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Sugarcane Bagasse As Potentially Low-Cost Biosorbent

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Abstract

Sugarcane bagasse (SB) is one of the major residues obtained from agriculture, every year millions of tons of SB have been produced by the sugarcane agribusiness. This abundant residue has been showed potential as biosorbent in wastewater treatment. SB, *in nature* or chemically modified, has been widely reported as a promising sorbent for the removal of dyes or heavy metals from aqueous medium. The application of SB in oil sorption is rarer, especially for the treatment of used motor oil wastewater. However, in this chapter, we show that this material has good oil sorption capacity when compared to other commercial and natural sorbents. This study evaluates the effect of several coupling agents over SB in used motor oil sorption as well as the influence of surfactant in this process.

Keywords: engine washing wastewater, oil sorption, sugarcane bagasse, dye sorption, biomass

1. Introduction

Agricultural waste by-products, such as sugarcane bagasse (SB), rice husk, coconut husk, sisal, and so on, have been extensively studied as a potential sorbent material for removing contaminants from water and wastewater [1]. These materials have many advantages such as the abundance, low cost, floatability, good flexibility and mechanical strength, and environmentally friendly properties. Besides, the potential use of several agricultural by-products as biosorbent is supported by its native adsorption capacity derived from their main constituents such as cellulose, hemicellulose, and lignin. These are polymeric structures with high content of hydroxyl and carboxyl groups, which have a strong influence in the adsorption capacity of different chemicals present in the aqueous medium.

SB, in nature or chemically modified, has been reported as a potential renewable sorbent for wastewater treatment [2, 3]. Millions of tons of SB have been generated every year by the sugarcane agribusiness, encouraging its reuse and recycling. The sugarcane industry is based mainly on the production of sugar and ethanol, which generates huge volumes of SB and sugarcane trash [4, 5]. After sugarcane is milled for juice extraction, bagasse is obtained as a residue, which corresponds to about 25% of the total weight, and is composed of approximately 40% cellulose, 24% hemicellulose, and 25% lignin. The hydroxyl groups are the most abundant and reactive sites in this biopolymer and are used to attach a variety of functional groups [6]. The higher content of cellulose in the SB biomass favors its hydrophilicity, which improves its interaction with cationic species in aqueous medium. As a result, SB has been widely used for the removal of heavy metals and dyes from wastewater [7–9].

1.1. Sugarcane bagasse as a sorbent for heavy metals removal

The utilization of unmodified or modified SB as an adsorbent have been described as a cheaper and effective technology for the removal of metal ions from wastewater [10, 11]. The metal ion-binding mechanism of adsorption on SB is attributed to its abundance of hydroxyl groups from cellulose, in which aqueous medium favors ion exchange or complexation with metal ions. Batch studies [12] using natural SB as a sorbent for removal of Cd(II) show the maximum adsorption at pH 6. The pH dependence of Cd(II) uptake was linked to both the surface functional groups and the metal ion species predominant in aqueous solution. The species Cd^{2+} and $\text{Cd}(\text{OH})^+$ are predominant at pH lower than 6, while the groups on surface are protonated and cannot bind to metal ions in solution. Besides, at very low pH, the surface groups are associated with the hydronium ions (H_3O^+), negatively affecting the interaction with the metal cations. When the pH increases, the surface affinity with the metal also increases, and adsorption is improved.

The metal ion-binding capacity of SB can be intensified by the introduction of surface groups with capacity chelating as carboxylate or amine [13–15]. The introduction of carboxylic functions ($-\text{COOH}$) on cellulosic fiber, lignin, and hemicellulose can be performed via cyclic anhydride reaction. **Figure 1** displays an example of succinylation [16] reaction as an alternative route to attach carboxylic acid onto the cellulose.

Pereira et al. describe the chemical modification of SB by ethylenediaminetetraacetic (EDTA) dianhydride (EDTAD) in order to improve Zn^{2+} adsorption [14]. The EDTA molecule is a

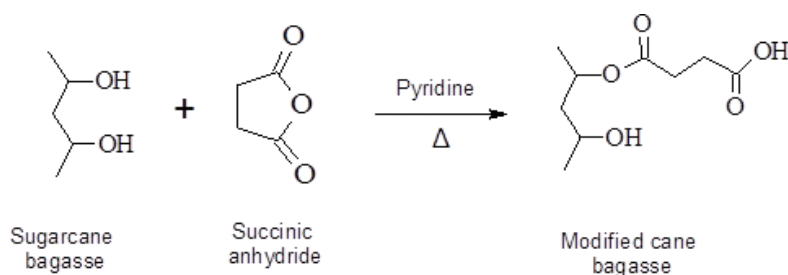


Figure 1. Scheme of possible synthetic route for introduction of carboxylic groups.

chelating group that enhanced the metal complexation on fiber surface. Other compounds such as citric acid and phthalic anhydrides have also been used for SB modification, resulting then in an increase of adsorption capacity for these fibers [9, 13, 17, 18]. **Table 1** summarizes some studies where SB in nature or modified form is used to remove heavy metals from aqueous environment.

1.2. Sugarcane bagasse as an adsorbent for removal of dyes

With the expansion of textile sector, dyes in wastewater have become a serious environmental problem. Dyes are organic chemical compounds that appear colored due to the presence of chromophore groups such as nitrous, azo, and carbonyl [19, 20]. The release of dye waste into water bodies affects the life in aquatic environments, causing the ruining of soils and poisoning

Metal ion	SB/modification	Sorption capacity (mg g ⁻¹)	References
Cd ²⁺	SB without modification	69.06	[12]
Zn ²⁺	Ethylenediaminetetraacetic dianhydride	105.26	[14]
		45.45	
Pb ²⁺	Without modification	26.67	[17]
	<i>Pleurotus ostreatus</i> (U2-11)	36.31	
	<i>Lentinula edodes</i> (U6-1),	27.68	
	Basidiomycetes	39.93	
	<i>Ganoderma lucidum</i> (U12-6)	36.00	
Co ²⁺	Trimellitic anhydride	1.153	[3]
Cu ²⁺		0.979	
Ni ²⁺		0.849	
Cu ²⁺	Tetraethylenepentamine	0.016	[18]
Cu ²⁺	Succinic anhydride	114	[15]
Cd ²⁺		196	
Pb ²⁺		189	
Co ²⁺	Phthalic anhydride	0.561	[3]
Cu ²⁺		0.935	
Ni ²⁺		0.932	
Cr ³⁺	Without modification	16.21	[13]
	NaOH	26.41	
	Citric acid	17.2	
	NaOH/acylation with citric acid	31.28	

Table 1. SB in nature or modified as metal sorbent reported in the studies.

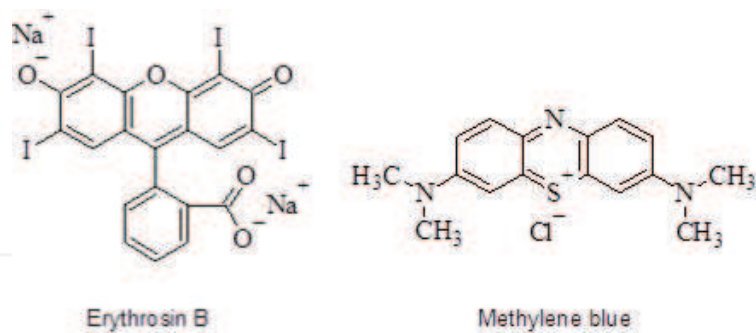


Figure 2. Example of cationic (erythrosin B) and anionic (methylene blue) dyes.

of drinking water. Besides, dyes cannot be removed by conventional treatment methods, and are resistant to aerobic digestion. As an alternative method, physical removal of dyes from effluent through biosorption has been extensively studied [8, 21]. A dye molecule is characterized by the presence of chromophore groups, which are responsible for producing the color, and also by groups known as auxochromes such as carboxylic acid, sulfonic acid, amino, and hydroxyl groups. These auxochromes are responsible for impacting or shifting of a particular color when attached to a chromophore, and also used to influence the dye solubility. In fact,

Dyes	SB/modification	Sorption capacity (mg g ⁻¹)	References
Crystal violet	Oxidation with H ₃ PO ₄ -NaNO ₂	1018.2	[8]
Auramine O		571.8	
Methylene blue	Maleic anhydride	30.4	[25]
Rhodamine B	Without modification	51.3	[2]
Methylene blue		28.0	
Acid Alizarin Violet N	Without modification	20.8	[26]
Methyl red	Phosphoric acid	11.0	[21]
	Without modification	5.7	
Congo red	Without modification	39.8	[24]
Methylene blue	Ethylenediaminetetraacetic dianhydride	115.3	[27]
Crystal violet	Meldrum's acid	552.7	[7]
Methylene blue	Ethylenediaminetetraacetic dianhydride	192.3	[19]
Gentian violet		357.1	
Methylene blue	Succinic anhydride	434.3	[16]
Gentian violet		1133.7	
Erythrosin B	Without modification	333.3	[22]
Methylene blue		1000.0	

Table 2. Examples of dyes adsorption by SB.

the auxochromes are important to enhance the affinity of the dye toward the fibers. As a result, natural fiber, as SB, presents greater potential to remove dyes from wastewater.

Basically, dyes can be classified as cationic or anionic. **Figure 2** shows, for example, the molecules of erythrosin B (EB) and methylene blue (MB). Cationic dyes carry a positive charge in their molecule, while anionic dyes carry a negative charge [22–24]. In aqueous solution, the dye molecules will present positively or negatively charged as a function of pH, and the electrostatic interaction with the fiber surface will direct the adsorption process. Therefore, the dyes adsorption route by SB can be described in a similar way as observed for metal ions. At low pH, the surface of SB became positively charged and the cationic dye adsorption will decrease, while for anionic dyes the reverse process occurs. In contrast, at high pH, the cationic dye removal will increase because the surface appears negatively charged and the anionic dye adsorption became inhibited. **Table 2** summarizes some results of adsorption of dyes using SB, natural or modified, as a sorbent.

2. Sugarcane bagasse-based sorbents for motor oil removal

Research involving oil sorbents was firstly encouraged by the great environmental accidents generated by oil spills at sea [28–30]. In these cases, the adsorption processes are more suitable to remove and recover the oil. The sorbent material facilitates a transformation from liquid to solid phase, and then oil can be removed together with the sorbent. The main characteristic of crude oil sorbent material is the hydrophobicity and oleophilicity in order to attract oil preferentially to water. However, the amount of sorbents added to an oil polluted environment is a critical factor, because the inappropriate and excessive use can present subsequent waste disposal problems. It is especially important when organic synthetic products are used as sorbents. Synthetic sorbents, as polypropylene, do not degrade and are very expensive. Therefore, agricultural waste by-products were firstly used as an alternative oil sorbent to replace the conventional and non-degradable sorbent used to clean up oil spills. These biosorbents are biodegradable, renewable, abundant, and low cost. Teas et al. [31] compared the oil sorption capacity of cellulose with the expanded perlite and polypropylene in artificial seawater containing crude oil. These authors observed that for crude oil, the sorption capacity of cellulose overtakes the other sorbents. When light cycle oil and light gas oil were used in artificial seawater, they observed lower sorption by cellulose in relation to polypropylene, but similar behavior to expand perlite. The oil sorption capacity of vegetal fibers observed by several authors has been attributed to the interaction with hydrophobic sites in the biomass. Lignocellulosic fibers contain both hydrophilic and hydrophobic groups; however, the cellulose structure has hydrophilic nature with excellent wettability. The chemical functionalization of cellulose can increase its hydrophobic character, which is possible by changing the hydrophilic groups, hydroxyl (–OH) in the raw coir cellulose to hydrophobic hydrocarbons [32–34]. The biomass acetylation has been extensively used to increase its oil sorption capacity. Sun et al. [35] observed that SB acetylated presents greater machine oil sorption capacity ($13.5\text{--}20.2\text{ g g}^{-1}$) than polypropylene fibers (10 g g^{-1}). **Table 3** summarizes other studies that have been demonstrated the potential sorbent of agricultural by-products to oil removal and the effect of biomass modification in the sorption capacity.

Adsorption/sorption conditions	Sorbent/treatment		Sorption capacity (g g ⁻¹)	References
Crude oil in dry conditions	<i>Dacryodes edulis</i> leaf	Unmodified leaf	3.440	[33]
		Acetylated with acetic anhydride	4.990	
	Raw cotton		30.5	[36]
Crude oil in distilled water	Sisal (<i>Agave sisalana</i>)		3.0–6.4	[28]
	Coir fiber (<i>Cocos nucifera</i>)		1.8–5.4	
	Sponge-gourd (<i>Luffa cylindrica</i>)		1.9–4.6	
Crude oil in artificial seawater	Mangrove barks	Untreated bark	1.666	[37]
		Treated with oleic acid	3.333	
		Treated with palmitic acid	3.333	
Machine oil in distilled water	Sugarcane bagasse	Untreated bagasse	8.9	[35]
		Acetylated with acetic anhydride	11.4–16.5	
	Sugarcane bagasse modified with acetic anhydride		13.5–20.2	[38]
Synthetic effluent of crude petroleum	Sugarcane bagasse		6.65	[39]
Motor oil in water	Natural wood fibers		33–43	[40]
Wastewater of used motor oil	Natural wood fibers		5.56	[41]
Engine oil in dry conditions	<i>Ceiba pentandra</i> (L.) Gaertn. fibers (Kapok) packing density 0.02 g mL ⁻¹)		47.4	[42]
Used engine oil in dry conditions			50.8	

Table 3. Examples of oil sorption by biomass.

In spite of crude oil sorption favored by hydrophobic surface, in the case of used motor oil, this behavior is not completely precise. The crude oil and fresh motor oil composition is based on a mixture of hydrocarbons, and hence present affinity for hydrophobic sorbent. However, the composition of used motor oil has been changed through thermal degradation and contamination from generated waste in the engine. This process leads to the formation of low-molecular weight compounds and oxidation products [43]. Recently, Guilharduci et al. [44] showed that introduction of hydrophilic groups on SB improve the oil sorption from engine washing wastewater. Besides, it was also verified that surfactant present in wastewater affect the motor oil sorption.

The biomass acylation is well established in the literature, and the method is based on the reaction of hydroxyl groups (–OH) of the fiber surface with acyl groups (RCO–). Considering that the reaction with fiber is a heterogeneous reaction, not all hydroxyl groups is esterified,

and a nonuniform product may be obtained [32, 35, 38, 45]. In the case of SB, the acetylation with acetic anhydride should introduce the methyl group and increase hydrophobicity at surface. Other acylating agents such as maleic [46], phthalic [9], and succinic [16] anhydrides have been used to introduce carboxylic groups on fibers and increase its surface hydrophilicity.

Another class of coupling agents recognized as efficient to promote the fiber hydrophobicity or hydrophilicity are the silanes. They have been extensively used to modify the surface of natural fibers to produce composites with thermoplastics or thermosets [47]. The aminosilanes, as the aminopropyltriethoxysilane (APS), are the most reported coupling agents for natural fibers. Recently, APS was successful used to modify the SB surface in order to improve its hydrophilic properties, and increase its oils sorption capacity [44].

Despite the great demand for treatment of effluents containing used oil, there is a scarcity of literature on this problem. In this study, this subject is explored in order to verify the potential of SB fibers for oil wastewater treatment. The effect of several coupling agents over SB capacity for sorption of used motor oil is investigated as well as the influence of surfactant in this process.

2.1. Methodology

2.1.1. Reagents

Acetic anhydride (99%), maleic anhydride (96%), phthalic anhydride (99%), succinic anhydride (97%), and aminopropyltriethoxysilane (APS) (97%) were purchased from Sigma-Aldrich and used without further purification. The other analytical grade reagents were obtained from Merck (Brazil) and were used as received. The used motor oil used in this study was obtained from Retifica del Rei (São João del Rei, MG, Brazil). SB was supplied by Cachaça Coqueiro (Nazareno, MG, Brazil). The bagasse was washed repeatedly with distilled water to remove all the dirt particles. The washed fiber was then dried in an oven (Q314M, Quimis) at 60°C for 24 h under a flow of air. It was subsequently ground and sieved through 30 mesh sieves (TE648, Tecnal). The resulting natural fiber was designated as SBN and was used as the starting material to produce the modified SB.

2.1.2. Fiber modification

Thermal treatment of SBN at 200°C for 24 h was performed to obtain the sample SB-200. The SBN acylation was obtained based on previous reports [44]. Briefly, the fiber was firstly soaked in 10% NaOH solution (using a ratio of 1 g/1 mL) at 0°C for 2 h. The bagasse was then washed with Milli-Q water until neutral pH and dried at 60°C for 24 h. Following this procedure, 70 g of the cleaned bagasse was placed in a 1 L round-bottom flask containing 300 mL of acylating agents and 200 mL of acetic acid. The mixture was acidified by adding 1 mL of H₂SO₄ and maintained under agitation for 24 h at 60°C. At the end of this period, the solution was filtered and the product was rinsed first with ethanol and then with water until the pH reached around pH 7.0. After this procedure, the sample was dried at 60°C for

24 h. The acylating agents used were acetic anhydride, maleic anhydride, phthalic anhydride, and succinic anhydride, which produce the samples SB-Acet, SB-M, SB-P, and SB-S, respectively.

The procedure of SB silanization was based on previous report [44]. Basically, the SB is firstly washed with Milli-Q water at 60°C until no color was observed in the washed water, next the fibers are mixed with APS, using a 5/2 mass ratio of SBN/APS, and dispersed into 400 mL of acetone. The suspension was placed in a bottle with glass spheres and left on a roller-conveyor (TE500/1, Tecnal) for 24 h at 200 rpm. The excess of reagents were Soxhlet-extracted with acetone for 24 h. Subsequently, the fiber was dried in an oven at 60°C for 24 h, under a flow of air. The result sample was denominated as SB-APS.

2.1.3. Fibers characterization

Fourier transform infrared (FTIR) spectra were obtained in the range 400–4000 cm⁻¹, using a Perkin-Elmer 1720 FTIR spectrometer. The samples were mixed with KBr (Merck, spectroscopy grade) in an approximate ratio of 10/1. The resulting mixture was pressed into pellets and the spectra were acquired using 300 scans with resolution of 4 cm⁻¹. Determination of C and N contents was carried out with a Thermo Fisher Scientific, Flash 2000 Analyzer.

2.1.4. Sorption experiments

Sorption test was performed using a synthetic used motor oil/water mixture with an oil concentration of 10.0 g L⁻¹. The used motor oil sorption was determined by immersing 1.0 g of fiber in 100 mL of the solution of oil/water mixture. After 24 h, the sorbent was removed, and dried for 24 h at room temperature. The oil sorption was calculated using the following equation:

$$Q = \frac{m_f - m_i}{m_i} \quad (1)$$

where Q is the oil sorption capacity (g/g), m_f is the total mass of dry sorbent (g) after sorption, and m_i is the mass of dry sorbent before sorption (g). The water sorption was determined by the same procedure but using only water.

The experiments of oil sorption were also carried out in solutions containing sodium dodecylbenzenesulfonate in a proportion of 0.05–0.30% in the dispersion of used motor oil/water. The surfactant concentration was determined following the methodology established by the American Public Health Association (Water Environment Federation & APHA 2005).

2.2. Results and discussion

Table 4 summarizes the samples prepared in this study. Results from elemental analysis of nitrogen and carbon in each sample are also presented in **Table 4**. It is noticed that the highest content of nitrogen obtained for SB-APS in relation to SBN fiber, which is attributed to amino groups from APS.

Sample	Treatment	Elemental analysis	
		N%	C%
SBN	<i>In nature</i>	0.18	45.7
SB-200	24 h at 200°C	0.28	48.2
SB-Acet	Modified with acetic anhydride	<i>Not detected</i>	45.1
SB-M	Modified with maleic anhydride	0.12	44.8
SB-P	Modified with phthalic anhydride	0.14	46.1
SB-S	Modified with succinic anhydride	<i>Not detected</i>	43.1
SB-APS	Modified with aminopropyltriethoxysilane	2.15	42.5

Table 4. SB-based sorbents produced in this study.

The modification introduced on the SBN was evaluated by FTIR and results are shown in **Figures 3** and **4**. For SBN spectra, four peaks are clearly observed between 3600 and 3307 cm^{-1} characteristic of stretching vibration of hydroxyl groups, which are associated with lignocellulose structure. Upon acylation with acetic anhydride, the intensity of —O—H absorption band decrease because of hydroxyl groups reduction after reaction. In the same region of spectra, for SB-M only a diffuse band, centered at 3387 cm^{-1} , is observed, and can be associated to hydrogen bonding in the hydroxyl groups. Similar profile is observed for SB-P and SB-S, in which, it is possible to identify two peaks less intense, at around 3600 and 3186 cm^{-1} , also attributed to hydrogen bonding in the hydroxyl groups. These results confirm the modification of surface, which decrease the free hydroxyl groups at fiber surface. Successful esterification can be supported by three important absorption band around 1736, 1367, and 1242 cm^{-1} , correspondent to the stretching vibration of carbonyl groups (C=O), C—H stretching, and C—O stretching characteristic of ester molecules. These bands can be clearly observed for SB-Acet, SB-S, and SB-M, however, with less intensity for SB-P, which can be associated with possible lower acylation obtained for this sample.

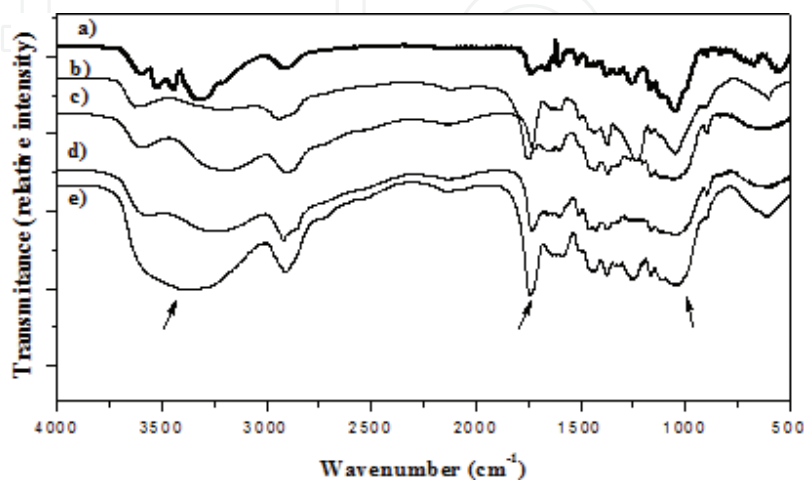


Figure 3. FTIR spectra of (a) SBN, (b) SB-Acet (c) SB-P, (d) SB-S, and (e) SB-M.

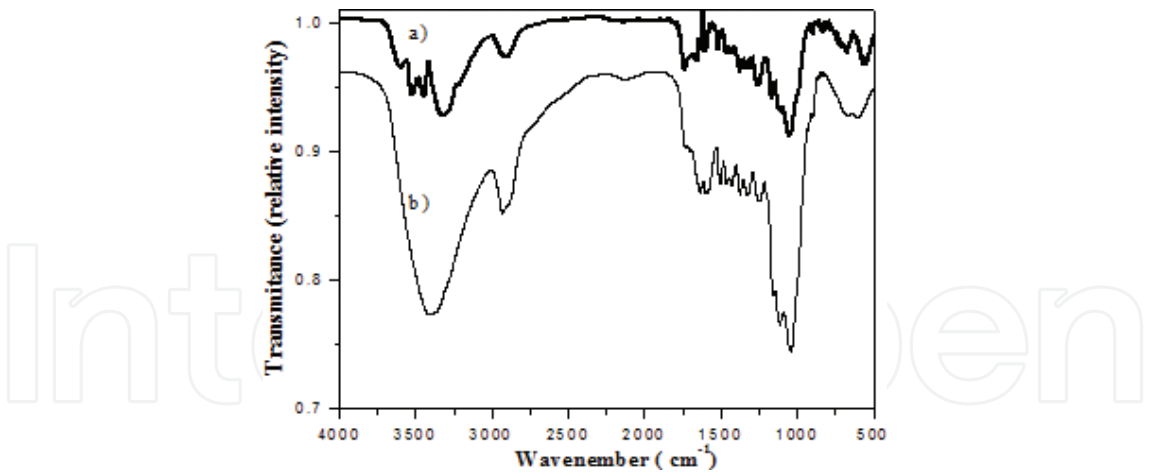


Figure 4. FTIR spectra of (a) SBN and (b) SB-APS.

For sample SB-APS, the FTIR is very similar to SBN (Figure 4). The characteristics of absorption bands of —Si—O—Si— at 700, 1030, 1145, and 1187 cm^{-1} overlapping with groups present in the biomass surface hinder its identification [47]. However, in the region of 3600–3000 cm^{-1} , the APS modification leads to emerging of strong band, centered at 3398 cm^{-1} , attributed to hydrogen bonding from hydroxyl groups. This behavior suggesting that hydrophilic groups (—NH_2) from APS could be interacting with free hydroxyl groups.

The effect of SB modifications over oil and water sorption capacity can be observed in Figure 5. It is possibly observed that samples SB-APS, SBN, and SB-200 show higher affinity with water and also with used motor oil. This result suggests that the introduction of hydrophilic groups on SBN improve the used motor oil sorption. Guilharduci et al. [44] showed that used motor oil presents more affinity to hydrophilic surface than for crude oil. As previously discussed in the introduction, this behavior can be attributed to the chemical differences between used motor oil and new motor oil. Used motor oil can contain sludge, metal residues, and various

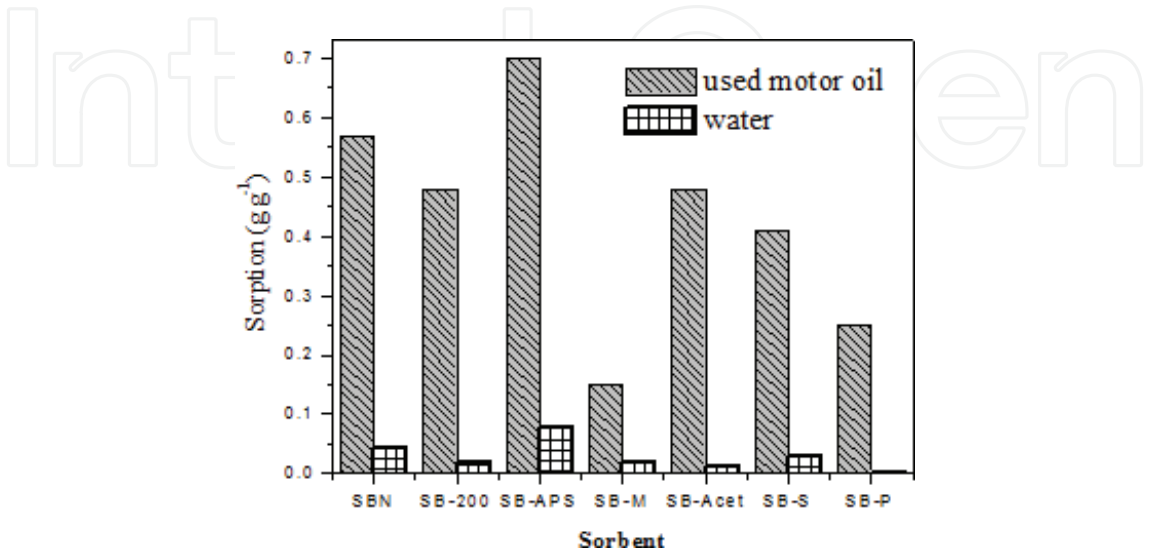


Figure 5. Oil and water sorption capacity for SB samples.

other contaminants. Therefore, the presence of polar amino end groups (NH_2) from silane structure favors the interaction with the constituents of the used motor oil. In contrast, the acylation of SBN was not effective to improve affinity with the constituents of used motor oil resulting in decreased adsorption capacity.

In spite of the higher oil sorption obtained for SB-APS (0.71 g g^{-1}) in relation to SBN (0.60 g g^{-1}), the difference was about 15%, which is relatively small. It is important to take in account that the fiber modification with silane groups is a costly and complex process. Considering the abundance, low cost and efficiency of SBN, this material presented the best cost benefit for use as sorbent of wastewater of used motor oil.

Previous studies showed that the surfactants in used motor oil wastewater can affect its sorption by natural fibers [44]. Surfactants are widely used in various industrial processes for oil recovery, because of their physicochemical characteristics for emulsification, dispersion, and solubilization [48]. They have the ability to reduce the oil-water interfacial tension, increase the capillarity, and change the wettability of the adsorbent surface. Then, in order to evaluate the impact of surfactants on the SBN sorption capacity, batch experiments were carried out using an anionic surfactant, sodium dodecylbenzenesulfonate, in a dispersion of oil (1.0 g)/water (0.1 L). The results are summarized in **Figure 6(a, b)**.

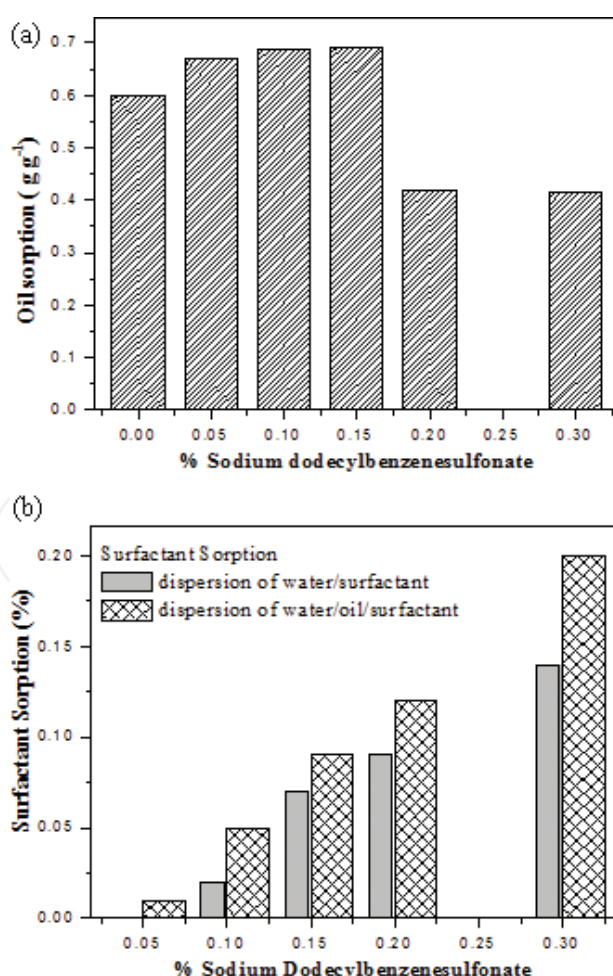


Figure 6. (a) The effect of anionic surfactant in the oil sorption and (b) surfactant sorption in dispersion with and without oil.

The used motor oil sorption shows slight increase (13%) in low concentration of surfactant, and afterward, the sorption decreases almost 42%. When the surfactant concentration increases, the hydrophobicity of fiber in aqueous solution decreased as a result of lower interfacial energy between water and fibers. In parallel, the oil-water interface is improved although micelles or microemulsions formation, which probably affects the oil sorption by SBN and improve surfactant interaction with the fiber. In this condition, the surfactant sorption is likely increased by SBN surface. This suggestion can be evaluated by the analyses of surfactant sorption in the water dispersion with and without oil. The results of these experiments are depicted in **Figure 6(b)**. Based on this result, it is noticed that surfactant sorption not only increases when its concentration increases but also is enhanced in the oil/water dispersion.

3. Conclusion

In this chapter, the great potential of SB in the natural and chemically modified form is shown to be applied as sorbent material for oil removal from aqueous medium. The performance of SBN to remove used motor oil from wastewater was around 0.6 g g^{-1} . This value increases around 13% in the presence of surfactant at 0.05–0.15% in water/oil dispersion. SB also showed efficient sorption of anionic surfactant in water dispersion with or without oil. The results support the use of SB as a natural sorbent as a substitute for commercial synthetic oil sorbents in the treatment of wastewater of used motor oil.

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