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Study of the efficacy of the membranes produced on a laboratory scale for filtration: a proposal to improve the water quality of the hydrographic sub-basin of Ribeirão das Pedras, Diamantina, MG (Brazil)

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Abstract— *Population expansion and technological development generate an increase in the use of natural resources, especially water resources, and, as a consequence, the amount of waste and chemical residues dumped into the effluents increases. With the end of the garimpo in the town of Diamantina, the spring that cuts through the city has notoriously had its flow reduced and its slopes visibly diminished and devastated by erosion, which reminds us of the need to install recovery and preservation programs. Among the pollutants, we can highlight the heavy metals, which cause great concern because of their toxicity. The present work sought to use the membrane filtration process. For this purpose, membranes were prepared using the chitosan biopolymer. The membranes were tested for the capacity to absorb metal ions. Water samples were collected from the hydrographic subbasin Ribeirão das Pedras, located in the city of Diamantina, MG, Brazil. The membranes were efficient, and the detection of possible improvements in the formulations, as well as sample collections, should be performed during other periods of the year.*
Keywords—***Adsorption, Biopolymer, Chitosan.***

I. INTRODUCTION

Areas that present "symptoms" such as the absence or reduction of vegetation cover, deposition of litter, and erosion, among others, are considered to be degraded because the lack of recovery of areas degraded by mining and panning, disorderly urbanization and lack of sanitation has caused a decrease in the amount of drinking water. A more sustainable and economically viable way to treat this resource must be sought (PEREIRA, 2004) [1].

The Ribeirão das Pedras sub-basin is particularly important because it is the source of water for the city of Diamantina (APA Pau de Fruta - COPASA, in the upper part of this sub-basin). After feeding the water catchment dam, the main course crosses two country clubs and the drainage network in the middle course crosses the entire length of the Biribiri State Park. It then flows into the Pinheiro River near the homonymous locality. However, some of the sources are in the vicinity of the headquarters of the districts of Sopa and Guinda, which discharge their sewage and other wastes into waterways of the system, in addition to collecting residues from areas degraded by diamond mining (NEVES; HORN; FRAGA, 2008) [2].

The impact of human activity is a matter of concern, and it is currently a controversial subject. It is no longer possible to implement a project or discuss any plan without considering the impact on the environment. In the case of a region degraded by mining activities, work to

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maintain the natural form or return to these characteristics is important. Thus, projects aimed at the recovery of these áreas with the expansion of environmental technologies aimed at the recovery of degraded áreas are possible. There are a number of methods for the treatment and recovery of degraded areas, especially for river basins (CHU, 2002) [3] .

Among the technologies with a possibility for use are precipitation, reduction, stabilization/solidification, membrane filtration, ion exchange and adsorption. Some are effective, but not financially viable (CHU, 2002) [3]. In this context, Förstner & Wittmann (1979) [4] and Salomons & Förstner (1984) [5] were studied concentrations of heavy metals and their amendments by processes of precipitation, complexation, biological assimilation and adsorption, observed that these contaminants accumulate in sediments over time. In this way, sediments are an important reservoir of metal concentration in the evaluation and diagnosis of environmental quality in a basin (TRINDADE *et al.*, 2018) [6]. Among these methods, adsorption is one of the most efficient in the removal of heavy metals (CHU, 2002) [3]. And, the membrane filtration process stands out because it is a simple and accessible process, which is based on the retention of metal ions on its surface (RIVAS *et al.*, 2011) [7].

Compared with traditional separation technologies, membrane technology has the advantages of high separation efficiency, low energy consumption, and simple operation (SHI. YUAN; CAO, 2001) [8] .
Membrane separation technology, wich uses natural, artificial and selective membranes, can isolate, grade, and enrich gas or water by means of two-component or multicomponent systems ZHENG, 1999 ([9] cited by WANG *et al.*, 2012 [10]).Thus, several low-cost adsorbents have been developed to evaluate the capacity for removal of these heavy metal ions (BAJLEY *et al.*, 1999) [11], as well as membrane filtration techniques utilizing natural polymers.

Among the natural polymers that can be used in the membrane filtration process to assist in the water and effluent treatment process, chitosan, a natural polymer obtained from the deacetylation of chitin found in insect carapaces, can be included. Crustaceans are easily obtained in residues from the fishing industry. In addition to having several applications, it is characterized by its efficiency in the removal of heavy metals with the advantage of being low cost, non-toxic, biocompatible and biodegradable (DALLAN, 2005) [12]. Therefore, chitosan has been widely investigated for the removal and recovery of metal ions from industrial effluents. This material possesses amino and hydroxyl groups, which are quite reactive and make it a polymer of great industrial interest. Studies on the adsorption of heavy metals by chitosan, mainly for the Cd, Cr, Cu, Ni, Pb and Hg ions, can be found in the literature (NGAH; ENDUD; MAYANAR, 2002) [13] .

The biopolymers, such as chitin (KURITA, T. SANNAN, Y. IWAKURA, 1979[14]; BARAN; BICAK; BAYSAL, 2007 [15], starch (ZHANG; CHEN, 2002 [16]) and chitosan (NGAH; ENDUD; MAYANAR, 2002[13] ; VIEIRA; BEPPU, 2003[17]) are among these low-cost adsorbents that are especially important because of their chemical structures, physicochemical characteristics, chemical stability, high reactivity and selectivity for metal ions. Chitosan also has the property of moldability in various forms (films, spheres, microspheres, membranes, etc.), and it has different surface area-to-mass ratios that maximize the adsorption capacity and minimize the effects of loss of charge and clogging of the bed in adsorption columns. The literature describes several studies on the removal of heavy metals, such as copper, lead, cadmium, zinc, mercury, among others, in monocomponent systems using chitosan in its natural and crosslinked (VIEIRA; BEPPU, 2003) [17].

The objective of this study was to determine the efficiency of chitosan-based membranes for the removal of possible contaminants, heavy metals, present in water samples collected in the Ribeirão das Pedras hydrographic subbasin, Diamantina, MG, Brazil. The samples were subjected to measurements of pH, conductivity, turbidity, hardness, chloride, COD and atomic absorption analyses. Subsequently, vacuum filtration tests were performed using the four different membrane formulations produced on a laboratory scale. Tests were performed before and after filtration to evaluate the efficiency of chitosan membranes for the removal of metallic ions. Thus, the four chitosan-based membrane formulations were characterized for filtration capacity by employing the SEM and atomic absorption analyses.

II. MATERIAL AND MÉTHODS

2.1 Material

During the preparation of the membranes, the following materials were used: chitosan (Sigma), cellulose acetate (Sigma), glacial acetic acid (Synth), succinic acid and NaOH (Synth).

2.2 Methods

Water samples were collected from the subbasin of Ribeirão das Pedras, Diamantina, MG, Brazil for

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thefiltration tests. For this purpose, characterization tests were performed on the water samples used in the filtration and on the membranes produced on a laboratory scale.

2.2.1 Characterization of the samples collected in the subbasin of Ribeirão das Pedras

The samples collected at points 1 to 7 were submitted to physicochemical analysis: pH, conductivity, turbidity, hardness, chloride, COD and atomic absorption. All of the analyses were performed in triplicate.

pH analysis - The hydrogen ion potential (pH) was measured using the mPA 210 pHmeter.

Conductivity analysis - The conductivity was measured by the Conductivity Meter (Lutron brand Cd-4303), and the results were expressed in µS/cm.

Turbidity analysis - Turbidity was measured with the Multiprocessor Turbidimeter (Hanna Instruments -HI 9370), and the results were expressed in UNT.

Hardness analysis - The hardness analysis was determined by triplicate, and the results were expressed in mg/L.

Chloride analysis – The chloride concentration was determined by triplicate, and the results were expressed in mg/L.

COD Analysis - The Chemical Oxygen Demand (COD) was measure by triplicate, and the results were expressed in mg/L.

Atomic Absorption Analysis - Absorption analysis on a flame atomic absorption spectrometer was carried out on a Spectrum 50B model Varian® spectrometer, and the results were expressed in ppm.

2.2.2 Preparation of filter membranes

For the preliminary study of the efficiency of chitosan-based membranes, four formulations of chitosan-based membrane were prepared: MQ1, MQ2, MQA and MQE. The commercial membrane, named COM, was also used as a parameter for comparison, as is shown in Table 1.

Table 1. Formulation data, method and acronym of membranes

Formulation	Method	Acronym
Comercial	Comercial	COM
1% Chitosan + 1%Acetic acid	Dispersion	MQ1
2% Chitosan + 1% Succinic acid	Dispersion	MQ2
1% Chitosan + 1% Cellulose acetate	Dispersion	MQA
2% Chitosan + 1% Acetic acid	Stretch	MQE

The formulations were prepared by two methods: a dispersion method and a stretching method (Table 2). The dispersion method consisted of total solubilization of the chitosan biopolymer and distribution of the filmogenic solution in disposable petri dishes. The stretching method consisted of immersing the membranes in 5% NaOH (m/m) solution after the drying step and then accommodating them to manual stretching and exposing them to drying. Both methods were based on the distribution of the filmogen solution in disposable petri dishes and drying in a forced circulation chamber at 40 °C for 12 hours.

Table 2. Methods of formulation of the membranes produces

Membrane	Method
MQ1	In 100 mL of 1% aqueous acetic acid solution, 1 g of chitosan was dispersed and then kept under continuous stirring (100 rpm) for 60 minutes or until complete solubilization.
MQ2	In 100 mL of a 1% aqueous succinic acid solution, 2 g of chitosan was dispersed and then kept under continuous stirring (100 rpm) for 60 minutes or until complete solubilization.
MQA	In 100 mL of 1% acetic acid solution, 1 g of chitosan was dispersed and then kept under continuous stirring (100 rpm) for 60 minutes or until complete solubilization. Simultaneously, the mixture of 1 g of cellulose acetate in acetone, also maintained under continuous stirring (15 minutes), was prepared. Then, the two solutions were distributed in the same beaker and maintained under continuous agitation until complete solubilization.
MQE	In 100 mL of 1% acetic acid solution, 1 g of chitosan was dispersed and then kept under continuous stirring (100 rpm) for 5 hours or until complete solubilization. The solution was then distributed into Petri dishes and dried in a forced circulation oven (50 °C) for 24 hours. After this period, the membranes were immersed in 5% (m/m) NaOH.

2.2.3 Characterization of filter membranes

The MQ1, MQ2, MQA and MQE membranes were characterized by MEV micrographs and by the retention capacity for possible metal ions in the filtration operation.

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The quantity of ions retained in the membranes was determined by the atomic absorption analysis.

2.2.4 Filtration analysis

For the vacuum filtration, the membranes were cut into 4-cm diameters and subjected to filtration, first with the commercial membranes, and then with the chitosan membranes. The water samples used in the filtration stage were collected in the sub-basin of Ribeirão das Pedras, Diamantina, MG, Brazil. A 1000-mL water sample was used for each filtration test. The filtration tests were performed with the use of a bench microfiltration/ultrafiltration (MF/UF) unit manufactured by PAM Membranas Selectivas Ltda.

2.2.5 Analysis by Scanning Electron Microscopy (SEM)

The samples were cut into small squares, affixed to cylindrical stubs, metallized for 40 minutes, and then subjected to scanning electron microscopy analysis using the CamScan-3200 LV Shimadzu-Japan equipment.

III. RESULTS AND DISCUSSION

3.1. Location of sample collection

Water samples were collected at seven distinct points, of the hydrographic sub-basin of the Ribeirão das Pedras, Diamantina, designated: 1 through 7 (Fig. 1 - 8).

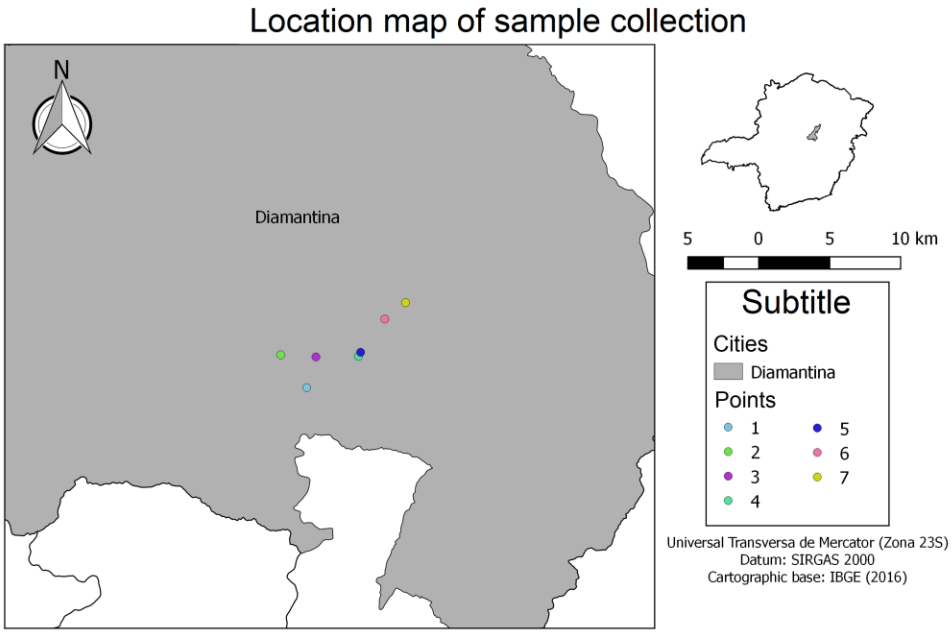

Fig. 1. Location map of sample collection

Fig. 2. Point 1 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

3.2. Visual characterization of the samples

The samples collected in the sub-basin of Ribeirão das Pedras, in Diamantina, MG, in addition to the basic characteristics of Latitude, Longitude, Depth, Temperature, Bed, Aspect, Color, and Fluxo, were also classified according to the visual characteristics, as follows:

• Point 1 - At the collection point 1, the presence of algae, insects, mud and fish was observed. It was also verified that there was a tardigrade in a bog at the point of collection.

• Point 2 - At the collection point 2, the presence of fish, tadpoles, reeds and mosses was verified. The collection point was situated between a ciliary forest in the form of isolated capons.

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Fig. 3. Point 2 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

Fig. 4. Point 3 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

Fig. 5. Point 4 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

• Point 3 - At point 3, a fillet of water was observed inside the bed with a width of 1.5 m of river bed. There was a predominance of gravel bars and the presence of vegetation.

• Point 4 - The collection point was made in the COPASA Pau de Fruta dam, a dam below the level of the dyke, where there was fine sandy material.

Fig. 6. Point 5 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

Fig. 7. Point 6 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

Fig. 8. Point 7 of collection of the water samples from Ribeirão das Pedras, Diamantina, MG, Brazil

• Point 5 - In point 5, suspended flocs were observed in the watercourse with rocky outcrops in the bed, which formed small, isolated pools on the side, in addition to the presence of tufts and grasses on the margins. It should be noted that said point is located just below the highway bridge.

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• Point 6 – The sample from point 6 was collected in the main well of the Toca waterfall, where gravel bars were detected at the outlet of the waterfall, in direct contact with quartizite and shale.

• Point 7 - Collection point 7 was located in a well of clean water, in a water course barred by a rock outcrop and a sand side bar at the exit of the well (downstream), with the presence of fish and vegetation on the banks. To facilitate the identification and analysis of the parameters, annotations were made on the samples collected for each collection point, such as: Latitude; Longitude; Depth; Temperature (T); Bed; Aspect; Color and Flow (Table 3).

3.3 Physicochemical characterization of the samples

The samples collected in the subbasin of Ribeirão das Pedras were analyzed for pH, Conductivity Turbidity, Hardness, Chlorides and COD. The pH of all the samples was slightly acidic. The conductivity of sample 6 was high, when compared to the other samples. The turbidity of sample 1 was the highest. There were no differences in the hardness, chloride and COD of the samples analyzed (Table 4).

Table 3. Characteristics of collection points

Parameter	Sample						
	1	**2**	**3**	**4**	**5**	**6**	**7**
Latitude (E)	639233	637321	639810	642806	642948	644574	646001
Longitude (N)	7979376	7981613	7981551	7981689	7981981	7984365	7985562
Depth (cm)	30 - 40	10 - 40	25-35	30 - 350	10 – 50	10 - 500	30 - 200
T (ºC)	18.9	20.2	16,5	21.3	22	19	20.1
Bed (m)	0 – 1.8	5	8	0.20x0.20	1 – 5	0.20x0.20	6x5
Aspect	Clear	Clear	Clear	Clear	Clear	Cloudy	Clear
Color	Yellow	Transparent	Cristaline	Cristaline	Cristaline	Cristaline	Cristaline
Flow	Still	Slow	Fast	Contained	Slow	Medium	Medium

Table 4. Physicochemical characterization of water samples

Parameters	Samples						
	1	**2**	**3**	**4**	**5**	**6**	**7**
pH	5.44	5.91	6.00	5.98	6.61	6.64	5.69
Condutivity (µS/cm)	4.60	2.40	15.5	1.20	21.6	17.80	3.20
Turbidity (UNT)	3.55	0.44	0.82	0.59	1.80	0.74	1.52
Hardness (mg/L)	1.23	0.50	0.50	0.40	0.60	0.63	0.70
Chloride (mg/L)	0.30	0.20	0.30	0.30	0.30	0.20	0.20
COD (mg/L)	3.05	3.10	3.06	3.03	3.10	3.37	3.00

There were no alterations in the pH values measured in the water samples collected in Ribeirão das Pedras, Diamantina, MG. Thus, the values were in accordance with the Resolution 357/05 of CONAMA (CONAMA, 2005) [18], which establishes the limits between 5 and 6 for pH. Figueiredo *et al*. (2014) [19] in their research conducted in the Rio Doce basin, detected mean values of pH were 6.9, 6.6 and 7.2 in the observed years, respectively, which shows a medium close to neutrality. However, several biological and physico-chemical processes can influence the observed pH range.

The higher the dilution of the solutes, the lower the electrical conductivity Poch (1999) [1] in turn, relates the changes of salinity to the lithological nature of the land, on which, or within which, the waters percolate. In addition, it also takes into account the influence of domestic and industrial effluents. In the present study, points 3, 5 and 6 presented higher values of conductivity, (15.5, 21.6 e 17.8 µS/cm), respectively. These values are concomitant with the highest pH values (6.0, 6.61 and 6.64), respectively, indicating the non-dilution of the solutes.The conductivity is dependent on the concentrations of ions present in the aquatic environment, which in turn is influenced by T and pH [20] (WETZEL, 2001). This fact may have been highlighted in section 4, with a lower conductivity reading (1.24 µS / cm).

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Under natural conditions, hardness is a chemical characteristic of water from dissolution of calcareous rocks or other minerals containing calcium and magnesium (CORDEIRO *et al*., 2012) [21]. In the present work, even if samples were collected at points that make up a hydrographic basin, composed of rocky regions, only point 1 (1.23 mg / L), being the highest hardness value found among the other collection points (Table 4). The hardness values observed were within the allowed values, according to the Article 16 of Ordinance 518/04 (BRASIL, 2004 [22]) of the Ministry of Health, which establishes a maximum of 500 mg/L of hardness for drinking water.

The parameters of color and turbidity are highly correlated in environments where there are high concentrations of iron [23] (MESQUISA *et al*., 2016). This fact can justify the behavior of collection point 1, with turbidity values 3.55 UNT (Table 4), yellow color and still flow (Table 3).

In this job, the turbidity of all the collection points were within the legal limits because CONAMA 357/05 establishes limits that cannot exceed 40 UNT (CONAMA, 2005) [18] and the Ordinance 518/04 of the Ministry of Health establishes a maximum of 5 UNT for water potability (BRASIL, 2004 [22]).

According to Article 16 of Ordinance 518/04 of the Ministry of Health and Article 14 of Resolution nº 357/05, the maximum concentration of chlorides present in the water is 250 mg/L. Chloride is a major ionic constituent of groundwater and its presence is directly related to point sources and diffuse anthropogenic contamination and the geological and geochemical characteristics of the area aquifers (BAHIA *et al*., 2011) [24]. Thus, all the samples were within the legal limit (CONAMA, 2005 [17]; BRASIL, 2004 [22]). Thus, the samples collected at points 1, 2, 3, 4, 5, 6 and 7 did not present discrepant values. Therefore, the results indicate that the samples can be classified as waters of unpolluted rivers and do not violate resolutions of CONAMA or the Ministry of Health.

3.4 Scanning Electron Microscopy (SEM)

SEM analyzes were performed on all the membranes (COM, MQ1, MQ2, MQA and MQE) at 500X magnification. As shown in Fig. 8-11, the texture of each of the membranes produced could be visualized, and the relationship between the analyses performed in the present work regarding the filtration time to which they were submitted could be compared.

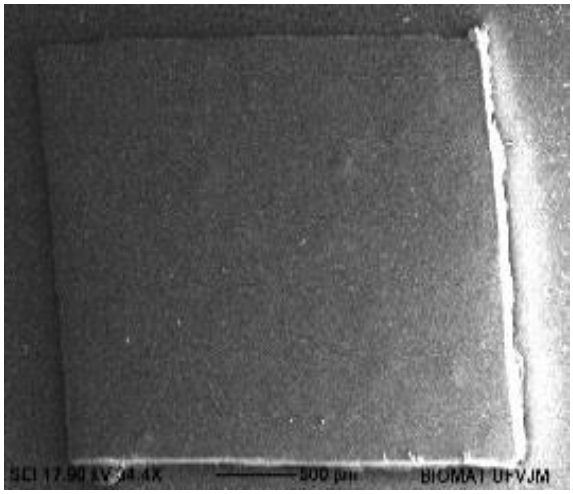

Fig. 9. MEV (plane view) of MQ1 membranes

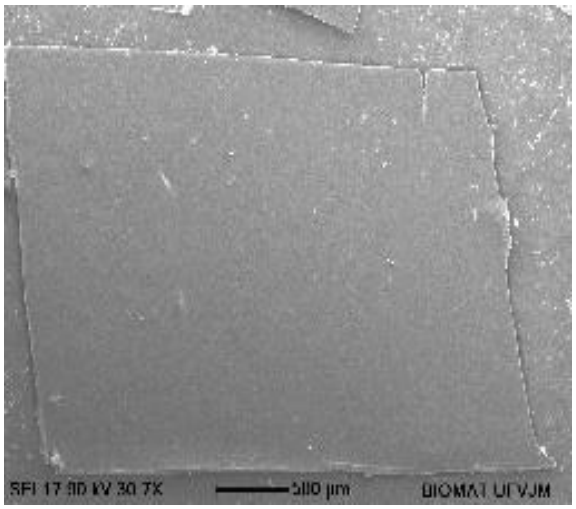

Fig. 10. MEV (plane view) of MQ2 membranes

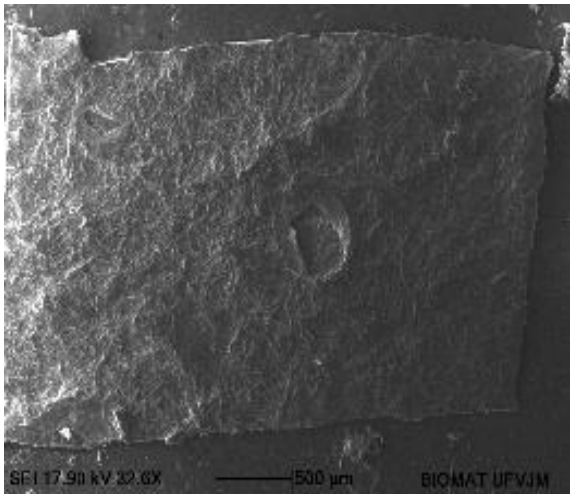

Fig.11. MEV (plane view) of MQA membranes

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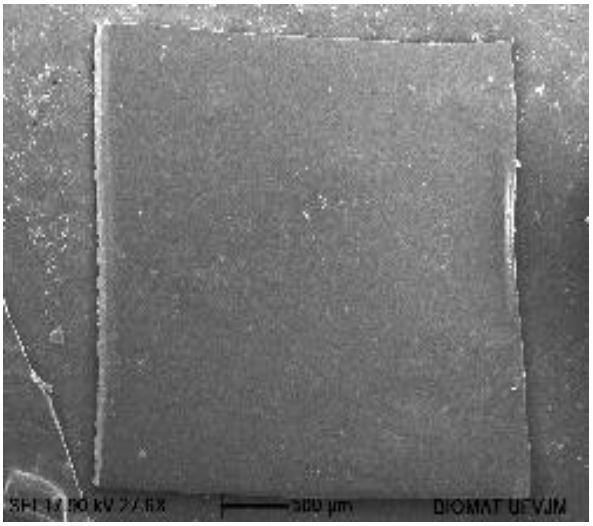

Fig. 12. MEV (plane view) of MQE membranes

As shown in Fig. 8-11, a basically uniform structure could be observed for the samples MQ1, MQ2 and MQE; however, the structure of the MQE sample was more uniform than those of samples MQ1 and MQ2, in which the formation of small reliefs and cavities could be seen, possibly related to inadequatr solubilization of the filmogenic solution or deficiencies in the drying step. The MQA sample had a distorted surface, with cavities and reliefs visible to the naked eye. However, it was the sample that presented the best distribution and interweaving of the fibers, and it was the sample most visually similar to the commercial sample (Fig. 12).

3.5 Atomic absorption analysis

The atomic absorption analysis was performed on the samples collected in the sub-basin of Ribeirão das Pedras, Diamantina, MG. The samples 1-7 were analyzed before and after the filtration step. As shown in Figure 14, a higher concentrations of copper and manganese ions, respectively, were found in samples 1 and 6. The presence of iron was detected in all the samples (1-7).

changes in the samples filtered with the other membranes; there were small changes in the concentration of copper ions in Point 1, which had higher concentrations of this metal. The largest reduction (62.5%) was observed for the MQE membrane, whereas a reduction of 37.5% was found for the other membranes.

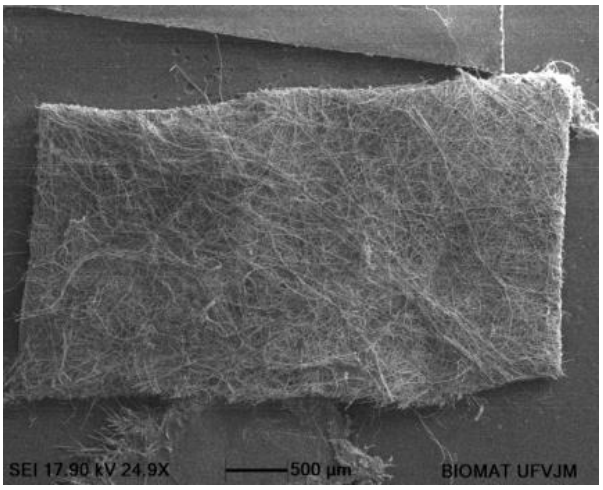

Fig.13. SEM (plane view) of COM membranes

As can be seen in Figure 15 and 16, no changes in the concentrations of heavy metals were found in the samples filtered with the COM membrane. There were. The greatest decrease in concentration of the iron ion, mean 31.5%, was obtained with the MQE membrane, where as smaller average reductions were obtained with the other membranes (MQ1 - 13.2%, MQ2 - 19.1% and MQA - 14.9%). The role of chitosan in increasing the adsorption capacity of metals is emphasized because the membranes with the best results were those made with 2% chitosan, to the detriment of those with only 1% chitosan.

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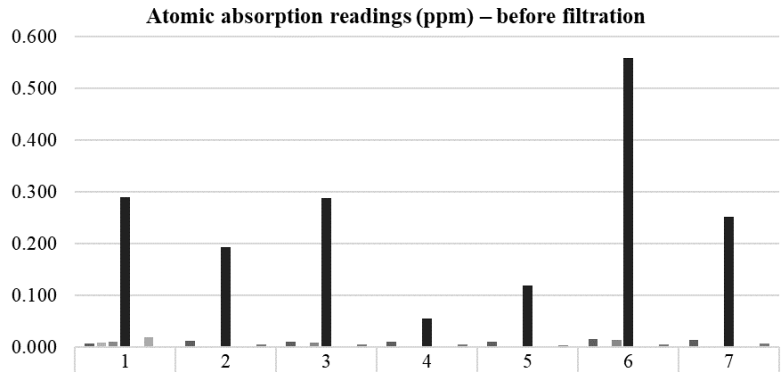
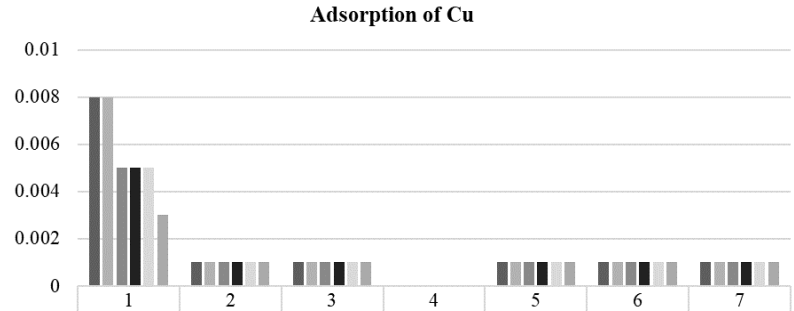


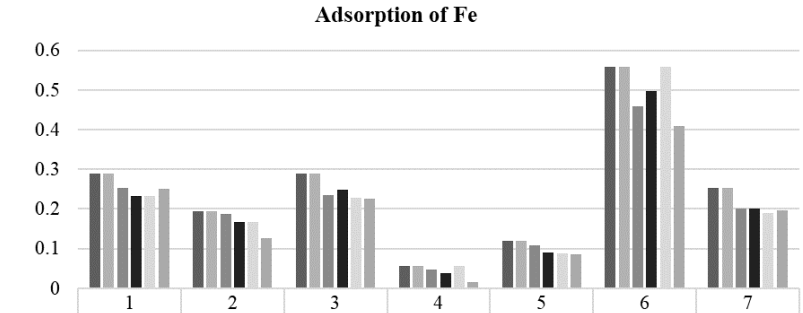
	1	2	3	4	5	6	7
Zn	0.007	0.012	0.010	0.010	0.010	0.016	0.014
Cu	0.008	0.001	0.001	0.000	0.001	0.001	0.001
Mn	0.010	0.002	0.009	0.001	0.001	0.014	0.001
Fe	0.289	0.193	0.288	0.055	0.119	0.558	0.252
Cd	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Ni	0.019	0.000	0.000	0.000	0.000	0.002	0.000
Pb	0.000	0.004	0.005	0.004	0.003	0.004	0.007

Fig.14. Results of the atomic absorption analysis of the samples collected in the subbasin of Ribeirão das Pedras



	1	2	3	4	5	6	7
Antes	0.008	0.001	0.001	0.000	0.001	0.001	0.001
COM	0.008	0.001	0.001	0.000	0.001	0.001	0.001
MQ1	0.005	0.001	0.001	0.000	0.001	0.001	0.001
MQ2	0.005	0.001	0.001	0.000	0.001	0.001	0.001
MQA	0.005	0.001	0.001	0.000	0.001	0.001	0.001
MQE	0.003	0.001	0.001	0.000	0.001	0.001	0.001

Fig.15. Adsorption of Copper



	1	2	3	4	5	6	7
Antes	0.289	0.193	0.288	0.055	0.119	0.558	0.252
COM	0.289	0.193	0.288	0.055	0.119	0.558	0.252
MQ1	0.253	0.188	0.235	0.048	0.109	0.458	0.201
MQ2	0.233	0.168	0.248	0.038	0.089	0.498	0.200
MQA	0.232	0.168	0.228	0.055	0.088	0.558	0.189
MQE	0.250	0.126	0.225	0.015	0.085	0.408	0.196

Fig.16. Adsorption of Iron

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IV. CONCLUSION

It is necessary to make collections in different periods of the year because the results obtained with the analysis of the water samples collected in mid-August were compared with the standards required by ordinance No. 518/2014 (BRASIL, 2004 [22]) and nº 2914/2011 (MS, 2011) of the Ministry of Health and resolution nº 357/2005 (CONAMA, 2005) [18] and nº 430/2011 (CONAMA, 2011) [25] of CONAMA, and all the samples were within the established standards.

The efficacy of the membranes made with the chitosan biopolymer was also confirmed because of the increase in the capacity for adsorption of metals. The membranes with the best results were those made with 2% chitosan, whereas less efficient absorption was obtained with the membranes prepared with only 1% of chitosan or with the commercial membrane that does not possess chitosan in its composition. However, new experiments are underway to increase the resistance of the membranes.

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