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This study evaluates the performance of a polymeric microfiltration membrane, as well as its combination with a coconut granular activated carbon (GAC) pretreatment, in a gravitational filtration module, to improve the quality of water destined to human consumption. The proposed membrane and adsorbent were thoroughly characterized using instrumental techniques, such as contact angle, Brunauer–Emmett–Teller) and Fourier transform infrared spectroscopy analyses. The applied processes (membrane and GAC + membrane) were evaluated regarding permeate flux, fouling percentage, pH and removal of *Escherichia coli*, colour, turbidity and free chlorine. The obtained results for filtrations with and without GAC pretreatment were similar in terms of water quality. GAC pretreatment ensured higher chlorine removals, as well as higher initial permeate fluxes. This system, applying GAC as a pretreatment and a gravitational driven membrane filtration, could be considered as an alternative point-of-use treatment for water destined for human consumption.

Keywords: water quality; activated carbon; membrane

Introduction

Clean drinking water is essential to human life and good health. Consumption of untreated water leads to infectious diseases, including diarrheic ones and others that are faecal–oral in nature [1]. The main indicator of the sanitary quality of drinking water is the coliform, particularly *Escherichia coli*, since a high count of this bacterium is an indication of contamination from a septic system or other faecal pollution source (drinking waters must have *E. coli* levels equal to 0 MPN/100 ml) [2].

According to World Health Organization [3], many people in the world do not have access to improved drinking water, almost all of them in developing regions. Moreover, there is a lack of access to adequate water supply in outlying regions, such as rural areas. Even when centralized treatment plants are available, contamination of piped drinking water may occur between the distribution system and the final consumption point, that is, 'between tap and mouth' [4]. Variation in raw water quality according to seasons and rainfall is a complicating factor in the task of assuring safe drinking water by centralized treatments. These variations can also lead to the addition of chemicals at super dosage.

Chlorine and its products are the most commonly used agents used in water disinfection, because they are cheap and effective, destroying or inactivating microorganisms that may cause water-borne diseases [5]. However, during chlorination, natural organic matter (NOM) reacts with chlorine and produces a number of byproducts with harmful long-term effects [6]; a complex mixture of chlorine byproducts is formed and more than 300 different types of DBPs (disinfection byproducts) can be identified [7,8].

Decentralized systems may be used as a household water treatment method and are especially useful for areas without a water supply system, or when the centralized system does not ensure water quantity and/or quality. Sobsey *et al*. [9] reported that point-of-use treatments improve household drinking water quality and reduce diarrhoeal disease risks. Peter-Varbanets *et al*. [4] presented a review about point-of-use systems for water pathogen removals based on the membrane separation process.

Membrane separation is a suitable process for water treatment, since it can provide an absolute barrier for bacteria and viruses, in addition to removing turbidity and colour [10]. Most membrane processes are pressure driven and require pumps to promote the transmembrane pressure, resulting in energy consumption and relatively high costs of auxiliary equipments. Alternatively, membrane processes can be designed to operate with gravity as the driving

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force. Huang *et al*. [11] highlighted that the application of low-pressure membrane technologies for water treatment has grown since the beginning of the past decade, the 1990s. Peter-Varbanets *et al*. [12] listed a few known membrane processes that use gravity as the driven force for water treatment.

The gravity-driven membrane process is an effective technology to improve the quality of the water by removing pathogenic microorganisms, and is a suitable point-of-use method to obtain safe drinkable water, since it is independent of energy supply, chemicals, or complex process control [12]. However the process effectiveness also depends on a proper method for the maintenance and cleaning of membranes.

Essentially, fouling is a major constraint during separation using membranes. Its occurrence leads to a decline in membrane permeability. Integration of a pretreatment with the membrane filtration process has been widely employed to reduce membrane fouling and/or to improve the removal of certain contaminants [13,14]. Kweon *et al*. [15] evaluated the advantages of coagulation and powdered activated carbon (PAC) adsorption pretreatments on a membrane filtration pilot plant installed in Korea, suggesting the removal of floc aggregates that are formed after coagulation between PAC stages. Matsui *et al*. [16] proposed the utilization of smaller particles of PAC in order to create a more permeable cake layer on the membrane surface. In fact, pretreatment effects on membrane fouling reduction can be small or even negative, depending on factors such as the source water quality and membrane properties [11]. These parameters must be carefully analysed in order to improve the process effectiveness. In order to bypass possible fouling increase caused by PAC, Schideman *et al*. [17] proposed the application of granular activated carbon (GAC). On the other hand, Papastavrou *et al*. [18] observed a faster adsorption of organic material based on chemical oxygen demand (COD) using PAC rather than GAC. Previous studies reported the application of GAC with membrane filtration for water treatment [19,20]. Huang *et al*. [11] signalized the importance of additional studies to understand the effects of GAC pretreatment on membrane fouling. GAC made from coconut is a premium carbon for residential or commercial drinking water treatment applications, since it performs better than standard carbon charcoal filtering media in removing chlorine and volatile organic compounds [21].

This study evaluates the application of a polymeric microfiltration membrane in a gravitational filtration module and its combination with coconut GAC pretreatment. The main objective is to propose a simple module that ensures water quality for human consumption and is to be applied as a household filter. The proposed membrane and adsorbent were thoroughly characterized using instrumental techniques, such as contact angle, Brunauer–Emmett–Teller (BET) and Fourier transform infrared (FTIR) spectroscopy analyses. Filtration tests were carried out in order to evaluate membrane fluxes, resistances and fouling, in addition to colour, turbidity, excess chlorine and *E. coli* removals in the collected permeate.

Materials and methods

Materials and characterization

Mixed cellulose ester microfiltration membrane (pore diameter = 0.65 μm) was purchased from ADVANTEC (Japan). Membrane hydrophilicity and groups on the membrane surface were measured in this work in order to complement the manufacturer's information. The hydrophilic and/or hydrophobic characteristics of the membrane were determined using a DataPhysics contact angle system (OCA, Germany). Groups on the membrane surface were determined by FTIR spectroscopy using an Avatar 360 ESP FT-IR spectrometer (Thermo Nicolet, Madison, WI). Spectra were recorded in the range of 500–4000 cm^{-1} at a resolution of 4 cm^{-1} by averaging 30 scans.

Commercial GAC 20 × 40 mesh made from coconut was supplied by BAHIACARBON (Brazil). GAC pore characteristics (surface area, pore volume and mean pore size) were measured in this work in a Quantachrome Autosorb Automated Gas Sorption System (Florida, USA). BET specific surface areas were determined from the 77 K N_2 adsorption–desorption isotherms. Micropore area (S_m) was determined by means of the *t*-method of Halsey. The procedure is the same as that employed in the BET surface area measurement, but it extends the pressure range to higher pressures. Pore volume (V_p) and average pore diameter (d_p) were calculated using the Horvath–Kawazoe (HK) equation derived independently from the Kelvin equation.

Gravitational filtration system

Figure 1 shows the gravitational filtration system proposed in this work. This system has a 20 l main tank (1) to store raw water and to supply the feed tank (2). In addition, there is an overflow (3) to keep the level of raw water constant. The feed tank is placed at a particular height from the filtration module (4), corresponding to the required pressure. In this work, tests were carried out at 30,397.5 Pa (3.1 m water column). The flat membrane is fixed at the bottom of the filtration module. The effective membrane surface area is 1.962×10^{-3} m^2. Permeate is collected perpendicularly to the membrane area, by means of a dead-end operation.

Additional tests were carried out to compare the effects of GAC pretreatment on membrane permeability and permeate quality. For these tests, a packed-bed filter was placed inside the filtration module, before the flat membrane. This filter was obtained by packing 150 g of GAC in a cartridge of 20 cm height acrylic cartridge.

This process is proposed without chemical cleaning, which would greatly simplify the maintenance and operation of membrane filtration systems [12].

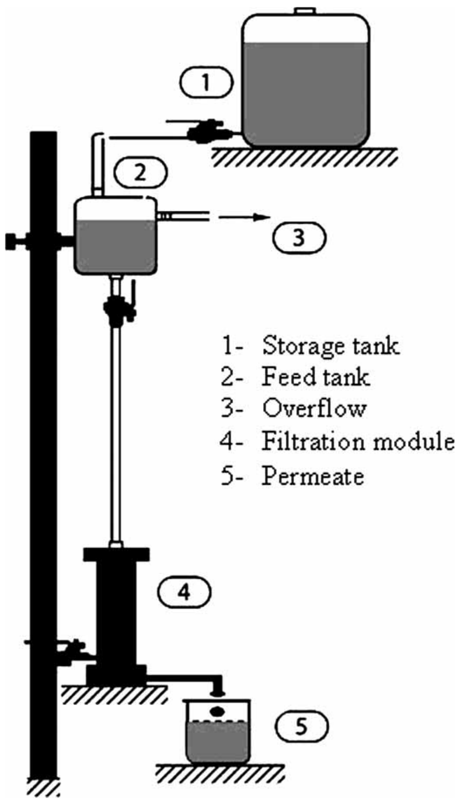

Figure 1. Gravitational filtration system scheme.

Tests in the proposed gravitational membrane system (with and without the GAC pretreatment) were performed in five sequential steps.

(1) Determination of initial permeate flux for deionized water filtration with a clean membrane.
(2) Filtration of deionized water artificially contaminated with *E. coli* between 1.0×10^5 and 1.0×10^6 CFU/100 ml.
(3) Four subsequent filtrations of tap water for 120 min, totalling 480 min of filtration.
(4) Second filtration of water artificially contaminated with *E. coli*, as described in step (2). This test was carried out to evaluate membrane efficiency for *E. coli* removal after tap water filtration.
(5) Determination of final permeate flux for deionized water filtration.

Water samples and analyses

Two types of water were used in this work: deionized water artificially contaminated with *E. coli* and tap water from Maringa city (Brazil).

An agar plate diffusion procedure was used to prepare deionized water artificially contaminated with *E. coli*. A standard culture of *E. coli* was enriched in a nutrient broth for 20–22 h. A bacterial suspension was prepared from this enriched culture to a turbidity of 0.5 of tube #1 in the MacFarland scale using sterile isotonic saline solution. The standardized innoculant were spread over the surface of 15 cm Petri dishes containing 6 mm of Mueller–Hinton agar using sterile cotton swabs. After 15 min of pre-diffusion, 15 mg of the prepared sample was placed over the seeded agar plates. The plates were then incubated at 37°C for 20–22 h and the diameters of the inhibition zones were measured. These assays were done in duplicate.

After the filtration of contaminated water, *E. coli* removal was evaluated using the membrane filter technique for members of the coliform group, as described in the Standard Methods for the Examination for Water and Wastewater [2].

The characteristics of the tap water available at the Chemical Engineering Faculty (Maringá, Brazil) are presented in Table 1. Residual chlorine concentration in this tap water was adjusted to 1.5 mg l^{-1} using a solution of 14.0 wt% sodium hypochlorite. Removal of colour, turbidity and free chlorine, pH variation and total volume of filtrated were also measured after filtration. Turbidity and colour were measured with a Hach 2100N turbidimeter and a Hach DR-A colorimeter, respectively. Free chlorine residuals were measured using a HACH DR/4000 spectrophotometer.

Table 1. Characteristics of tap water used in step (3).

Parameter	Value
pH	7.80
Colour (UC)	4.93
Turbidity (UT)	1.15

Membrane resistance analysis

Resistances due to different fouling mechanisms were determined in order to investigate the fouling behaviour. Resistances were calculated following the resistance-in-series model adapted from Schafer *et al*. [22]:

$$J = \frac{\Delta P}{\eta(R_m + R_p + R_c)} \tag{1}$$

where J is permeate flux [kg m^{-2} s^{-1}], ΔP is transmembrane pressure [kg m^{-1} s^{-2}], η is dynamic viscosity [kg m^{-1} s^{-1}] and R denotes a resistance [m^2 kg^{-1}]: R_m is membrane hydraulic resistance, R_p is resistance due to pore blocking and R_c is resistance due to cake formation.

Each resistance was experimentally measured in the gravitational system with and without GAC pretreatment. Membrane hydraulic resistance, R_m, was determined measuring the flux of deionized water through a clean membrane sheet. In this case, the others resistances are equal to zero.

The sum of resistances due to pore blocking and cake formation ($R_p + R_c$) was determined measuring the flux of deionized water with the fouled membrane, that is, with the membrane that was used for raw water filtrations without clean procedures.

After that, the fouled membrane was gently cleaned with a sponge to remove the cake layer from the surface. Deionized water was then filtered once again through this same membrane sheet to obtain the resistance due to pore blocking, R_p [23].

Membrane fouling percentage ($\%F$) was calculated according to Equation (2), as proposed by Balakrishnan *et al*. [24]. This percentage represents the drop in deionized water flux after the tests of filtration with raw water:

$$\%F = \frac{(J_i - J_f)}{J_i} \times 100 \quad (2)$$

where $\%F$ is the membrane fouling percentage, and J_i and J_f are deionized water fluxes in clean and fouled membranes, respectively.

Results and discussion

Characterization of materials

The obtained contact angle was 66.3°, showing that the chosen membrane is slightly hydrophilic. This value was averaged over 12 measurements from two membranes.

The FTIR spectrum of the clean membrane is shown in Figure 2. Peaks at 1650, 1280, 1068 and 841 cm^{-1} confirm the presence of ester groups on the membrane surface, as described by the manufacturer. The obtained spectrum is similar of those reported by Rajam and Ho [25] for an unmodified mixed cellulose ester membrane.

Results of activated carbon characterization are presented in Table 2. The activated carbon used in this work is predominantly microporous, with a micropore area corresponding to 94.73% of the total surface area, and an average pore diameter of 20.04 Å. These values are in qualitative agreement with those reported by Ji *et al*. [26] and Li *et al*. [27]. These characteristics of the proposed adsorbent ensure a high adsorption rate. Moreover, the material is obtained from a vegetal source (coconut shells), giving the advantage of sustainability.

Table 2. Textural characteristics of the activated carbon used in this work.

Surface area ($m^2\,g^{-1}$)	Micropore area ($m^2\,g^{-1}$)	Total pore volume ($cm^3\,g^{-1}$)	Average pore diameter (Å)
715.46	677.77	0.3856	20.04

Permeate fluxes

Figure 3 presents flux values along the filtration of tap water with and without GAC pretreatment. Discontinuities are observed, since the filtration was carried out in four sequential steps of 120 min.

These results show that the initial flux obtained with GAC pretreatment is greater than without it. However, after approximately 150 min, the flux achieved the same stabilized value with both proposed processes. Another important observation from these obtained results is that the flux decay is faster in the process without GAC pretreatment than in the process with GAC pretreatment.

Regarding the membrane working alone at lower transmembrane pressures, Peter-Varbanets [12] observed similar stabilized fluxes (4–10 $l\,h^{-1}\,m^{-2}$) filtering river and lake water for 30 days with a polyethersulfone ultrafiltration membrane at 65 mbar.

Meier [28] also observed similar fluxes for nanofiltration experiments with PAC suspended in water. Gai and Kim [29] studied the effects of PAC with an immersed flat-sheet membrane and observed smaller transmembrane pressure values for the system working with PAC.

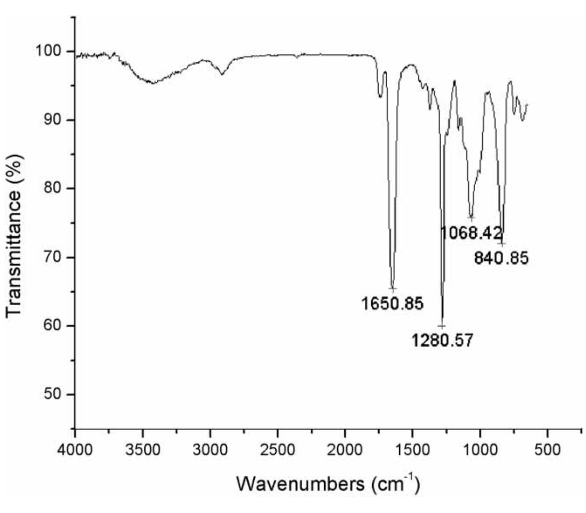

Figure 2. FTIR spectrum of the mixed cellulose ester microfiltration membrane (pore diameter = 0.65 μm).

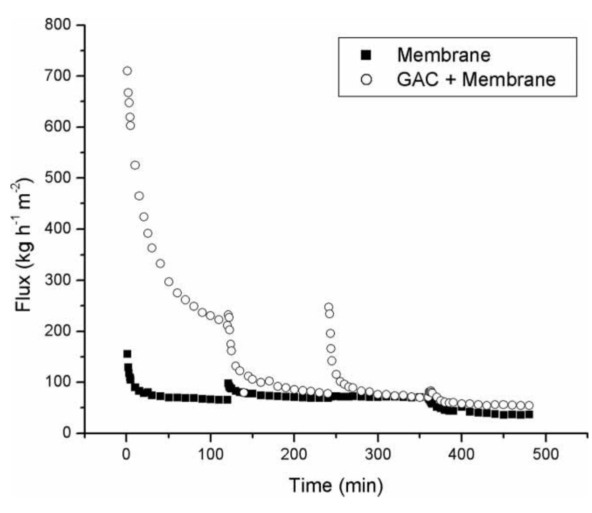

Figure 3. Permeate flux of tap water with and without GAC pretreatment.

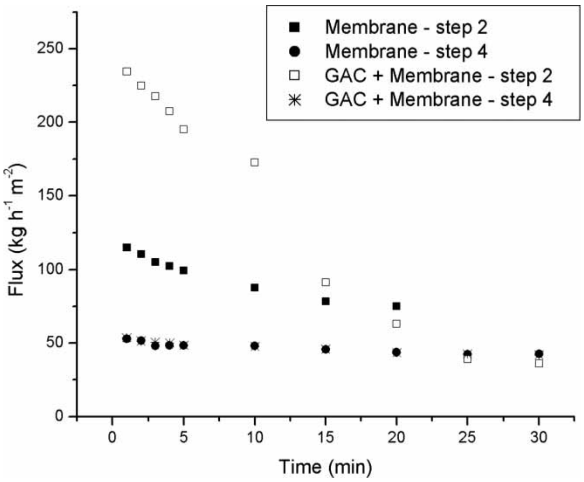

Figure 4. Permeate flux of water contaminated with *E. coli* with and without GAC pretreatment.

Figure 4 presents the obtained fluxes filtrating water artificially contaminated with *E. coli*. In this case, two separated assays were carried out: one before tap water filtration, as described in step 2, and another one after the tests with tap water (step 4). The obtained results show that the flux achieves the same value after 20 min of filtration. The initial flux is higher with GAC pretreatment working with a clean membrane. However, after the test with tap water, the initial flux is smaller, and the GAC pretreatment does not ensure higher initial fluxes.

Fouling percentages, calculated by Equation (2), and membrane resistances, calculated following the resistance-in-series model, are shown in Table 3.

The coupling of GAC treatment with membrane filtration decreased the membrane fouling. Kang and Choo [30] observed that pretreatments with powdered carbon had almost no effect on membrane fouling mitigation. This result is probably related with the portion of NOM that is removed by the adsorbent.

The results presented in Table 3 show that the combination of the 0.65 μm membrane with activated carbon adsorption pretreatment affected resistance values. The membrane hydraulic resistance (R_m) was determined only for the system without GAC, since R_m is an inherent property of the used membrane.

The resistance due to pore blocking (R_p) increased when the combined process was applied, while the resistance due to cake formation (R_c) decreased. The total resistance (R_t) is smaller in the combined process. In fact, GAC addition decreased the resistance caused by cake formation.

Table 4. Results obtained for the evaluated parameters.

		Removal (%)				
Process	pH	Colour	Turbidity	Free chlorine	E. coli	Filtered volume (l)
Membrane	7.17	100	73.3	94	100	4.4
Membrane + carbon	7.95	100	71.8	95	100	12

Impact of purification processes on water quality

Results regarding colour, turbidity, free chlorine, *E. coli* removal and pH are presented in Table 4 for the proposed processes. Table 4 also presents the volume that was filtrated with both of the proposed systems (with and without GAC pretreatment) at the same filtration time (480 min).

These results show that both evaluated processes provided filtered water with similar quality. According to the analysis of variance (one-factor ANOVA) to $p < 0.05$, only the chlorine removal presented significant difference between both analysed processes. In this case, the p-value ($p = 0.0031$) is almost 16 times smaller than the limit. For turbidity removal and pH variations, the p-values are equal to 0.1106 and 0.9924, respectively. The variables regarding colour and *E. coli* removal were not statistically analysed, since there is no standard deviation and variance between the obtained values for both proposed processes.

Phan *et al.* [21] reported the higher performance of coconut GAC in removing chlorine.

The combination of activated carbon with membrane filtration increased the obtained volume of filtrated water during the analysed time (480 min). Gai and Kim [29] analysed the influence of PAC pretreatment during 64 days of water filtration, and observed that PAC prolonged the continuous filtration time by mitigating membrane fouling. However, since the stabilized flux tends to be the same with both processes after 200 min of filtration (Figure 3), the filtrate volume in the long-term may be the same. The effect of GAC pretreatment must be investigated on permeate for at least during 500 days in order for it to be applied as a household filter.

Conclusions

The obtained results showed that it is possible to enhance the quality of tap water in a gravitational driven membrane process, where pumps are not required. The membrane process guarantees the water quality, mainly in terms of

Table 3. Fouling percentages and membrane resistances for the both proposed processes.

Process	Fouling (%)	R_m ($m^2\,kg^{-1}$)	R_p ($m^2\,kg^{-1}$)	R_c ($m^2\,kg^{-1}$)	R_t ($m^2\,kg^{-1}$)
Membrane	96.02	4.74×10^7	1.30×10^8	9.41×10^8	11.9×10^8
Membrane + GAC	85.11		3.71×10^8	0.563×10^8	5.47×10^8

bacteria removal. GAC pretreatment ensured higher chlorine removals, as well as higher initial permeate fluxes. The combination of membrane filtration and activated carbon adsorption processes reduced the resistance, mainly due to cake formation. This system, applying GAC as a pretreatment and a gravitational driven membrane filtration, could be considered as an alternative point-of-use treatment of water destined to human consumption.

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