

*Carla da Silva¹, Cintia Barros¹, Yure Queiros¹, Luiz Marques¹, Ana Maria Louvise²
and Elizabete Lucas¹*

ADSORPTION OF BTX AND PAHs BY POLYMERIC RESINS PACKED IN A FIXED BED: SEMI-INDUSTRIAL EVALUATION

¹*Federal University of Rio de Janeiro (UFRJ), Institute of Macromolecules(IMA), Laboratory of
Macromolecules and Colloids for Petroleum Industry (LMCP), Av. Horacio Macedo,
2030, Ilha do Fundao, 21941598, Rio de Janeiro, Brazil; michelefrota@ima.ufrj.br*

²*PETROBRAS, Av. Horacio Macedo, 950, Ilha do Fundao, 21941915, Rio de Janeiro, Brazil*

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Abstract. Polymeric resins were synthesized by suspension polymerization on a pilot scale and packed in an acrylic column for use in a semi-industrial elution system, to study their behavior as adsorbents of oily water. The performance in removing benzene, toluene and xylene (BTX) was greater than 95.9 %, and the efficiency in retaining polyaromatic hydrocarbons (PAH) remained near 96.3 %.

Keywords: produced water, petroleum, BTX, PAH, polymeric resins, adsorption.

1. Introduction

The treatment of the oily water from petroleum production poses a growing challenge because of the need to protect the environment, in conformity with increasingly strict environmental legislation [1-3]. In Brazil, the rules of oil discharges are established by the National Council of Environment [4]. The produced water contains many toxic compounds, which relative quantities vary depending on the oil well [5], and this water can be disposed or re-injected [6, 7]. For disposal, compounds such as benzene, toluene, ethylbenzene and xylene (known as BTEX) and the polyaromatic hydrocarbons group (PAHs) are of particular concern due to their relative solubility in water [8]. These compounds are extremely toxic and potentially carcinogenic, and as such being can cause significant damage to marine flora, fauna and to human. Their average concentration in produced water is around 40 parts per million (ppm), but this can range as high as 1000 ppm, depending on the production well [9, 10].

Polymeric resins have been developed for treating oily water and have been an effective alternative for removal of various types of compounds [11]. In a previous study by our research group, we assessed the purification level of synthetic oily water samples achieved by treatment in fixed-bed columns packed with vinyl and acrylic polymeric resins, in a bench-scale system. The results showed that the amphiphilic structure of the resins was highly efficient in retaining components with different degrees of polarity. In this study we evaluated the saturation, regeneration and mechanical integrity of the polymeric resins, on a bench scale, with satisfactory results [12].

The objective of this work was to synthesize the same resins on a pilot scale and assess their use to treat oily water on a semi-industrial scale, with focus on removal not only of petroleum, but also of mono- and polyaromatic components.

2. Experimental

2.1. Synthesis of the Resins

2.1.1. Reagents

Divinylbenzene – DVB (Nitriflex S.A., Brazil) and methyl methacrylate – MMA (Metacril S.A., Brazil) were used as received, with benzoyl peroxide (BPO) employed as an initiator. The solvents heptane (HEP) and toluene (TOL), with P.A. purity, were acquired from VETEC Quimica Fina (Brazil). Animal gelatin and NaCl were acquired from Theoberg Importacoes e Exportacoes Ltda. (Brazil) and VETEC, respectively.

2.1.2. Preparation of aqueous and organic phases

Before carrying out the polymerization, two phases were separately prepared: an aqueous phase (AP) and an organic phase (OP). The AP was prepared by dissolving NaCl and animal gelatin in water, at the temperature of 313 K, in a mixing vessel with mechanical stirring and heating, connected with a reaction vessel. Concentrations of 2 % p/v of NaCl and 1 % p/v of gelatin were used for both the reaction with DVB and that with MMA-DVB (67/33 in monomer mol). The OP was prepared by mixing the monomers and initiator (1 % BPO in relation to the monomer mass) under an inert atmosphere at room temperature, in a reaction vessel with the capacity of 100 liters, equipped with an external heating jacket and stirring. The organic phase was pre-polymerized at 323 K for 30 min under mechanical stirring at 120 rpm. After this reaction time, diluent mixtures of TOL/HEP (70:30) for the DVB reaction and of cyclohexane for the MMA/DVB reaction were added to the pre-polymerized solution in the one-to-one proportion (diluent/OP). In both cases the mixture was left under stirring to homogenize the system [13, 14].

2.1.3. Polymerization

The aqueous phase prepared previously was added to the organic phase under mechanical stirring at 120 rpm to produce a suspension. This suspension was maintained at 363 K for 24 h for the conventional suspension polymerization itself. The AP/OP ratio was kept at 3:1 (v/v) in the two reactions.

2.1.4. Purification

At the end of the reaction, the polymer beads were treated with a solution of HCl 1M and then were washed in distilled and deionized water. After this pre-treatment the beads were washed repeatedly with distilled water and with ethanol until the filtrate was totally transparent. The beads were then screened in a Telastem Model G industrial sieve apparatus, utilizing screens with mesh sizes of 25, 45, 100, 140, 200, 270 and 325. The resin beads were then washed again, this time with methanol, and dried in an oven with forced air circulation at 333 K for 48 h [14].

2.1.5. Characterization

The apparent density of the resin beads was determined by employing an adaptation of the ASTM method D1895 [15]. The specific area was determined by nitrogen adsorption data, at different relative pressures, at the temperature of liquid nitrogen, by the BET method

[16], using a Micromeritics model ASAP 2010 analyzer. The pore volume and average pore size distribution of the beads were determined by mercury porosimetry in a Micromeritics Autopore 9420 device [17]. The external morphology of the polymer beads was observed under an Olympus SZ10 optical microscope coupled to an Olympus digital camera, and scanning electron microscope JEOL, JSM-5610LV.

2.2. Preparation of Contaminated Aqueous Systems

2.2.1. Reagents

The oily produced water and crude oil came from production platforms in the Campos Basin, Brazil. Distilled and deionized water was produced in a Gehaka OS10LX reverse osmosis system. Toluene, benzene, an isomer mixture of xylene, hexane, NaCl and CaCl₂, with P.A. purity, were acquired from VETEC Quimica Fina (Brazil). Natural bentonite were acquired from Industria Bentonit Uniao Nordeste and was used as received.

2.2.2. Produced water spiked with solids

About 1200 mg of natural bentonite were added to 55 l of produced water in a vessel. The bentonite was added slowly under constant stirring to produce a mixture of approximately 20 ppm.

2.2.3. Produced water spiked with crude oil

About 35 l of produced water were placed in the vessel of the semi-industrial water treatment system assembled for this experiment (50 l capacity). Then under stirring at 7000 rpm by an Ultra-Turrax T-50 device, a quantity of crude oil at the intended concentration was added slowly during 10 min. Then 15 l of saltwater were added (55000 ppm at a proportion of 10:1 of NaCl/CaCl₂) and the stirring rotation was increased to 9000 rpm for 20 min.

2.2.4. Produced water spiked with aromatic hydrocarbons

A 10000 ppm solution of BTX (mixture of benzene, toluene and xylene) in distilled water was prepared in a volumetric flask placed in a Retsch UR1 ultrasound device for 30 min. The system was then kept under magnetic agitation for 40 min. The resulting solution was then added to water vessel of the semi-industrial system, which already contained 35 l of oily produced water, to form a system with an additional 300 ppm of BTX.

2.3 Water Treatment in the Semi-Industrial System

2.3.1. Column packing

An acrylic column (height of 80 cm and internal diameter of 2.93 cm) was used to enable better visualization of the retention of contaminants. The column initially contained DVB in the lower half and MMA/DVB in the upper half. Before packing, a screen with the mesh of the order of 200 micrometers was placed at the bottom of the column to retain the material lost during the packing and elution process. For packing, the resin was added at the top of the column using a Chromatonix dry column packer, which provides upward and downward movement to the column so that the filler settles by gravity. After packing, another mesh screen was placed at the top of the column.

The elution direction was the same as that used in packing the column. Elution was performed with deionized water at a flow of 200 ml/min to pressurize the system and assure efficient column packing. After this final step, the system was ready for the start of the tests.

2.3.2. Elution

The semi-industrial elution system is composed of a vessel to hold the contaminated water, a ½-HP pump to control the pressure of water entering the column (12 bar), a pressure differential transducer to measure water pressure in the column (made of acrylic and filled with polymeric resin), an outlet hose for collection of the treated water, and a control panel containing a pressure gauge along with the controls. Water can be eluted at a flow of up to 460 ml/min. As stated above, the elution was carried out in the same direction as the packing. Fig. 1 shows a diagram of the system, identifying each element and how they all are connected.

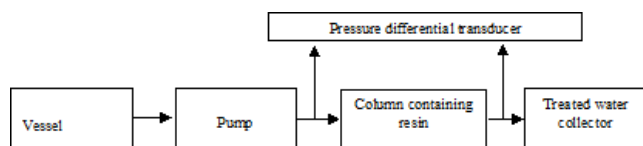


Fig. 1. Diagram of the semi-industrial water treatment system

2.3.3. Characterization of water

Aliquots of water were removed before and after elution through the column to measure the levels of solids, oil, BTX and polyaromatic hydrocarbons.

2.3.4. Analysis of total suspended solids (TSS)

First, a Millipore HA membrane of cellulose esters, with the pore diameter of 0.45 µm, was placed in a

Petri dish with a glass lid, which was placed in a forced-air oven at 333 K for 3 h, then left in a desiccator for 20 min and weighed (W_1). After this procedure, the membrane was adapted to a Millipore filtration kit, under vacuum, and a determined volume V (ml) of the sample (about 500 ml) was passed through the membrane. After that, 50 ml of toluene and 50 ml of distilled water were passed through the membrane. After drying in the forced-air oven for approximately 12 h at 333 K and being left to rest in the desiccator for 20 min, the membrane was weighed again (W_2). The TSS was calculated by Eq. 1.

$$\text{TSS (ppm)} = (m \text{ (mg)} \cdot 1000) / V \text{ (ml)} \quad (1)$$

where $m = W_2 - W_1$

2.3.5. Analysis of the total oil and grease (TOG) concentration by fluorescence spectroscopy

The aliquots for analysis were extracted with hexane in a proportion of 1:10. The organic phase was added to a quartz cuvette with four polished faces and analyzed under an excitation wavelength of 350 nm and emission wavelength of 440 nm in a Varian Cary Eclipse fluorescence spectrophotometer. The values were determined by using a calibration curve obtained previously [18].

2.3.6. Analysis of aromatic and polyaromatic hydrocarbons by gas chromatography

The concentrations of BTX in water were measured in a Shimadzu CG-17A gas chromatograph, equipped with a flame ionization detector (FID) and coupled to a DB-5 column (30 m x 0.25 mm). The most appropriate temperatures for the injector and detector were 503 and 523 K, respectively. The temperature started at 313 K for 2 min and then was raised to 393 K at a rate of 10 K/min. One µl of samples was injected in split mode (1:10), employing nitrogen as the carrier gas, at a flow of 1 ml/min [19, 20]. The levels of polyaromatic hydrocarbons (PAHs) were measured by gas chromatography in the laboratory of the Rio de Janeiro State Federation of Industries (FIRJAN).

3. Results and Discussion

Previous results obtained by bench-scale experiments [12] demonstrated the good potential of polymeric resins to retain oil and grease. Since the morphological structure and hydrophilicity degree of polymeric materials influence their performance, pilot-scale synthesis tests under controlled conditions were run to assure obtaining resins with characteristics similar to those that could be obtained and evaluated in the bench scale [12]. The polymeric materials obtained, based on

DVB and MMA/DVB, were packed in series in the column of a semi-industrial elution system, in which the material was subjected to extreme conditions of pressure increase, elution speed and oil concentration. The system ability to retain compounds of the BTX and PAH families was also assessed.

3.1. Characterization of the Polymeric Materials

Table 1 presents the characterization results of the DVB and MMA/DVB polymeric materials. As desired, the synthesis performed on a pilot scale reproduced the morphology of the resins obtained earlier on a bench scale [12]. The size range of the beads packed in the column was 45–100 mesh, the same as used previously in the bench-scale experiments.

Table 1

Characteristics of the polymeric materials

Polymer	A , m ² /g	V_p , cm ³ /g	D_p , Å	d_{ap} , g/cm ³
DVB	646	0.86	146	0.33
MMA/DVB	71	0.60	460	0.41

Notes: A is specific area; V_p is pore volume; D_p is mean pore diameter; d_{ap} is apparent density

3.1.1. Evaluation of the contaminant retention capacity

The evaluation of the contaminated water treatment system was divided into four phases, always using the same column containing polymeric resin beads. The first phase entailed monitoring the level of total suspended solids (TSS) in the produced water spiked with bentonite, with elution of 110,000 ml. During this phase, the total oil and grease (TOG) content was also monitored. In the second phase, the column performance was assessed in treating the produced water with extra petroleum added, with elution of 649,000 ml and TOG monitoring. In the third phase the system capacity to retain monoaromatic hydrocarbon contaminants was evaluated using produced water spiked with petroleum and BTX (benzene, toluene and xylene), with elution of a further 284,000 ml of contaminated water and measurement of TOG and BTX levels. Finally, at the fourth phase, the column capacity was evaluated to retain polyaromatic hydrocarbons, with elution of 288,000 ml of contaminated water. Generally, 1,331,000 ml of contaminated water were eluted through the column, which corresponds to approximately 2,500 times of the column bed volume.

3.1.2. First elution phase

At this stage of the study, the influence of the suspended solids in water contaminated with only a small

crude oil concentration was investigated. For this purpose, oily produced water (TOG \cong 30 ppm) was mixed with approximately 20 ppm of bentonite. The volume eluted was 110,000 ml (about 204 times the bed volume), at a flow of 65 ml/min, and the TOG and TSS were measured, by fluorimetry and gravimetry, respectively, before and after water passed through the treatment column. The TOG of the eluted water was near zero ppm during the entire study, proving the system high efficiency in removing oil in a semi-industrial scale. This result also shows that the volume of water so eluted was not yet sufficient to saturate the column. With respect to the solids present in water, no influence was noted on the system behavior for the total volume eluted. The TSS measurement indicated that the column retained about 50 % of the solids, reducing the TSS from 21.2 to 10.6 ppm, as shown in Table 2. The practically constant pressure values indicate that the retention of solids by the column did not block the passage of the liquid. However, the retention of solids by the column leads us to believe that with increased elution, the system performance might deteriorate to some extent, such as by clogging of the column, with consequent increased pressure of the system, and/or by a reduced elution volume until reaching saturation. This tendency evidences the drawbacks of the presence of solids, which should be removed before the oil-contaminated water is passed through the column.

The retention of only 50 % of the suspended solids can be attributed to the size differences of the particles of resin (90 to 190 μ m) and bentonite (> 50 μ m). The smaller particles probably elute through the column in the empty spaces left between the resin beads. Therefore, it is expected that the use of resins with smaller bead size will be able to retain a greater quantity of solids. This reasoning suggests that the prior treatment of water to remove suspended solids could be done by placing a pre-filter containing the same polymeric resins, but with smaller particle sizes, which are produced in smaller quantity during the polymerization process.

3.1.3. Second elution phase

The objective of this phase was to reach the column saturation stage with oil as the only contaminant in water. For this purpose, the system was submitted to more drastic conditions than utilized during the first elution phase, with an increase in the oil concentration (TOG values fluctuating between 150 and 350 ppm) and a rise in pressure from 200 to 1000 kPa. After elution of 649,000 ml of water (1200 times the column bed), the TOG levels were near 0 ppm, demonstrating that even after elution of a high volume of water with a high oil concentration, the column saturation point was not reached.

Table 2

Efficiency removal of fine particles present in produced water in a fixed-bed column containing polymeric resins

Volume eluted, ml	Input TSS, ppm	Output TSS, ppm	Retention percentage, %	System pressure, kPa
0.0	0.0	0.0	0.0	0
10000	21.2	10.6	50.0	200
25000	21.2	10.9	48.6	210
55000	21.2	10.3	51.4	200
65000	21.0	10.5	50.0	190
80000	21.0	10.5	50.0	200
110000	21.4	10.7	50.0	200

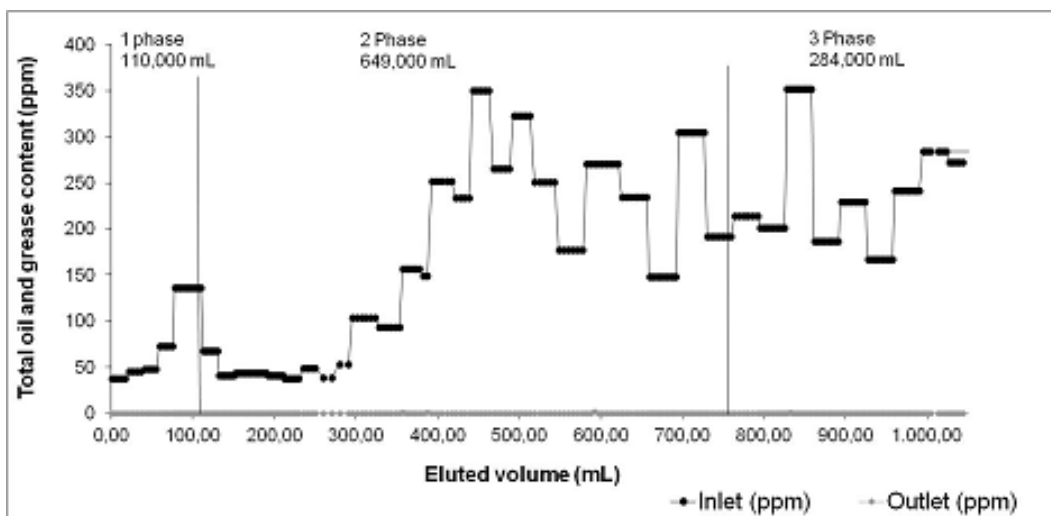


Fig. 2. Monitoring of the total oil and grease (TOG) concentration during 3 phases of the study

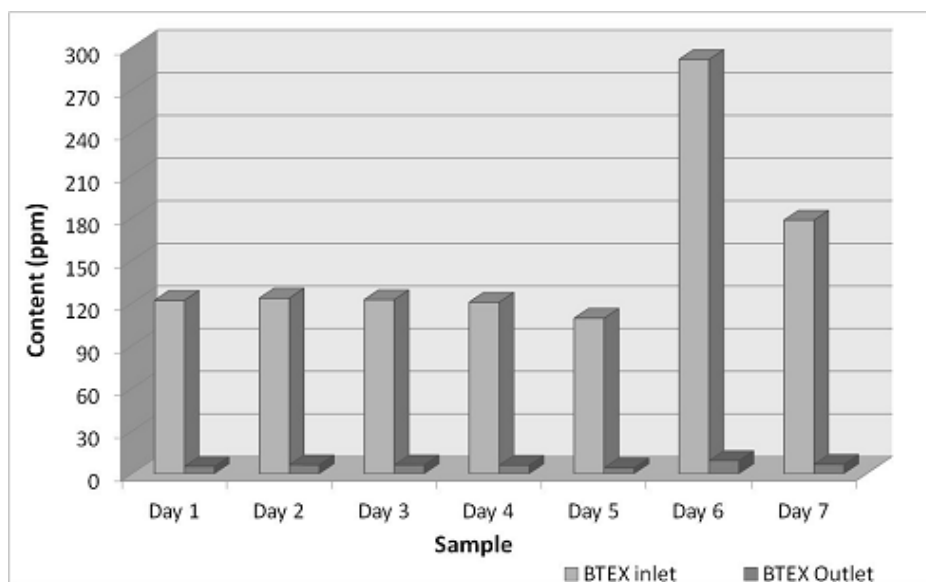


Fig. 3. BTEX retention by the adsorbant column filled with mixed polymeric material

3.1.4. Third elution phase

At the third phase the same conditions were maintained for flow (365 ml/min), pressure (1000 kPa) and oil concentration (around 350 ppm) as at the second phase. Besides this, monoaromatic hydrocarbons were added to the system. At this phase, 284,000 ml of contaminated water was eluted, and the TOG and BTX concentrations were measured.

With respect to the oil retention, according to the TOG measurements, this concentration remained near zero ppm. Fig. 2 presents the TOG profile of the contaminated water and water eluted during the first three phases, during which about 2,500 times the bed volume was eluted without reaching saturation of the column.

The acrylic material used to make the column allowed visual observation of contaminants retention. The filler varied in coloration, from light yellow to black, suggesting that the oil components were adsorbed selectively, at least partly. The column content gradually became darker, as the volume of contaminated water eluted increased. At the end of the study, about 80 % of the material in the column was dark colored, which can be an indication that the column was still capable of retaining more oil before reaching saturation.

The semi-industrial system was thus highly efficient in reducing the TOG content, with the capacity to adsorb over 840 mg of oil for each 1 g of dry resin, demonstrating its excellent potential for use on a full industrial scale to treat produced water in the petroleum industry.

To test the BTX retention capacity, the produced water was spiked with a theoretical concentration of 300 ppm. However, the BTX concentration after

preparation of each batch was only about 120 ppm (as can be observed in Fig. 3). This difference can be explained by the losses during the process of preparing and homogenizing the oily water, because these compounds are volatile, and also because of their low solubility in water: 0.053 g/100 ml for toluene and 0.02 g/100 ml for xylenes, in the temperature range from 293 to 298 K [21]. The aliquots of water eluted in the system had an average concentration of 6 ppm (Fig. 3), representing a reduction of 95 % of the BTX in water. This result proves the potential application of the resin, on a semi-industrial scale, to remove aromatic hydrocarbons of the BTX type from oily water.

3.1.5. Fourth elution phase

In this phase, only produced water was utilized, with previous quantification of polyaromatic hydrocarbons levels. About 288,000 ml of produced water (~533 times the bed volume) were eluted through the column during 24 h at an average flow of 200 ml/min. Table 3 presents the concentrations of various contaminants present in the produced water (input) and the eluted water 12 and 24 h afterward (output). The polymers affinity for the polyaromatic compounds was also satisfactory: the system continued to be highly effective in removing PAHs from water. The mean adsorption efficiency was 99.3 % after 12 h and 96.3 %, after 24 h of treatment.

It is important to stress that the excellent performance was attained with the column already containing solids and different types of contaminants, to a certain extent reproducing various situations that would be found in real application.

Table 3

Retention of polyaromatic hydrocarbons by a system containing polymeric resin packed with mixed filler

Polyaromatic	Input, ppm	Output after 12 h, ppm	Output after 24 h, ppm
Naphthalene	0.50	0.04	0.27
Acenaphthylene	0.48	-	-
Acenaphthene	0.36	-	-
Fluorine	1.63	-	-
Phenanthrene	7.27	0.05	0.2
Anthracene	0.34	-	-
Fluoranthene	0.56	-	-
Pyrene	0.83	-	-
Benz[a]anthracene	0.19	-	-
Chrysene	0.62	-	-
Total PAHs	12.77	0.09	0.47
Efficiency (%)		99.3	96.3

Note: (-) not detected

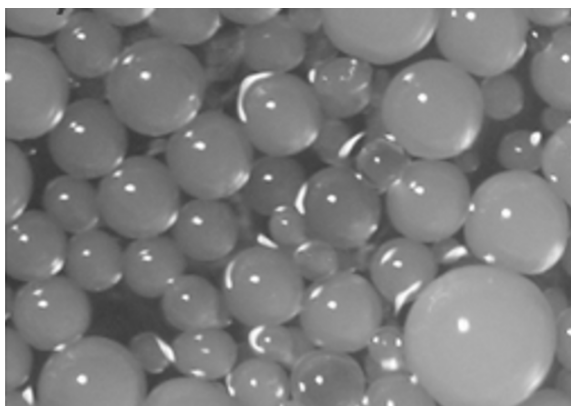


Fig. 4. Optical micrographs of the polymeric resin beads before using to treat contaminated water

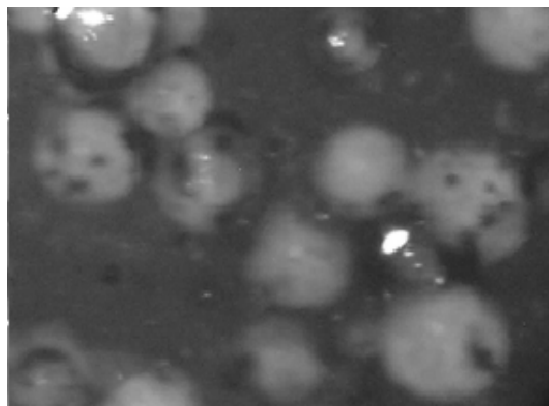


Fig. 5. Scanning electron images of the polymeric resin beads before using to treat contaminated water

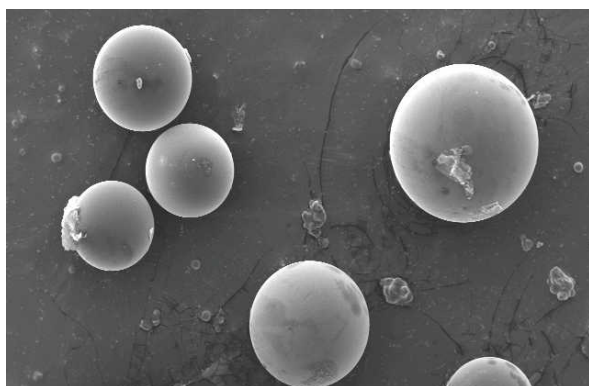


Fig. 6. Optical micrographs of the polymeric resin beads after using to treat contaminated water

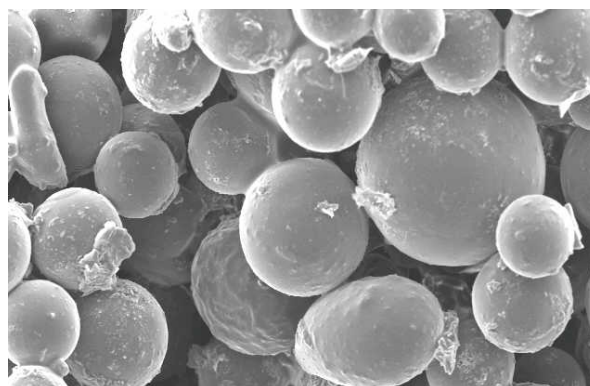


Fig. 7. Scanning electron images of the polymeric resin beads after using to treat contaminated water

3.1.6. Evaluation of the morphological integrity of the beads

Another important aspect of the present application is the resistance of the resins after undergoing extreme mechanical stress, caused by the pressure imposed by the system. Due to the high flow rates applied during the process, the column packed with polymeric materials came under pressures of nearly 1000 kPa. Figs. 4 and 5 present the micrographs of the polymeric material before being inserted in the column and Figs. 6 and 7 show the material at the bottom of the column after the end of the study. It can be seen that the material remained spherical and did not suffer any fractures. This result indicates that the resin is able to withstand a regeneration process for subsequent reuse, which is extremely important when considering the industrial use of this process, especially on offshore oil platforms.

4. Conclusions

The semi-industrial system composed of a column containing polymeric resins based on poly(divinylbenzene) and poly(divinylbenzene-co-methyl methacry-

late), under real conditions of produced water, exhibited a similar performance on retaining oil, mono- and polyaromatic hydrocarbons as the bench one. The system was able to remove more than 98 % of oil, even after elution of 2,500 times through the column bed volume. For mono- and polyaromatic hydrocarbons, the retention was above 95 %, a very satisfactory result as well. The results indicate that the system has an excellent performance and longevity, attributed mainly to the chemical affinity of the resins for the contaminant compounds and the high surface area of the beads, facilitating contact for adsorption. These results along with the mechanical resistance of the polymer beads under severe conditions imposed by the semi-industrial system definitively show that it can be scaled up for industrial application.

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АДСОРБЦІЯ БТК ТА ПАВ ПОЛІМЕРНИМИ СМОЛАМИ НА СТАЦІОНАРНОМУ ШАРІ: НАПІВПРОМИСЛОВА ОЦІНКА

Анотація. Синтезовані суспензійною полімеризацією на пілотній установці полімерні смоли, якими була заповнена акрилова колона, для використання її в напівпромисловій системі елюювання. Вивчена поведінка полімерних смол як адсорбентів нафтовмісних вод. Встановлено, що ефективність вилучення БТК (бензол-толуол-ксилоли) становить більше ніж 95,9 %, а вміст ПАВ (поліароматичних вуглеводнів) зберігається на рівні 96,3 %.

Ключові слова: пластова вода, нафта, БТК, ПАВ, полімерні смоли, адсорбція.