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# ACTIVATED CARBON BASED COMPOSITE MEMBRANES: CHARACTERIZATION AND SELECTIVITY IN FILTRATION TESTS[1]

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**Abstract**

Composite membranes based on epoxy resin and activated carbons were developed for use in purification of drinking water contaminated by bacteria and others pathogenic, typically found at the north-east of Brazil. The untreated waters, especially in areas where various species of animals transit and drink, are frequently contaminated with their waste, thus, bacteria growth in the drinking water distribution systems which can lead to several problems including deterioration in water quality, violation of water quality standards, acceleration of pipe corrosion and in incidence of bacteriological diseases like cholera, diarrhea in infants and dysentery, specially between the poor population which is a factor of particular impact in lowering the human development index in municipals and country as a whole. In this work, characterization and evaluation of the selectivity and permeability of composite membranes made of national activated carbon was carried out. The study was performed with mercury porosimetry measurements, the physical properties using Arquimedes method and by the quantification of aerobic, heterotrophic cultivated bacteria from contaminated water. The results showed that the membranes have apparent porosity between 45%-55%, pore size distribution is predominantly of meso and macropores and the selectivity due to presence of bacteria in the polluted water filtered by the best selective membrane was more than 99%.

**Key words:** Composite membranes; Activated carbon; Selectivity; Bacteria; Water purification.

## MEMBRANAS COMPÓSITAS A BASE DE CARVÃO ATIVADO: CARACTERIZAÇÃO E SELETIVIDADE EM TESTES DE FILTRAÇÃO

**Resumo**

Membranas compósitas a base de resina e carvão ativado foram desenvolvidas visando o uso em purificação de águas doces contaminadas por bactérias e outros patógenos, tipicamente presentes no Nordeste do Brasil. As águas não tratadas, especialmente os acúmulos aonde transitam e bebem diversas espécies de animais, são freqüentemente contaminadas com dejetos e bactérias que causam um cem número de doenças como o cólera, diarréias infantis e disenterias, especialmente nos extratos da população menos assistida, sendo fator particularmente impactante na diminuição do IDH municipal e do país. Neste trabalho, foram caracterizadas e avaliadas quanto à seletividade membranas compósitas desenvolvidas com carvões ativados nacionais. A caracterização das membranas foi realizada por porosimetria de mercúrio, pela medida das propriedades físicas (método de Arquimedes) e pela quantificação de bactérias heterotróficas aeróbicas cultiváveis, provenientes de testes de filtração de uma água contaminada. Como resultado, as membranas mostraram porosidade aparente próximas de 60%, a distribuição dos poros das membranas foi predominantemente de meso e macroporos e a seletividade quanto à presença de bactérias cultivadas, a partir da água filtrada pela membrana mais seletiva, foram de 99%.

**Palavras-chaves:** Membranas compostas; Carvão ativado; Seletividade; Bactéria; Purificação de águas.

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## 1 INTRODUCTION

Membrane separation technology has recently received great attention as a new approach to supply stable and economically efficient water. The establishment of the proper operation factors for membrane separation system is important because raw waters are different with regions and their climates.
Membrane separation processes use semi permeable membranes to separate impurities from water or solvents. The membranes are selectively permeable to water and certain solutes. A driving force, which is often pressure or concentration gradient across the membrane, is used to force the water to pass through the membrane, leaving the impurities behind as a concentrate.[1] The advantages offered by the process include high stability and efficiency, low operating cost and capital, low energy requirement and also ease of operation.[2-5] Furthermore, it is also known for its reliability to be used in remote locations as membrane has no moving parts and thus making it mechanically robust.
Subjects related to the selection of membrane materials for a given separation are complex depending on the application and it remains one of the most technologically challenging factors. The other criteria to be taken into consideration are productivity, durability and mechanical integrity at the operating conditions that must be balanced against cost.[6] Generally, the permeation and selectivity are the most commonly compared basic performance characteristic. In a broader context of definition, permeability is the ability of the permeates to pass through a membrane meanwhile the ratio of permeability of the more permeable component to that of the less permeable is known as selectivity of the membrane. The higher the permeability of the membrane used, the smaller is the membrane area required, on the other hand, the higher the selectivity, the lower the driving force required. Such ideal conditions thus lower the capital and operating cost of the separation system. Therefore, for economical consideration, selectivity and permeation rate are important parameters to be maximized in order to provide commercially attractive outcome and achieve more efficient separation in all applications.[7] On the other hand, due to the significant disorder and complexity of the pore network, the characterization of porous membranes is extremely difficult. That is why the widely used experimental tool is permeability and selectivity measurements, which in principle give macroscopic information on the system.
Many groups worked with porous carbon and activated carbon membranes for separation and purification of gases and liquids. Ismail et al.[7] found that when non polar carbon nano tubes (CNTs) with narrow pore diameter are used as membrane, it is possible to allow the permeation of water molecules through the pore but not ions. The ions will face a large energy barrier hindering their entrance to the narrow pore while water requires little or no energy for the entry due to the formation of stable hydrogen bonds. Anderson et al.[8] studied the effect on the performance of a nano porous carbon (NPC) membrane upon exposure to condensable impurities typically found in natural gas (e.g. water, hexane and toluene) and non-condensable impurities typically found in synthesis gas (e.g. $H_2S$ and CO) has been determined in laboratory experiments. Small reductions in the permeance (less than 30%) and minimal reductions in selectivity have been observed, with the greatest impact at 35°C and less impact at 100°C. However, these relatively limited reductions in membrane performance suggest that such carbon membranes still offer some promise for pre-combustion applications. The elemental composition and bacteria attached in particles were investigated by Tao, Wei and Leilei[9] during granular

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activated carbon (GAC) filtration. The experimental results showed that trapped influent particles could form new, larger particles on GAC surface. The sloughing of individuals off GAC surface caused an increase in effluent particles in the size range from 5 µm to 25 µm. The selectivity for element removal in GAC filters caused an increasing proportion of metallic elements in the effluent particles.
In the present work, composite membranes produced from a mixture of micro, meso and macroporos activated carbon and epoxy resin, were used in permeability and selectivity experiments performed at room temperature.

## 2 MATERIALS AND METHODS

The composite membranes used in this work were fabricated from epoxy resin as a polymeric matrix and national grade activated carbon of different pore sizes named as: Cod A and cod A2 from a mixture of macro-meso activated carbon, cod CB macro-meso-micro and cod B of macro-meso activated carbon. The membrane was extruded and machined to form a cylinder (Figure 1). For example, the dimensions of the membranes cod B is 33.2 mm outer diameter, 27.2 mm inner diameter, inner height of 94 mm and 100 mm outer height with filtration area of 84.826 $cm^2$. The area of the permeation layer was calculated for each membrane so as to calculate the flow per surface area.

**Figure 1.** Prototype of AC composite membrane used in the research.

The morphology and pores distribution of the membranes was studied by scanning electron microscopy (SEM). The physical properties of the membranes were calculated by Arquimedes method, their porosity and pores volume and size distribution was carried by mercury porosimeter.
For the test of selectivity, samples of polluted water from a lake at Campo de São Bento at the city of Niterói, at state of Rio de Janeiro, Brazil, was used as raw water in this research. The samples were collected in plastic bottles only washed with alcoholic solution. Selectivity measurements were carried out on home designed apparatus (using polluted water as feed). Constant volume of water was fed into the AC composite membrane and a vacuum pump was used as driving force. The quantification of cultivated heterotrophic aerobics bacteria in the samples was carried out by the laboratory of microbiology of Division of Corrosion of the National Institute of Technology – MCTI/INT/DCOR, Rio de Janeiro, Brazil, on as received sample and

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after filtration with the three of the four membranes AC composite membranes studied and the selectivity percentage was calculated from the quantity of bacteria in the permeated water and from the bacteria measured in the as received water (feed).
The tests of selectivity and permeation were held also for a suspension of 1% sub micrometric α-alumina in water, using 50% diluted hydrochloric acid (HCl) as a deflocculating agent to reach a ph 4; the mixture was dispersed for 2 min in ultrasound device. The Permeation test was held for each AC membrane in a permeation cell; first with a tank containing water with ph 4 and then, with the suspension of 1% sub micrometric α-alumina in water. The experiments were performed using three different values of the pressures applied at the base and sides of the membranes: 1 kgf/cm$^2$ (0.9807 bar), 2 kgf/cm$^2$ (1.9613 bar) and 3 kgf/cm$^2$ (2.942 bar) for each membrane. For each pressure the flow was measured versus time. The time accumulated from 30 seconds to 3.600 seconds.
The total permeate volume P (measured in ml/m$^2$), through the membrane was given by Equation 1.

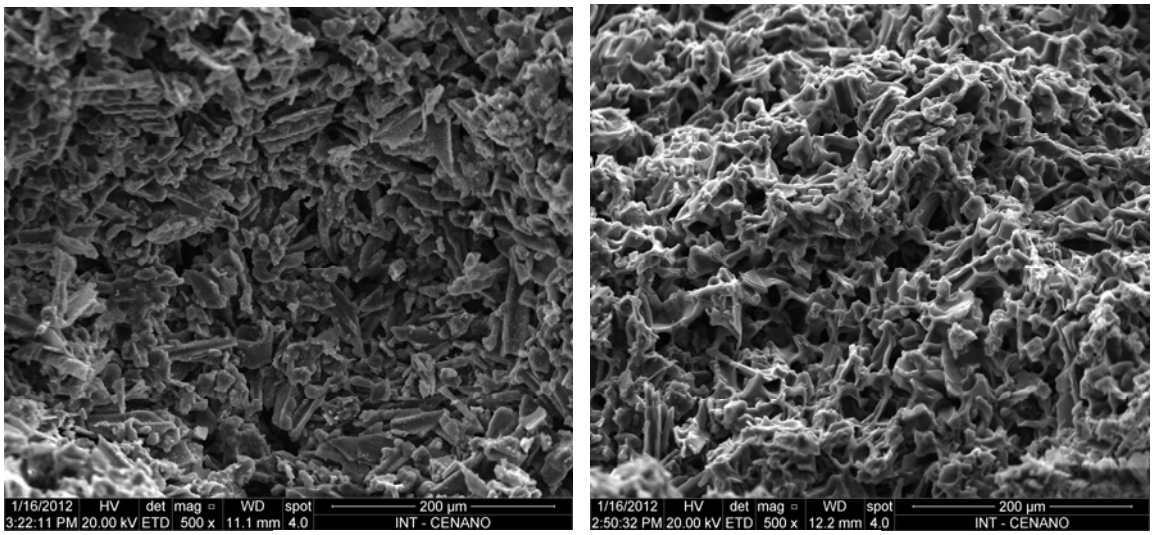
$$P = F_{perm}/A \qquad (1)$$

Where $F_{perm}$ is the total permeate volume side flow rate, and A the total area of the membrane.
Permeate samples were collected as well as samples of the feed for the analysis of particles size distribution of α-alumina in the suspension which was carried out in Zetatrac-particle size analyzer of Microtrac, model NPA15x, NPA25x.

## 3 RESULTS AND DISCUSSION

### 3.1 Morphology of AC Membranes

The morphology and pore distribution of the AC composite membranes coda and cod CB were shown in Figure 2, they demonstrate AC particle size in the range of microns, well impregnated with epoxy resin but they were randomly distributed which create voids between them. It can be seen from Figure 2a of membrane cod A that, the particles were flat and have 2-dimensional structure (almost rectangular); on the other hand, the particles of membranes cod CB (Figure 2b), have complex 3-dimensional shape.



(a) (b)

**Figure 2.** Activated carbon distribution in the composite membranes: (a) cod A, (b) cod CB.

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### 3.2 Physical Properties

Physical properties of the AC composite membranes determined by Arquimedes method was summarized in Table 1, the membranes codA2 and cod CB had the lowest porosity but not significant variations from the other membranes. The apparent densities were comparable for the membranes except that of cod A which has the lowest density and highest porosity.

**Table 1.** Physical properties of the composite membranes

| Membrane | Apparent specific mass (g/cm$^3$) | Apparent specific mass of solid part (g/cm$^3$) | Apparent Porosity (%) |
|---|---|---|---|
| Cod A | 0.613 | 1.359 | 54.897 |
| Cod A2 | 0.723 | 1.323 | 45.386 |
| Cod B | 0.685 | 1.399 | 50.325 |
| Cod CB | 0.677 | 1.308 | 48.202 |

The physical properties that were determined from mercury porosimetry were shown in Figure 3 to 6. The tests were performed first, by saturation of the superficial macropores of the membranes by mercury till a pressure of 1,500 psi, secondly, by the measurements of porosity in pressures from 1,500 psi to 33,000 psi.

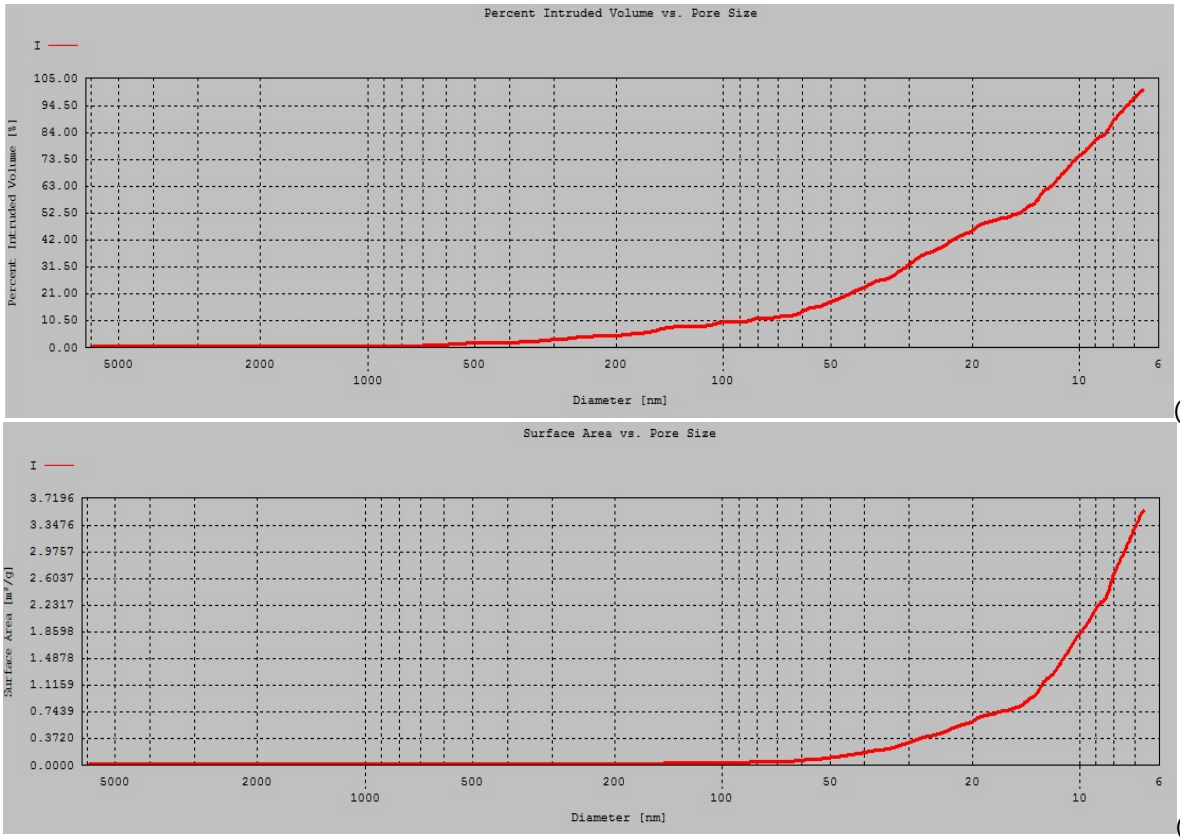


**Figure 3.** Mercury porosimetry of membrane cod A (a) Percent intruded volume vs. pore size; (b) surface area vs. pore size.

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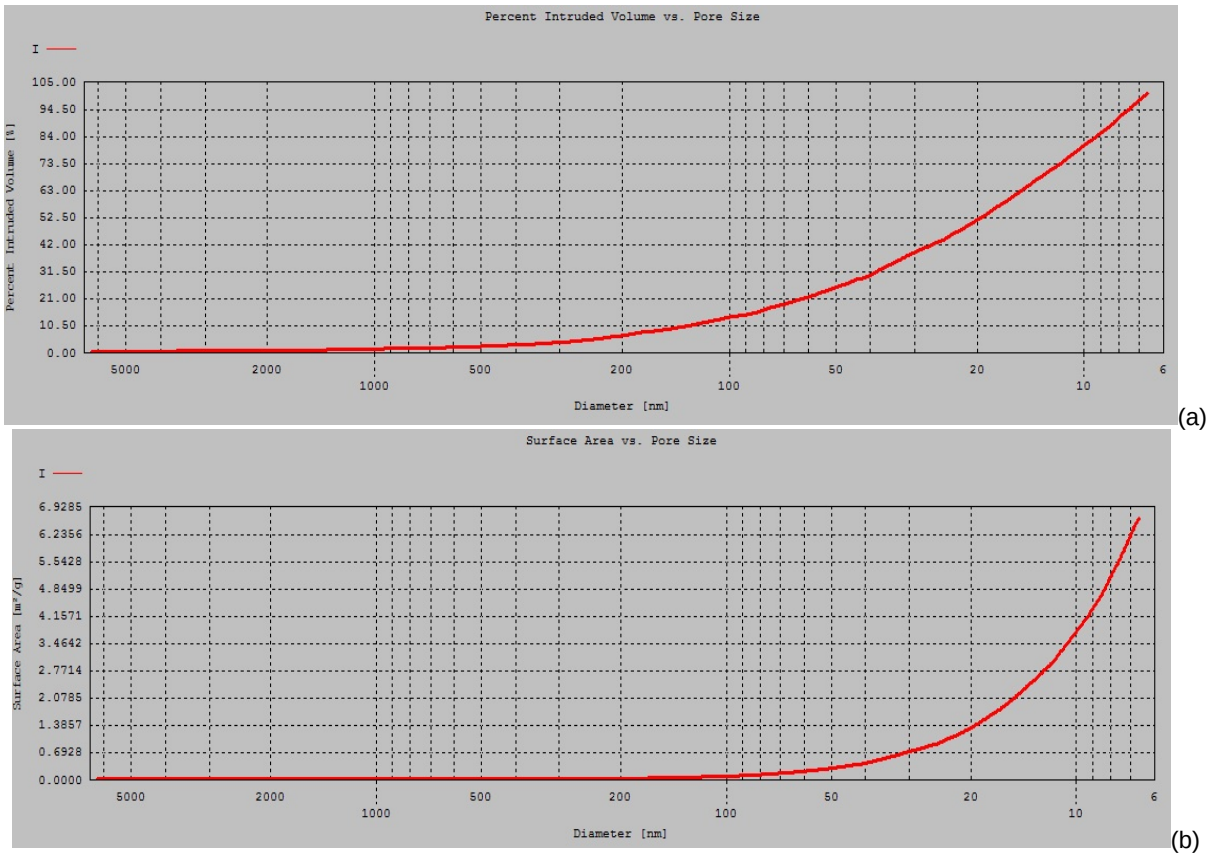


**Figure 4.** Mercury porosimetry of membrane cod A2 where, (a) Percent intruded volume vs. pore size; (b) surface area vs. pore size.

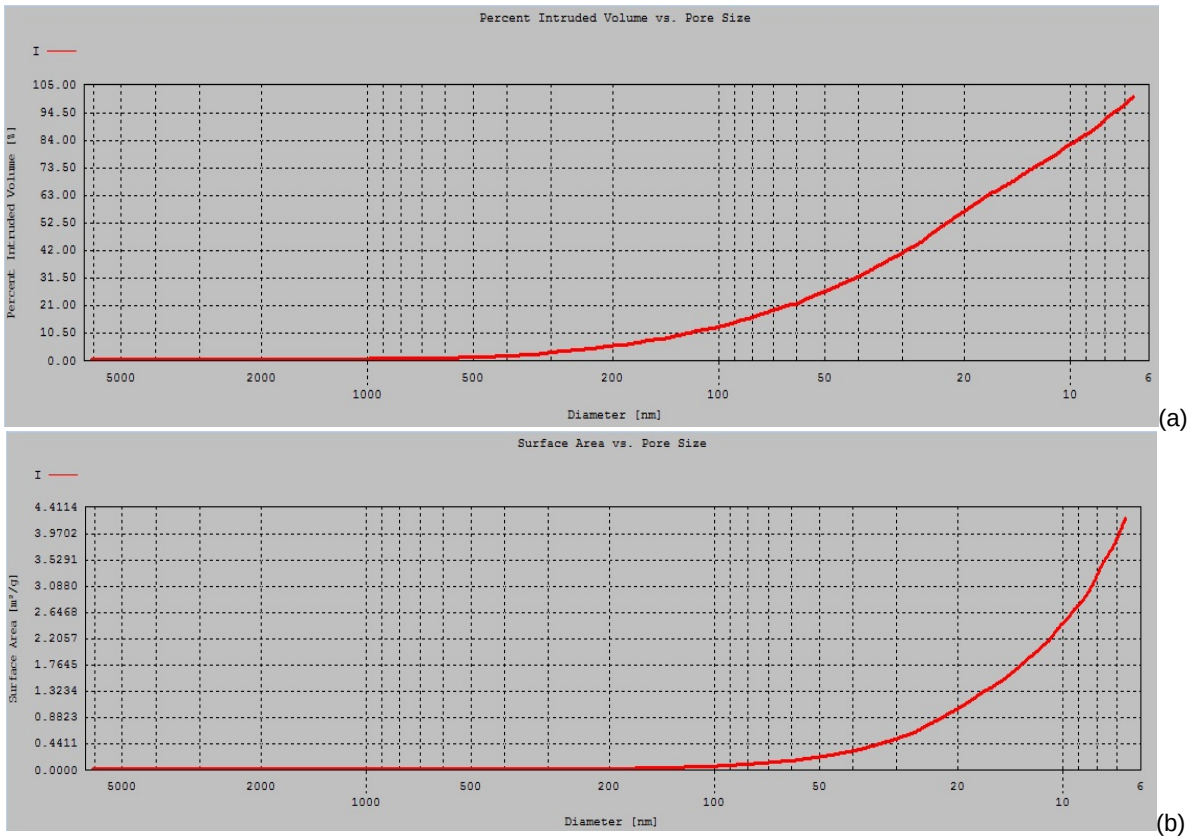


**Figure 5.** Mercury porosimetry of membrane cod B where, (a) Percent intruded volume vs. pore size; (b) surface area vs. pore size.

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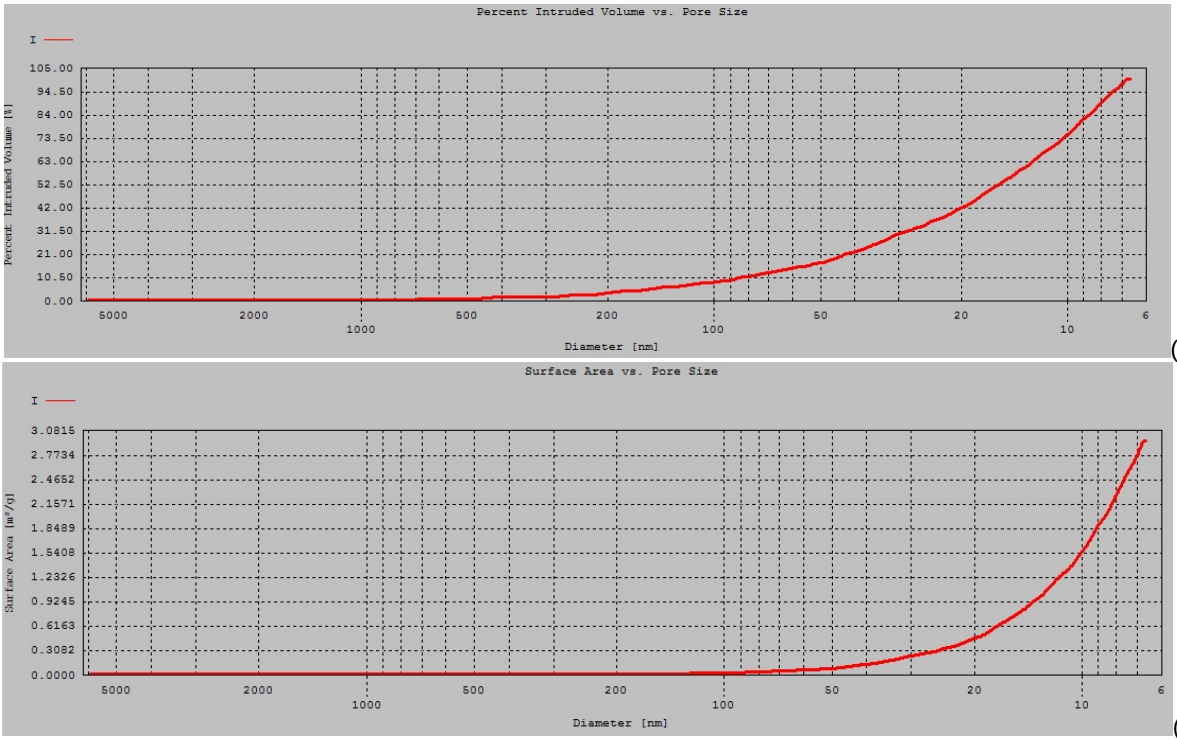


**Figure 6.** Mercury porosimetry of membrane cod CB where, (a) Percent intruded volume vs. pore size; (b) surface area vs. pore size.

The porosity of materials is defined according to the pore diameter as follows: microporous materials: 0.2 nm–2 nm, mesoporous materials: 2 nm–50 nm and macroporous materials: >50 nm. The measurements of mercury porosimetry in Figures 2 to 6 were performed as: the percentage intruded volume of mercury as a function of pore diameter; and surface area versus pore diameter, they were summarized in Table 2; columns 2 and 3 shows that the volume intrusion of mercury and surface area were situated in the range of meso and macropores. On the other hand, column 3 shows no change in the volume intruded in the mesopores range between the four membranes.

**Table 2.** The start of mercury transport in membranes as a function of pore diameter and percentage volume intruded in mesopores (<50 nm)

| Membrane | Pore size range (meso-macropores) of mercury volume intrusion variation | Pore size range (meso-macropores) of surface area variation | % volume intruded at pore size <50 nm (mesopores) |
|---|---|---|---|
| cod A | <700 nm | <100 nm | 85% |
| codA2 | <500 | <100 nm | 75% |
| cod B | <600 | <200 nm | 75% |
| cod CB | <500 | <200 nm | 85% |

## 3.3 Selectivity Tests

### 3.3.1 Test on contaminated water

In microbiology, Colony-Forming Unit (CFU) is used to determine the number of viable bacterial cells in a sample per mL. Hence, it tells the degree of contamination in samples of water, vegetables, soil or fruits.[10] The selectivity (removal efficiencies) of bacteria in the ultrafiltration process is shown in Table 3, an almost complete

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removal of bacteria (high selectivity >99%) was observed in all of the three membranes, regardless of the type of membrane, operating protocols or hydraulic conditions applied.

**Table 3.** Selectivity and quantification of the cultivated heterotrophic aerobics bacteria on the filtered water relative to as received polluted water containing 660 x 102 CFU

| Membrane | Bacteria permeated (CFU/ml) | Selectivity (%) |
|---|---|---|
| Cod A | -- | -- |
| CodA2 | $4.3x10^2$ | 99.35 |
| Cod B | $2.3x10^2$ | 99.65 |
| Cod CB | $0.3x10^2$ | 99.86 |

In membrane cod CB, being the one with the best performance (less permeability of bacteria), the bacteria removal efficiency goes up to over 99.86%. Previous studies[11] demonstrated that powdered and granular activated carbon can efficiently remove micropollutants from natural water sources used for drinking water. To date, the removal of micropollutants from wastewaters by activated carbon has not been extensively studied; Snyder et al.[12] found that a water reuse facility using granular activated carbon without regular replacement/regeneration provided little removal.

### 3.3.2 Membranes filtration measurements

The filtration capability of the AC composite membranes was tested at different applied pressures (the driving force of separation) using the suspension of 1% sub-micrometric α-alumina in water. The flow time varied from 30 s to 2 h, the results were shown in Figure 7.

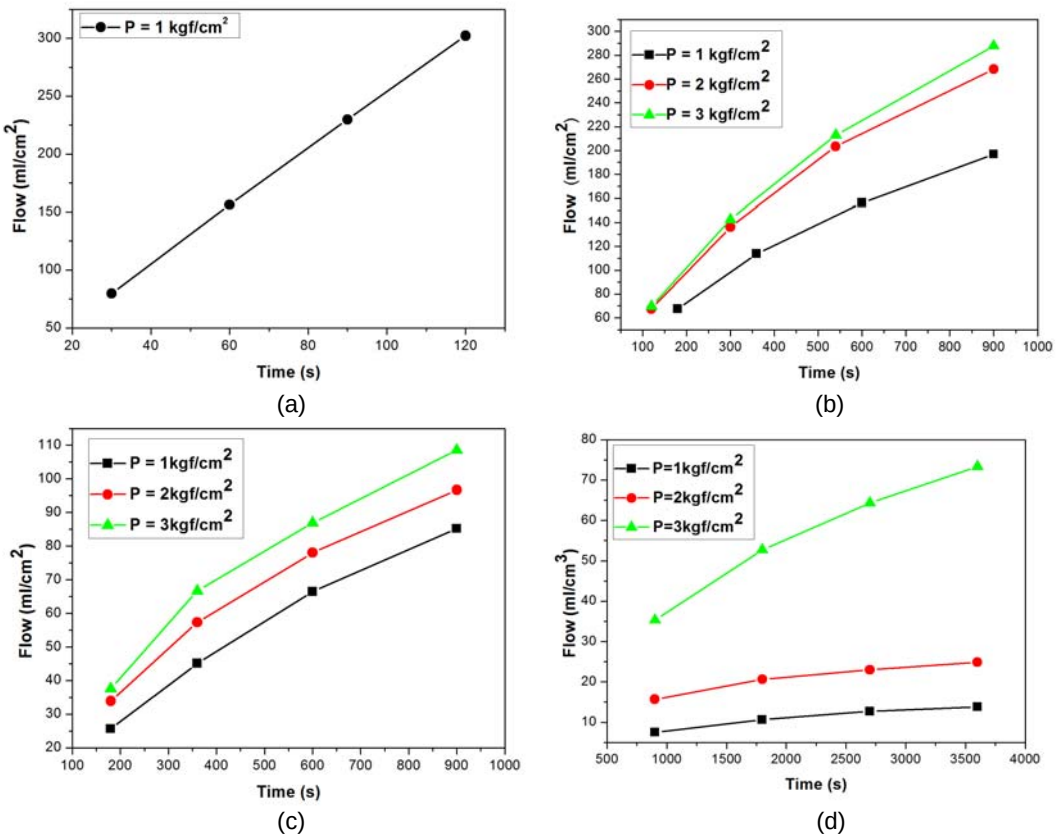


**Figure 7.** Plot of the flow versus time for the membranes: (a) cod A; (b) cod A2; (c) cod B; (d) cod CB.

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It can be noticed that at pressure p = 1 kgf/cm$^2$, membrane cod A is the one which has the highest values of flow in very short time, that is why its flow at higher pressure were not included in the graph due to the very high incomparable values at higher period of time. Membranes cod A2 demonstrate the same tendency of cod A, but for higher times with a little variation with pressure applied.
For longer periods of time, the flow of membrane cod B is very low compared to the previous ones, and almost comparable to membrane CB in its performance at the highest pressure. However, membrane CB shows the lowest permeation at the all applied pressure, this classified it as the membrane with the best selectivity which confirmed the results of bacteria separation from the polluted water mentioned above.
Taking into account the results of mercury porosimetry measurements and the porosity calculated in Table 2 were the membranes shows almost the same comportment, the only factor that contribute to the high difference in the selectivity and liquid permeation through membranes cod A and cod CB is their morphology revealed by SEM (Figure 2a), membrane coda showed flat AC particles which facilitated the permeation of the liquid through the pores and therefore the membrane has high flow rates but lower selectivity compared with membrane cod CB which has 3-dimensional morphology (Figure 2b) which made the flow of liquids more difficult through he pores contributing to better selectivity and lower flow.

### 3.3.3 Permeated particle size

A graph of mean volume diameter of the volume distribution represents the “average particle size”. The mean number diameter is the mean diameter of the number distribution, and is weighted to the small particles. The permeated solutions from flow experiments of the 1% sub-micrometric α-alumina suspension, together with raw water with ph 4, were analyzed for the average particle size distribution. From Figure 8 and Table 4, it can be inferred that the volume “50” percentile (the median diameter) of particles in the suspension that permeated through membrane cod A is 309.2 nm which is 59% lower than the corresponding median diameter of the raw water median diameter from number distribution is 254.34 which is 62% lower than the corresponding one of the raw suspension.

**Table 4.** Particle size distribution parameters of the suspension of 1% α-alumina in water permeated by membrane cod A

| % Passing | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension |
|---|---|---|---|---|
| | mean volume diameter (nm) | mean volume diameter (nm) | mean number diameter (nm) | mean number diameter (nm) |
| 10% | 603 | 225.5 | 560 | 187.2 |
| 50% | 752 | 309.2 | 677 | 254.34 |
| 70% | 835 | 360.0 | 744 | 287 |
| 90% | 996 | 499 | 858 | 346 |
| 99% | 1,360 | 776 | 1,016 | 502 |

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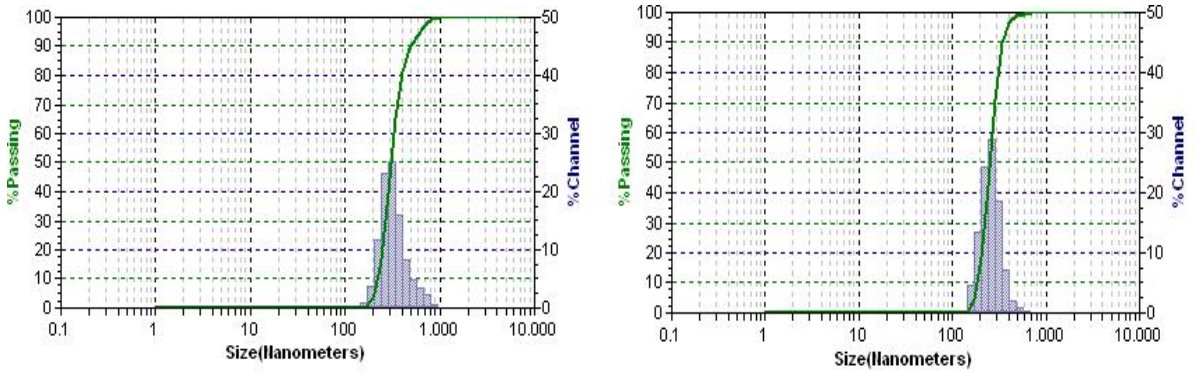


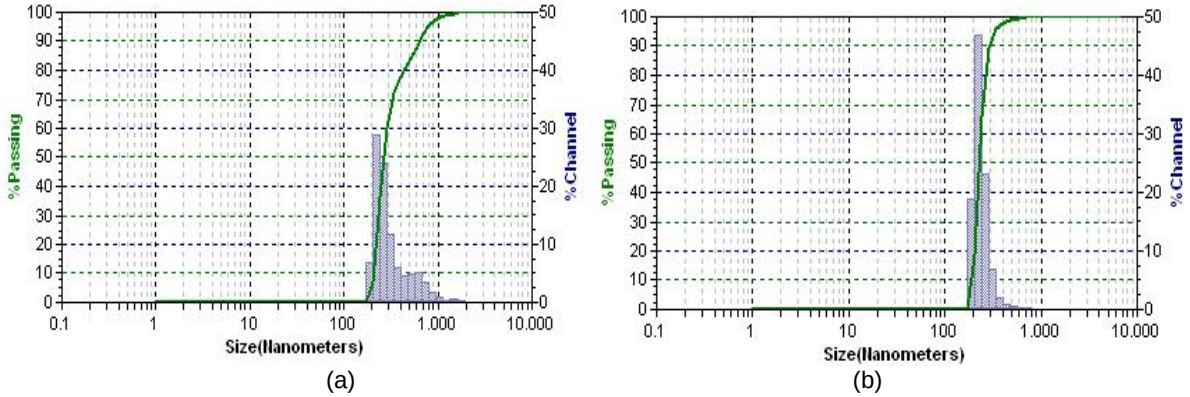

(a) (b)

**Figure 8.** Suspension of 1% α-alumina in water permeated by membrane cod A: (a) in volume; (b) in number.

The particle size distribution of particles in the suspension that permeated through membrane cod A2 are shown in Table 5 and Figure 9. The median diameter in the volume distribution is 266.3 nm which is 64.6% lower than that of the raw suspension, and the median diameter in the number distribution is 66% lower.

**Table 5.** Particle size distribution parameters of the suspension of 1% α-alumina in water permeated by membrane codA2

| % Passing | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension |
|---|---|---|---|---|
| | mean volume diameter (nm) | mean volume diameter (nm) | mean number diameter (nm) | mean number diameter (nm) |
| 10% | 603 | 209.2 | 560 | 193.7 |
| 50% | 752 | 266.3 | 677 | 229.1 |
| 70% | 835 | 331.0 | 744 | 245.4 |
| 90% | 996 | 631.0 | 858 | 293.5 |
| 99% | 1,360 | 1,346.0 | 1,016 | 509.0 |




(a) (b)

**Figure 9.** Suspension of 1% α-alumina in water permeated by membrane cod A2: (a) in volume; (b) in number.

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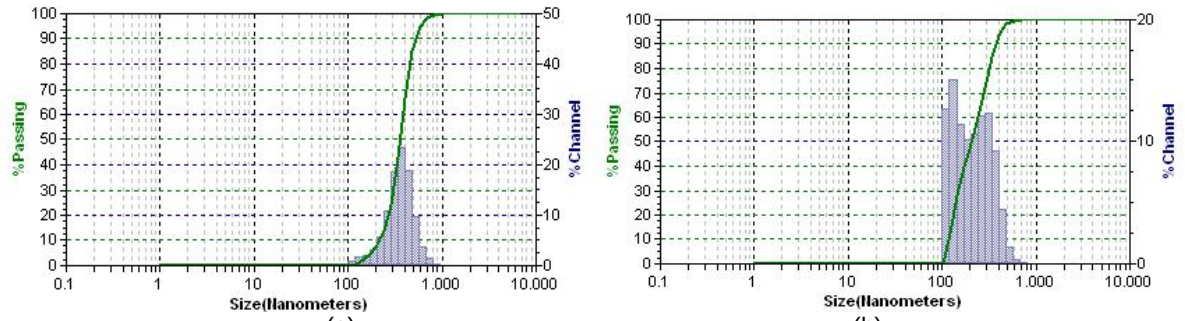
**Table 6.** Particle size distribution parameters of the suspension of 1% α-alumina in water permeated by membrane cod B

| % Passing | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension |
|---|---|---|---|---|
| | mean volume diameter (nm) | mean volume diameter (nm) | mean number diameter (nm) | mean number diameter (nm) |
| 10% | 603 | 80.6 | 560 | 70.9 |
| 50% | 752 | 109.0 | 677 | 93.5 |
| 70% | 835 | 120.8 | 744 | 106.4 |
| 90% | 996 | 137.4 | 858 | 123.8 |
| 99% | 1,360 | 158.0 | 1,016 | 146.5 |

The particle size distribution of particles in the suspension that permeated through membrane cod B Table 6 and Figure 10, the median diameter in the volume distribution is 85.5% lower than the corresponding on of the raw suspension, the median diameter of the number distribution is 86% lower.

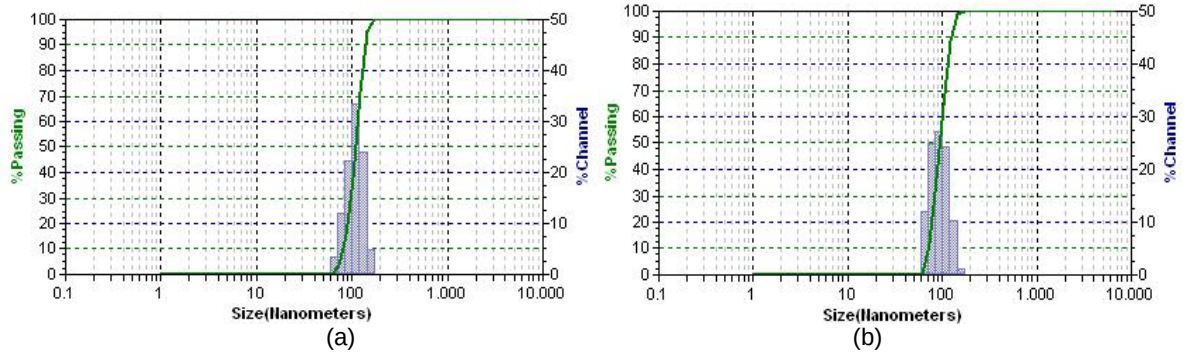



(a) (b)

**Figure 10.** Water permeated by membrane cod B: (a) in volume; (b) in number.

**Table 7.** Particle size distribution parameters of the suspension of 1% α-alumina in water permeated by membrane cod CB

| % Passing | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension | Raw water with ph 4 | Permeated α-$Al_2O_3$ Suspension |
|---|---|---|---|---|
| | mean volume diameter (nm) | mean volume diameter (nm) | mean number diameter (nm) | mean number diameter (nm) |
| 10% | 603 | 221.1 | 560 | 118.0 |
| 50% | 752 | 363.0 | 677 | 206.5 |
| 70% | 835 | 422 | 744 | 280.6 |
| 90% | 996 | 523 | 858 | 377.0 |
| 99% | 1,360 | 736 | 1,016 | 520.0 |




(a) (b)

**Figure 11.** Water permeated by membrane cod CB: (a) in volume; (b) in number.

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The particle size distribution of particles in the suspension that permeated through membrane cod CB Table 7 and Figure 12, the median diameter in the volume distribution is 52% lower than the corresponding on of the raw suspension, the median diameter of the number distribution is 62% lower. Analyzing the 50 percentile values of the diameter of the particles permeated through all membrane, it is clear that particles of permeated suspension have median diameter between 100 nm to 300 nm while the particles of the raw suspension have median diameter between 600 nm-750 nm in both type of distribution, this shows the tremendous cut off of the membranes, taking into a count that the particles of the suspensions are nanometrics but they formed agglomerates in the rage of 100 nm to microns even after dispersion with ultra sound.

## 4 CONCLUSIONS

From the above study, it can be concluded that the AC composite membrane have:

- high selectivity for bacteria and sub micrometric particles;
- advantageous in high filtration flux, long filtration time;
- excellent solid–liquid separation capacity; and
- morphology that plays an important rule in the selectivity and permeability properties of the membranes.

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