SBA-15 MOLECULAR SIEVE: SYNTHESIS, CHARACTERIZATION, AND APPLICATION IN OIL/WATER SEPARATION

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ABSTRACT

This work evaluates the use of molecular sieve SBA-15 as adsorbent in oil removal process. The synthesis and characterization tests used X-ray Diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDX), physical N₂ adsorption, Scanning Electron Microscopy (SEM), adsorption capacity in organic solvents (gasoline, kerosene, and diesel oil), and Foster Swelling. Following, this work performed the evaluation of SBA-15 as adsorbent in the process of removing oil from a synthetic effluent, through the finite bath system (Batch adsorption). Finite bath system tests used a factorial 2² experimental design with three experiments at the central point. At that stage, two variables were evaluated: initial oil concentration (100, 300, and 500 mg /L) and mechanical agitation (100, 200, and 300 rpm). Through the experiments, it was possible to observe the following response variables: percentage of total removal (% R_{em}) and removal capacity in equilibrium (q_{eq}). For comparison level, the experiments also evaluated response variables for the tests without mechanical agitation. Characterization techniques (DRX, EDX, SEM, Physical N₂ Adsorption) presented results compatible with the characteristics presented by mesoporous materials. Foster Swell tests confirmed the affinity of SBA-15 molecular sieve with the organic solvents used: gasoline, kerosene, and diesel oil. The testing system determined both finite bath oil removal percentage (% R_{em}) 97.04%, and removal capacity at equilibrium (q_{eq}) of 48.58 mg /g, indicating that the use of SBA-15 molecular sieve is a viable option in the oil removal process.

KEYWORDS

SBA-15; adsorption; oil removal process; organic solvent; effluent

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1. INTRODUCTION

Among the problems created by organic contaminants, the pollution of water by oils is one of the most troublesome. This happens because fossil fuels, such as petroleum and its by-products are raw material for the generation of energy in many industrial processes. The oil negatively impacts the aeration and the natural illumination of watercourses, due to the formation of a film insoluble in the surface, producing harmful effects on aquatic fauna and flora (Diagboya & Dikio, 2018; Rubio, 2007).

Since oil refineries are generally large consumers of water, they produce large amounts of effluent. Petroleum refining entails the generation of a significant amount of potentially polluting waste (Felix, 2006). The oily effluents display a very complex composition, which may contain oil (mineral, vegetable, or synthetic), fatty acids, emulsifiers, corrosion inhibitors, bactericides, and other chemical compounds (Gryta et al., 2001).

According to Gryta et al. (2001), most of the oily effluents are in the form of oil /water emulsions. The adsorption phenomenon was discovered around the 18th century. The first observations date back to 1973, when Scheele conducted experiments with activated charcoal and adsorbent, finding out that these materials could retain certain gases. Later in 1973, Lowitz observed the same phenomenon, performing experiments with solutions (Devi, 2017; Hamid & Shahadat, 2017).

Adsorption is a phenomenon in which molecules of a fluid phase adhere on a solid surface without chemical modification. In general, adsorption occurs as a result of unbalanced forces on the surface of a solid agent, the adsorbent, which holds certain molecules of the fluid and the adsorbate around its surface (Daí, 2018).

Today, there is a growing interest in preserving the environment, especially when it comes to the responsible use of non-renewable natural resources. Inspections by competent bodies evaluate environmental impacts of industrial processes and stimulate industries to buy into environmental preservation efforts by, for example, promoting the treatment of their effluents (Daí et al., 2018; Fu & Wang, 2011).

In the treatment of effluents, the use of mesoporous silica with narrow pore size distribution has attracted much attention for its use as support, because they provide better control of pore size, enhancing catalytic properties. Special emphasis has been given to SBA-15, a silica-based mesoporous material with uniform hexagonal channels ranging from 3 to 30 nm, very narrow pore size distribution, and high surface area (600–1000 m²/g) (Cheng et al., 2018; Huyen et al., 2018; Know & Chae, 2018; Ningmeng et al., 2018). Interestingly, SBA-15 also has thicker pore walls and better hydrothermal stability than MCM-41 (Cano et al., 2011; Garcia et al., 2018; Ghampson et al., 2010; Meloni et al., 2016; Rodrigues et al., 2012; Rodrigues et al., 2013; Zhang et al., 2018), a feature that can be relevant during the oil removal process.

The objective of this work is to evaluate, on a laboratory scale, the performance of the molecular sieve SBA-15 in the removal of oil in a synthetic oily effluent through the adsorption process.

2. EXPERIMENTAL

2.1 Preparation of SBA-15 molecular sieve

SBA-15 was synthesized according to the procedure reported in the literature (Rodrigues et al., 2013). First, 2 g of Pluronic P123 (Aldrich) were dissolved in 15 g of deionized water and 60 g of 2 mol/L HCl solution while stirring. Then, 4.25 g of tetraethyl orthosilicate (TEOS, 98 wt %, Sigma-Aldrich) were added to the solution while stirring. This mixture was stirred continuously for 24 hours. The gel mixture was then transferred to a Teflon-lined autoclave and kept static at 95 °C for two days. The product was centrifuged at 11000 rpm, washed with deionized water several times, and dried at 100 °C. The material was calcined in static air at 550 °C for 2 hours to remove the polymer template and to obtain the white powder SBA-15. The sample was synthesized with the following molar composition: 1.0 TEOS:0.017 P123:5.7 HCl:193 H₂O.

2.2 Characterization of the samples

X-ray energy dispersion spectrophotometer (EDX): Elemental analysis was determined through energy dispersive X-Ray spectrophotometry, in a Shimadzu EDX-700 instrument.

X-Ray Diffraction (XRD): Powder diffraction patterns were measured on a Shimadzu XRD 6000. The operational conditions were: Copper K α radiation at 40 KV/30 mA, goniometer velocity of 2°/min with a step of 0.02° over the 2 θ -range from 2° to 80°. The average diameter of the sample crystallites was determined by the Scherrer equation.

Nitrogen Adsorption (BET method): The textural characteristics of the analyzed samples were investigated by isothermal gas adsorption/desorption of N₂ at -196 °C using a Micrometrics ASAP 2020 equipment. The adsorption and desorption N₂ isotherms were obtained in the range of relative pressure (P/P₀) between 0.02 and 1.0. The values of the average pore diameter and surface area (S_{BET}) were obtained by the proposed method (BET).

Scanning Electron Microscopy (SEM): The micrographs of the samples were determined through a scanning electron microscope (XL30 EDAX).

Adsorption Capacity: The gasoline (Petrobras), diesel (Petrobras) and kerosene (Ares) sorption capacities were measured following a method based on the "Standard Methods of Testing Sorbent Performance of Adsorbents" (ASTM F716-82 and ASTM F 726-99). In this method, the adsorbent sample is weighed (minimum 1g), and the exact value is recorded. The test cell is filled with an initial layer of test liquid. The adsorbent is placed in a basket (a 200- stainless steel mesh basket), and, then, lowered into the test cell. The adsorbent should be allowed to float freely within the test cell. After 15 minutes $\pm$ 0.33 minute, the basket with the adsorbent is removed manually in a vertical direction and allowed to drain for 15 seconds $\pm$ 3 seconds. A tared weighing support is placed under the adsorbent sample/mesh basket set to catch any additional drips and the entire system is weighed.

All tests were conducted in duplicate with a minimum of two runs used for calculations. Based on the data obtained, the amount of adsorbed oil was calculated as a weight ratio of adsorbed oil to dry adsorbent. The adsorption capacity was obtained with Equation 1, referring to the adsorption efficiency (A_d) of the adsorbent tested, in percentage. In equation, P₁ is the mass of material after adsorption and P₂ is the mass of dry

adsorbent material, both in grams. The results of the adsorption capacity can also be reported in grams of solvent adsorbed per gram of adsorbent.

$$A_d = \left(\frac{P_1 - P_2}{P_2} \right) * 100 \quad (1)$$

Foster Swelling Test: This technique is employed to carry out studies on the compatibility of molecular sieve SBA-15. The organic solvents for the study of the swelling phenomena were: gasoline (Petrobras), diesel oil (Petrobras), and lubricating oil (Lubrax MG1). The test consisted in adding 0.5 g of the powder sample to 50 mL of solvent in a test tube. After 24 hours of rest, the reading of the adsorbent column volume (swelling of the adsorbent without agitation) was registered. Afterwards, the content of the test tub was stirred with a glass stick during 5 minutes. After an extra 24-hour settling period, a new reading of the adsorbent column volume (swelling of the adsorbent with agitation) was taken. The following parameters were fixed for the interpretation of the test: non-swelling (minor or equal to 2 mL/g of adsorbent), low swelling (between 3 and 5 mL/g of adsorbent), intermediate swelling (between 6 and 8 mL/g of adsorbent), and high swelling (more than 8 mL/g of adsorbent) (Kwon et al., 2018).

Analysis of variance of the separation process: A factorial design was proposed to analyze the influence of initial oil concentration in aqueous solution (C₀) and the mechanical agitation of the system (A) on the separation of emulsified oil in water emulsions. The variables obtained from this study were: the total oil removal percentage (%R_{em}) and the capacity of oil removal at equilibrium (q_{eq}). The analysis of variance was performed using the Minitab 16.1 Software. The values for levels (+) and (-) are shown in Table 1.

2.3 Finite bath system

The finite bath tests were prepared according to the experimental design. A total of 0.5 g of the adsorbent was added to 50 mL of oil (LUBRAX MG1) emulsified in water with concentrations 100, 300, or 500 ppm, depending on the test. After adding the adsorbents, the suspension (emulsion + adsorbent) was allowed to stir mechanically in an orbital shaker for 6 hours, to ensure the equilibrium of the system. The concentrations of oil at equilibrium were determined by absorbance

Table 1. 2² - factorial experimental design.

Test	Concentration (ppm)	Agitation (rpm)	Coded Concentration (ppm)	Coded Agitation (rpm)
1	100	100	-	-
2	500	100	+	-
3	100	300	-	+
4	500	300	+	+
5	300	200	0	0
6	300	200	0	0
7	300	200	0	0

analysis, using a UVvisible spectrophotometer. An analytical curve was constructed by plotting the absorbance versus the oil concentration, which ranged from 0 to 100ppm (0, 10, 20, 30, 40, 60, 80, and 100) of oil. After a six-hour period, the samples were filtered and, from the filtrate, a 10mL aliquot was collected from each sample. Following, 10 mL of chloroform were added to act as oil extractor in the liquid/liquid extraction, because the chloroform has a significant peak at 262nm. According to [Greenberg et al. \(1985\)](#) and [Henderson et al. \(1999\)](#), at this wave length the absorbance is used commonly to estimate the concentration of oil in produced water and indicate the bands of aromatic C-H. The samples were, again, stirred in the orbital shaker for 5 minutes at 100rpm. After stirring, there was a formation of two phases: a denser one of oil solubilized in chloroform, and another with the remaining water from the emulsion. The collection of the denser phase was done with a glass syringe and transferred to a quartz cuvette, where the liquids were put into the spectrophotometer for reading. The total oil removal percentage (%Rem) and the capacity of oil removal at equilibrium (qeq, in mg of

oil/g of adsorbent) were obtained with Equations 2 and 3, respectively:

$$\%R_{em} = \left(\frac{C_0 - C_{eq}}{C_0} \right) * 100 \quad (2)$$

$$q_{eq} = \frac{V}{m} (C_0 - C_{eq}) \quad (3)$$

Where, C_0 is the initial concentration of oil in the emulsion, in mg/L; C_{eq} is the final concentration of oil in the emulsion, in mg/L; V is the volume of adsorbate, in mL; and m is the mass of adsorbent, in g.

3. RESULTS AND DISCUSSION

Figure 1 shows the result of the X-ray of SBA-15 molecular sieve synthesized and calcined. SBA-15 gave three distinct diffraction lines due to SiO_2 planes (100), (110), (200), indicating the P6-mm hexagonal symmetry structure, as reported in literature ([Rodrigues et al.,2013](#); [Zhao et al., 1998](#)).

Figure 1. X-Ray patterns of the SBA-15: a) SBA-15 synthesized and b) SBA-15 calcined.

Table 2. Values of the 2 θ angles and their respective interplanar distances for the SBA-15 samples.

Samples	2 θ	hkl	d _(hkl) (Å)	a ₀ * (nm)
SBA-15 synthesized	0.80	100	110.4	12.7
SBA-15 calcined	0.96	100	92.0	10.6

* Parameter of hexagonal unit cell.

Table 3. Chemical composition of SBA-15.

Sample	SiO ₂ (%)	Impurities (%)
SBA-15	96.913	3.087

Table 4. Textural analysis of the calcined SBA-15.

Sample	S _{BET} (m ² /g)	V _{Ptotal} (cm ³ /g)	e (nm)
SBA-15	627	2.2	8

S_{BET}: specific area; V_{Ptotal}: pore volume; e: wall thickness

The internal structure of mesoporous blocks is constituted by a hexagonal arrangement of mesoporous cylinders, characterized by values of interplanetary space d₁₀₀ and a parameter of hexagonal unit cell a₀. Simple analytical equations evaluate the cell parameter from the data using the interplanar space d₁₀₀ (most defined peak) of the infinite hexagon X-ray diffractogram of uniform pores (Rodrigues et al., 2013). The parameter a₀ can also be written according to Equation 4. This parameter is represented by the distance between two centers closest to the cylindrical mesopores and reflects the periodicity of the system.

$$a_0 = d_p + a_H \quad (4)$$

Where, d_p is the diameter of the pores and a_H is the parameter of the mesoporous hexagonal arrangement. From the diffractograms of Figure 1, the network parameter a₀ for SBA-15 was calculated as shown in Table 2.

After the calcination of the SBA-15 molecular sieve, one can observe a contraction. This phenomenon occurs due to the removal of the Pluronic P123 agent.

The chemical composition of SBA-15 and the catalysts are presented in Table 3. The support and catalysts presented high levels of silica (SiO₂), which was expected because the structure of SBA-15 molecular sieves consists solely of silica. The sample presents a certain degree of impurity (3.087%). This refers to the contaminations present in the samples, reagents, and also to elements that present the same excitation energy (Ghampson et

al., 2010; Cano et al., 2011).

The textural analysis of the calcined SBA-15 support and of the catalysts is presented in Table 4. Three very distinct regions of the SBA-15 adsorption isotherm are observed (Figure 2): monolayer-multilayer adsorption, capillary condensation, and multilayer adsorption on the outer surface. The first one, given at low relative pressures (P / P₀ < 0.4), corresponds to the adsorption of nitrogen in the monolayer. In the second inflection between P / P₀ = 0.5 - 0.8 occurs the capillary condensation, characteristic of mesoporous materials. The third (P / P₀ > 0.9) can be attributed to the adsorption of the multilayers of the external surface (Zhao et al., 1998).

The combination of techniques (X-ray diffraction and Nitrogen Adsorption) allowed the calculation of the wall's thickness (Rodrigues et al., 2013; Zhao et al., 1998; Ghampson et al., 2010; Cano et al., 2011). Table 3 lists the specific area and total specific pore volume of the calcined SBA-15 molecular sieve. These characteristics are typical of SBA-15 structures synthesized under similar conditions (Cano et al., 2011). This attributes high resistance mechanical materials and the chance of application as catalytic supports in industrial processes where the catalysts are subjected to high temperatures and pressures.

These characteristics are related to materials with pores of constant cross-section (cylindrical or hexagonal). The P/P₀ inflection position between 0.60 and 0.80 confirms this structural feature of pores.

Figure 2. Adsorption-desorption isotherms of N_2 at 77 K of SBA-15.

Figure 3. Scanning electron microscopy of SBA-15.

Figure 3 presents images of microscopy molecular sieve SBA-15. Looking at the images, one can observe silica nodule formations in micrometric dimensions connected with each other by smaller particles forming collars interlocking, characteristic of SBA-15 molecular sieve (Rodrigues et al., 2013; Zhao et al., 1998).

The experimental results of the adsorption capacity for SBA-15 are shown in Table 5. From these results, it is possible to suggest that organic samples have potential for adsorption in all

solvents, in the following order: diesel > gasoline > kerosene.

The best result in absorption with SBA-15 was obtained using the solvent diesel. This fact probably indicates that there is a better interaction of SBA-15 with solvent diesel. This interaction is related directly to the chemical composition of compounds (gasoline: C_5 - C_{10} ; kerosene: C_{11} - C_{12} ; diesel: C_{13} - C_{17}). The adsorption test may be attributed to the viscosity of the solvent, since the viscosity results are: diesel 8 cP (Centipoise) (600

Table 5. Results of adsorption capacity in the presence of gasoline, diesel, and kerosene performed for SBA-15.

Solvent	SBA-15
Gasoline	1.788 (g/g)
kerosene	1.501 (g/g)
Diesel	2.145 (g/g)

Figure 4. Results of the Foster Swelling Tests performed in different organic solvents, with and without agitation.

rpm) and 3.5 cP (300 rpm); kerosene 2 cP (600 rpm) and 1 cP (300 rpm); and gasoline 2 cP (600 rpm) and 1 cP (300 rpm) (Rubio, 2007; Diagboya & Dikio, 2018).

Foster Swelling Tests verified the affinity with complex mixture of hundreds of hydrocarbons (gasoline, kerosene, and diesel). Figure 4 shows the results. In these assays, all samples were put in contact with different solvents (gasoline, kerosene, and diesel).

The treated sample presented a high expansion degree when tested in kerosene (10 mL/g of adsorbent). One could observe enhanced interaction (16 mL/g of adsorbent) after agitation of the sample. Conversely, when mixed with gasoline, the treated sample presented a low expansion degree despite performing agitation (6 mL/g of adsorbent). When diesel was used, the sample presented an intermediate expansion degree (8 mL/g of adsorbent), but after agitation

the expansion degree was higher (10 mL/g of adsorbent).

The efficiency of adsorbent in the removal of emulsified oil in water was evaluated by finite bath tests following a 2^2 factorial design (Table 1). The percentage of oil removal (% R_{em}) and the removal capacity (q_{eq}) are shown in Table 6.

Where C_0 refers to the estimated initial oil concentration in the emulsion; C , is the real concentration of oil in the emulsion; and C is the final oil concentration, after the assay. The results presented in Table 6, for agitation experiments, indicate that the highest percentage of total oil removal (% Rem) was found in Test 2 under the following conditions: agitation factor of 100 rpm and concentration initial oil 500 mg/L, when 97.04% of the emulsified oil was removed. The highest oil removal capacity at equilibrium (q_{eq}) was obtained in test 2, under the same conditions, with a value of 48.58 mg of oil/g of SBA-15.

Table 6. Results of the 2^2 - factorial experimental designs for treated SBA-15.

Assay	C_0 (mg/L)	Agitation (rpm)	C (mg/L)	% Rem	q_{eq} (mg/g)
1	100	100	16.14	83.86	8.39
2	500	100	14.22	97.04	48.58
3	100	300	9.45	90.55	9.05
4	500	300	20.49	95.90	47.95
5	300	200	9.75	96.75	29.02
6	300	200	9.45	96.85	29.05
7	300	200	9.57	96.81	29.04

Table 7. Results obtained in oil removal tests, without agitation.

Assay	C (mg/L)	Agitation (rpm)	C (mg/L)	% Rem	q _{eq} (mg/g)
1	100	0	5.43	94.57	9.46
2	500	0	15.89	96.82	48.41

Nevertheless, test 1 showed the lowest percentage of removal under the conditions: agitation factor of 100 rpm and initial concentration of 100 mg /L.

Table 7 shows data according to the experiments conducted without agitation.

SBA-15 molecular sieve removed 96.82% of emulsified oil, corresponding to a removal capacity of 48.41 mg of oil per gram of SBA -15, at high concentrations (500 mg / L). However, at low concentrations (100 mg / L) only 9.46 mg of oil per gram of SBA-15 were removed in equilibrium.

4. CONCLUSIONS

According to XRD, BET, EDX, and SEM results, SBA-15 has a structure that includes multi-level mesopores, crystallites, and grains within the original particles.

Foster Swelling test results indicate that the affinity with the SBA-15 in all organic solvents tested (gasoline, diesel, and kerosene), both with and without agitation. These adsorption capacity results suggest that organic samples have potential for adsorption in all solvents, with the following order: diesel > gasoline > kerosene.

Results obtained from the finite bath tests determined percentages of oil removal (% Rem) as high as 97.04%, and oil removal capacities at equilibrium (q_{eq}) of up to 48.58 mg/g. These results indicate that the SBA-15 molecular sieve presents a significant alternative for the traditional processes of removing oil from the water.

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