al. 2003) stands out.

This article presents the results obtained using sugar cane bagasse and vegetable sponge (*Luffa cylindrica)* in filters to retain paracetamol from aqueous solutions. Both bioadsorbents are widely available throughout the Brazilian territory with an estimated Sugar Cane Bagasse (SCB) production around five to twelve million tons per year (Austin, 2009).

MATERIALS AND METHODS

Reactants and instruments

SCB was obtained from Acronym for a distillery localized in the Vitória city, Espírito Santo State, Brazil (DISA), an alcohol distillery located at the north of the Espírito Santo State, Brazil. Vegetable Sponge (*Luffa cylindrica*) (VS) samples were donated by small producers from the central part of the Espírito Santo State. Samples of potable water were collected at a water treatment station from the Espírito Santo Sanitation Company (CESAN). Paracetamol was obtained from Farma Derm (Vitória-ES, Brazil); hydrochloric acid was purchased from Vetec (Duque de Caxias-RJ, Brazil) and sodium hydroxide from Dinâmica (Diadema-SP, Brazil). The following equipment was used: an analytical scale (Shimadzu Model AY 220), UV/Vis spectrophotometer (Biospectro Model SP-220), pH meter (PHTEK), magnetic stirrer (Nova Ética and Biomixer), laboratory oven (Quimis Model Q-317 B), industrial blender (FAET), ultrasonic device (Ultracleaner 1400), scanning electron microscope (SHIMADZU, model SSX 550), sputter coater (SHIMADZU, model IC-50 Ion Coarter), automated physisorption instrument (Autosorb-1, Quantachrome Instruments), peristaltic pump (Instrutherm, BP 1000) and specific particle size sieves (Granutest).

Bioadsorbent preparation

To remove the maximum amount of contaminants from the bioadsorbents, these were washed with water (pH 7.0) and then dried in a laboratory oven (60ºC) for 15 hrs. In the next step, the material went to an industrial blender with posterior sieving to obtain particles sizes between 1.19 mm and 4.76 mm for SCB and VS, respectively. Polyethylene containers were used to stock the bioadsorbents.

Procedures

Scanning electron microscopy (SEM). SCB and VS samples were covered with a thin layer of gold, using the sputter coater, and they were analysed with the scanning electron microscope. An electron beam of 10 kV was used, which allowed for obtaining micrographs of the physical structure of the natural adsorbent surfaces.

Surface area and porosity determination (SAAP). SCB and VS samples were analysed through N_2 adsorption/desorption isotherms at 77 K, at different times, using an automated physisorption instrument. This equipment was used with a programme to calculate the material's surface area and its average pore size, according to Brunauer-Emmett-Teller, BET (Brunauer et al. 1938).

Adsorption isotherm building

In this experimental set, the onset of saturation conditions at the bioadsorbent front was estimated for paracetamol. For this purpose, the following paracetamol concentrations (1, 5, 40, 60, 80 and 100 µM) were tested at pH 7.0. The pH of maximum adsorption (pH 7.0) was obtained in our laboratory. This value was used because of its use in waste treatment stations. For this purpose, 3.0 g of SCB or VS were utilised at flow rate of 30 mL/min. The paracetamol concentrations were indirectly quantified by absorbance measurements (λ = 250 nm) of the eluates.

Paracetamol removal from enriched water samples

During this stage, the efficiencies of the columns filled with SCB or VS were evaluated by passing enriched (5 µM paracetamol) pretreated water samples obtained from CESAN. The pH of these samples was around 7 and they had the following values for colour (4 mg PT-Co/L; Hazem Unity), total dissolved solids (160 mg/L) and turbidity (2.17 NTU; Nephelometric Turbidity Unity) in accordance with the requirements of the Brazilian government (CONAMA, 2004). Glass columns (75 x 30 cm) were filled with 3.0 g of each adsorbent, and then aliquots (100 mL) of the enriched water samples were percolated at 30 mL/min. The quantification of adsorbed paracetamol was carried out by the previously described procedure. It must be noted that, at this stage, the columns were also filled with sand and gravel besides SCB and VS, exactly as is done at the CESAN water treatment station (Figure 1). An identical column containing activated carbon (an adsorbent with recognised efficiency) was also assembled in order to compare the performance of the two natural adsorbents.

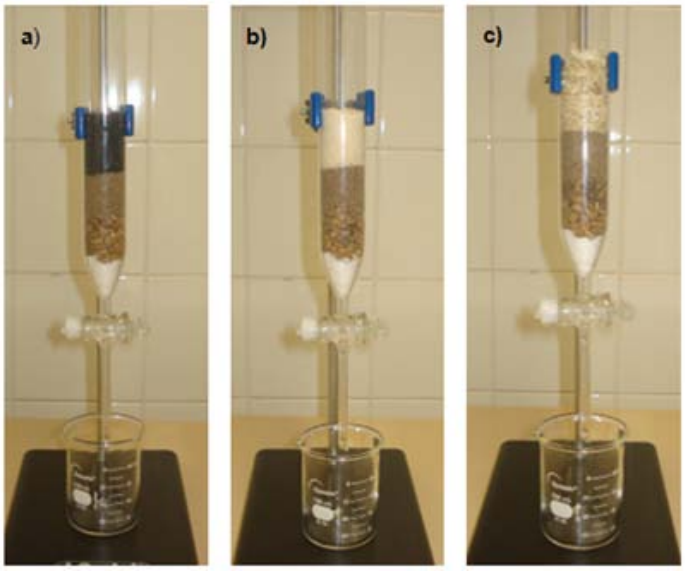

Fig. 1 Columns simulating real systems in water treatment plants: gravel, sand and AC (a); gravel, sand and SCB (b); and gravel, sand and VS (c).

RESULTS

SEM and SAAP analysis

The SEM micrographs of the two bioadsorbents (Figure 2) show that the SCB surface is smooth, with pores of about 1 µm in diameter (macro pores). In contrast, the VS surface is irregular and exhibits few pores.

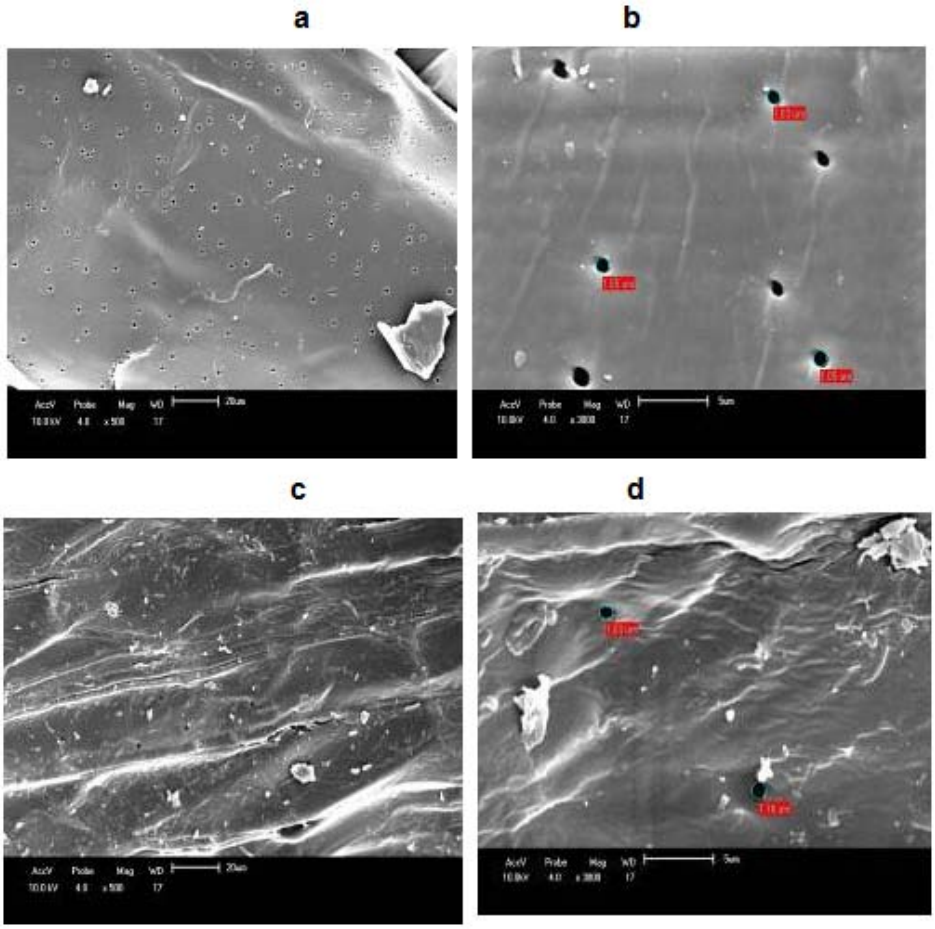

Fig. 2 SCB (particle ≤ 1.19 mm) and VS (particle ≤ 4.76 mm) surface SEM micr graphs. Figures (a) and (b) correspond to the SCB sample with magnifications of 500 and 3,000 times, respectively. Figures (c) and (d) correspond to the VS sample with magnifications of 500 and 3,000 times, respectively.

Table 1 summarises the SAAP analyses concerning both SCB and VS surface area and pore distributions. According to these results, both bioadsorbents have many macropores, with the average diameter exceeding 50 nm. However, SEM analysis (Figure 2) reveals that the SCB particles have a number of pores larger than those observed on the VS particles; additionally, SCB particles have a larger surface area.

Adsorption isotherm building and maximum adsorptive capacity (MAC_F) calculations

In order to verify the adsorptive capacity of the SCB and VS front for paracetamol dissolved in aqueous solutions, adsorption isotherms were constructed (Figure 3). In a next step, the isotherms (Figure 4) were linearised according to the mathematical model of Langmuir (Kannan and Murugavel, 2007) in

order to calculate the MAC_F. Table 2 presents the MAC_F values for both adsorbents, showing that SCB (MAC_F = 120.5 µg/g) was considerably more efficient than VS (MAC_F = 37.5 µg/g).

Table 1. Surface area and pore distribution of vegetable sponge and sugar cane bagasse using N_2 adsorption.

Sample	Surface area (m^2g^{-1})	Pore distribution (> 50 nm)
VS	0.98	Macro pores
SCB	1.49	Macro pores

It is possible to consider that the MAC_F values are appropriate for both bioadsorbents, because pharmaceuticals (as paracetamol) are commonly found in water for human consumption at ng/L or µg/L levels (Hilton and Thomas, 2003). These concentration levels ensure quantitative retentions of paracetamol before column saturation is reached.

Paracetamol removal from enriched water samples

After assessing the capabilities of SCB and VS for removing paracetamol from synthetic aqueous solutions, their efficiencies for removing paracetamol from enriched pretreated water samples (5 µM) were verified. Finally, SCB and VS efficiencies were compared with Activated Carbon (AC), commonly used in treatment plants. The data obtained show that filters containing SCB, AC and VS were able to absorb 60%, 45% and 40% of the available paracetamol, respectively (Figure 5).

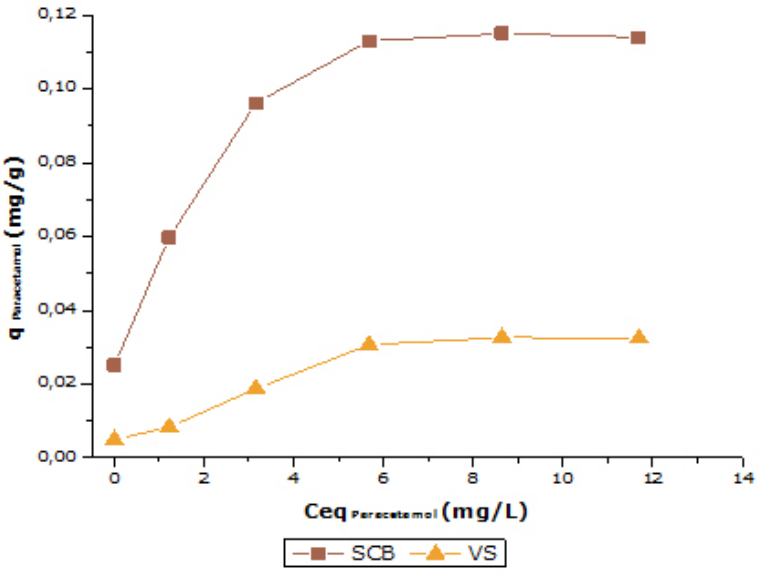

Fig. 3 Adsorption isotherm for paracetamol using columns packed with SCB and VS.

DISCUSSION

The SCB surface is smooth, with pores of about 1 µm in diameter (macropores). On the contrary, the VS surface is irregular and has almost no pores (Figure 2). Similar to the morphology, the surface area and porosity of the adsorptive material can be associated with its efficiency for removing pollutants.

The accessibility of adsorbate molecules to the adsorbent's pores is directly related to pore size and shape (Guo et al. 2008). According to IUPAC, pores are classified into micropores (< 2 nm diameter), mesopores (2 to 50 nm diameter) and macropores (> 50 nm diameter) (Tseng et al. 2003).

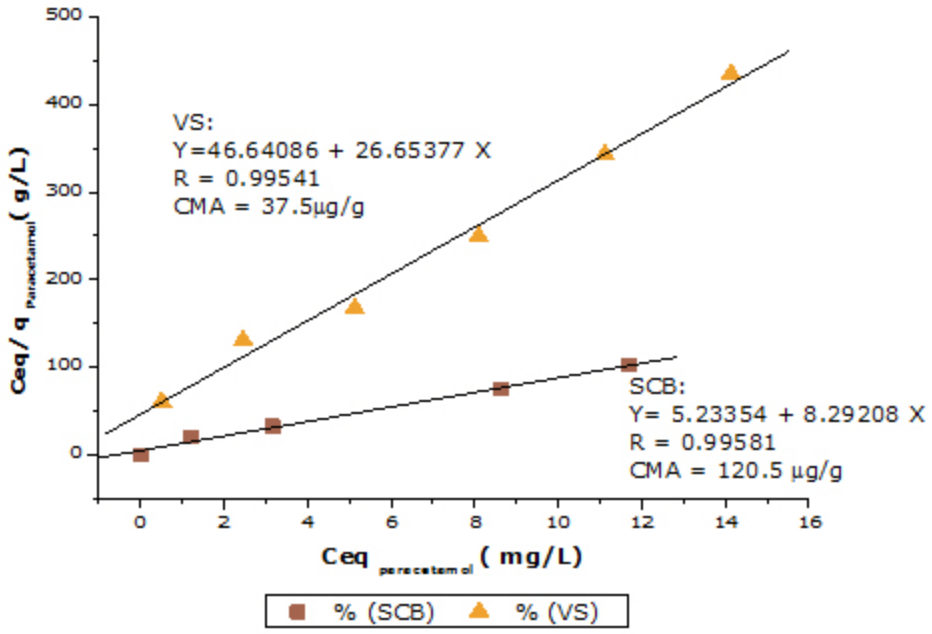

Fig. 4 Linearisation of the adsorption isotherms according to the mathematical model of Langmuir.

The SEM analysis (Figure 2) revealed that the SCB particles have a number of pores larger than those observed for VS particles; SCB particles also have a larger surface area (Table 1), which may represent a relative advantage regarding the physical interaction with paracetamol. However, the surface area of both adsorbents is low, when compared with AC, with an average surface area of 30 to 1,487 m^2/g (Ryu et al. 1999). Yurtsever and Şengil (2009) used resins containing tannins for the removal of Pb(II), and they demonstrated that these resins have high maximum adsorptive capacities, even with reduced surface area. This was ascribed to the interactions of lead ions with functional groups and not only to the deposition of the pollutant on adsorbent pores.

Besides presenting low surface areas and pores mainly distributed in the macropore range, the results revealed that the SCB particles presented considerable adsorptive potential, probably due to the formation of pollutant monolayers on the material surface, mostly ascribed to the probable large amount of functional groups that favour adsorption. This is because bioadsorbents are natural biopolymers mainly composed of cellulose, hemicellulose and lignin, in varying proportions, depending on soil properties and the plant's development stage. According to Meza et al. (2006), SCB is composed of about 50% cellulose, 25% hemicellulose and 25% lignin while VS is formed of 60% cellulose, 30% hemicellulose and approximately 10% lignin (Ghali et al. 2009). Infrared analysis of these biopolymers has revealed important groups capable of interacting with adsorbates such as C=O, -C-H_n, OH and aromatic rings (Tanobe et al. 2005; Sousa et al. 2009). Observation of the paracetamol structure (Figure 6) suggests that their functional groups are able to establish weak interactions with the chemical groups in the SCB and VS structures. Cabrita et al. (2010) performed an important investigation on paracetamol removal from aqueous media by using chemically modified activated carbon. In the cited work, the chemical properties of the activated carbon were modified by means of wet oxidation. The authors identified interesting surface heterogeneity, which was a determinant of the success attributed to the adsorbent. After oxidation, an increase in carbon wettability was observed, which favours the transfer of paracetamol molecules to the carbon pores.

Table 2. MAC_F values for SCB and VS.

Bioadsorbent	MAC_F (µg/g)
SCB	120.5
VS	37.5

As already mentioned, the maximum adsorption capacities of SCB and VS were 120.5 and 37.5 µg/g, respectively. These values can be partially explained because SCB, with particles≤ 1.19 mm, seems more compact and more uniform than VS (particles ≤ 4.76 mm) in the percolation column (Malkoc et al. 2006). This factor, allied to the more porous matrix of SCB when compared to VS (Figure 1), allows for greater penetration and permanence of paracetamol through the SCB bioadsorbent, thus increasing its adsorptive efficiency. Moreover, the MAC_F values were relevant, since the paracetamol concentrations observed in drinking water are of the order of ng/L (Rabiet et al. 2006; Conley et al. 2008).

The data show that SCB, AC and VS were able to absorb 60%, 45% and 40% of paracetamol from enriched water samples (Figure 5). The tested concentration of paracetamol (5 µM) was higher than the levels commonly found in water consumed by humans. In this way, the adsorption percentages above cited are sufficiently elevated for the use of both bioadsorbents in water treatment plants. Among the commercial adsorbents, AC is currently the most widely used in wastewater treatment. However, a high cost is ascribed to AC when employed in decontamination processes (Brandão et al. 2010). During this research, for example, it was found that the paracetamol removal capacity (in enriched samples) of SCB was higher than that observed for VS and AC. Furthermore, SCB is more economically feasible when compared to AC, since SCB is an agroindustrial residue abundantly produced and often wasted in Brazil.

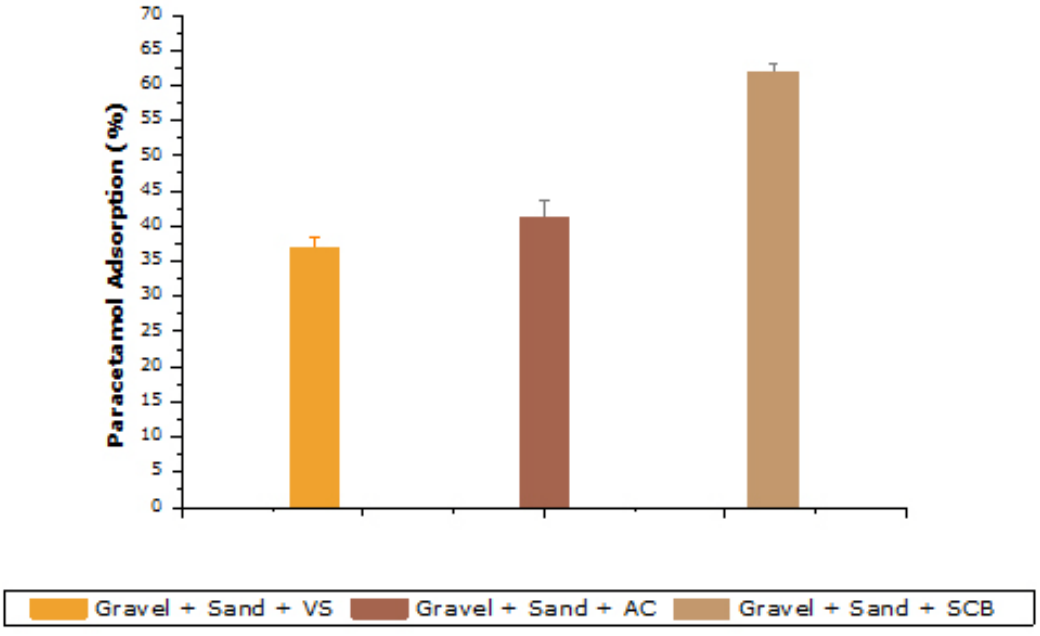

Fig. 5 Percentage of paracetamol removal for VS, AC and SCB in columns that simulate the treatment plants of CESAN.

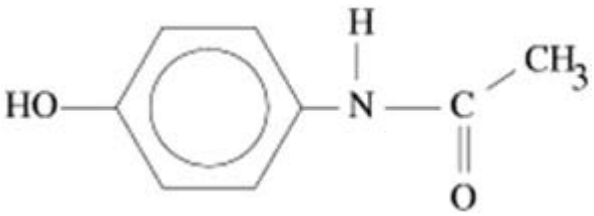

Fig. 6 Paracetamol molecular structure.

CONCLUDING REMARKS

The data suggest that SCB is more efficient than VS and AC for removing paracetamol from the water supply. This feature, as well as its wide availability and low costs, should stimulate more detailed studies concerning the use of SCB as an alternative filter in treatment stations.

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