

Study of Mandacaru (*Cereus jamacaru* DC), in natura and modified by microemulsion, as a biosorbent for diesel oil

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ABSTRACT. Biosorbents have emerged as an alternative to remove organic contaminants because they are natural products with a high availability, low cost, biodegradability, and good sorption capacity. The Mandacaru (*Cereus jamacaru* DC) was investigated as sorbent of diesel, and compared with the Mandacaru modified with microemulsions. The sorbents were also submitted to a physico-chemical characterization for FT-IR, TG/DTG, MEV and CHN-O. The granulometry range 500-150 μm of the Mandacaru particulate, this was the granulometry range chosen as the standard one, being used in the modification and sorption tests. The unclassified Mandacaru presented a medium sorption capacity of the diesel oil of 1.61 g g^{-1} . Whereas the Mandacaru modified with a microemulsion comprised of direct micelles (oil/water) showed a remarkable increase in the sorption capacity of 80-111% (3.08 to 3.54 g g^{-1}).

Keywords: Biosorbent; natural fiber; microemulsion; diesel oil S-10.

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Introduction

The remediation of spills and leaks of oil and its derivatives from water and soil, as well as relieving their environmental impacts, has been a constant concern for environmental organizations in Brazil and globally because of the high mobility and toxicity of hydrocarbons. The persistence of these pollutants in the environment causes continuous contamination by monoaromatic hydrocarbons, polycyclic aromatic hydrocarbons, and total petroleum hydrocarbons. The presence of these hydrocarbons can generate problems for several years and even decades (Rengasamy, Das, & Karan, 2011; Alegbeleye, Opeolu, & Jackson, 2017). Aromatic hydrocarbons are toxic at low concentrations and have detrimental biological effects, mutagenicity, and carcinogenicity. In particular, benzene and benzo(a)pyrene are a major environmental concern because they can be injurious to public health, especially when supply sources are affected (Ramalho, Aquino Sobrinho, Anjos, Dantas, & Silva, 2014; Rengarajan et al., 2015; Alegbeleye et al., 2017). In this context, several techniques have been proposed and used for cleaning and recovery of areas contaminated by oil spills, such as physical methods (Crick, Bhachu, & Parkin, 2014), chemical methods like *in situ* burning and solidifiers (Mullin & Champ, 2003; Rosales, Suidan, & Venosa, 2010; Sundaravadivelu, Suidan, Venosa, & Rosales, 2016), and bioremediation (Bamforth & Singleton, 2005; Crisafi et al., 2016). Among the physical methods, sorption has been widely used in oil leakage because it is simple and efficient (Wahi, Chuah, Choong, Ngaini, & Nourouzi, 2013; Boni, Oliveira, Souza, & Souza, 2016; Patowary, Pathak, & Ananthakrishnan, 2016; Yin, Zhang, Liu, Li, & Wang, 2016).

Among the physical methods, synthetic organic sorbents, such as polypropylene fibers and polyurethane foams, are the most commercially used in cases of oil spills because of their excellent oleophilic and hydrophobic properties (Zhang & Seeger, 2011; Gu et al., 2014; Wang, Zheng, & Wang, 2014; Wu et al., 2014; Xue, Cao, Liu, Feng, & Jiang, 2014). Despite the favorable properties of synthetic sorbents for oil removal, their low biodegradability is a major disadvantage. Most of these sorbents end up in landfills or are incinerated, which produces garbage and waste that are a source of pollution or make the remediation process expensive. Therefore, the use of biosorbents has emerged as an alternative method for the removal of organic contaminants because they utilize biomasses that are low cost and of great abundance, such as wood sawdust, leaf reject, sugarcane bagasse, coconut fiber, rice hull, and others. In addition to the sustainability of the method, they utilize biodegradable materials (Li, Zhu, Li, Na, & Wang, 2009; Rocha,

Nascimento, Campos, Silva, & Duarte, 2012; Li et al., 2013; Singh, Kendall, Hake, & Ramkumar, 2013; Bazargan, Tan, Hui, & McKay, 2014; Dong, Wang, & Xu, 2014; Chai et al., 2015; Dong, Xu, & Wang, 2015; Chen, Xu, Wang, Cao, & Sun, 2016; Oliveira et al., 2016; Patowary et al., 2016; Pintor, Vilar, Botelho, & Boaventura, 2016).

Brazil is one of the global leaders in raw materials for biomass production because a large portion of its territory is still covered by native forests. Among Brazilian biomes, the caatinga, a biome specific to Brazil, is one of the most remote and lesser studied biomes. The raw materials of its several plant resources have a vast potential in products for many industrial sectors, including nanotechnology (Rodal & Nascimento, 2006; Rocha et al., 2012; Nepomuceno, Santos, Oliveira, Glenn, & Medeiros, 2017). Among the cacti found in the Brazilian semiarid region, the Mandacaru (*Cereus jamacaru* DC) is an alternative biomass that can be used as a biosorbent. Several studies highlight the versatility of Mandacaru through its use as nutritive forage, food, fuel, in the field of technology, civil construction, ornamentation, and for many medicinal and industrial applications, with an emphasis on medicinal and forage use (Silva, 2015).

In this study, was investigated the sorption capacity of Mandacaru *in natura* and when modified by microemulsions for diesel oil.

Material and methods

Materials

The oil sorption experiments were conducted with S-10 diesel oil provided by Petrobras (Guamaré, Brazil). The physical and chemical properties of S-10 diesel oil are listed in Table 1. Mandacaru (*C. jamacaru* DC.) was collected in Caiçara de Rio dos Ventos, Brazil (latitude: -050° 45' 37 and longitude: -350° 59' 54). The chemicals used, butan-1-ol (Vetec, Rio de Janeiro, Brazil), toluene (Vetec, Rio de Janeiro, Brazil), gasoline (Natal, Brazil), lubricant oil (15w40, Rio de Janeiro, Brazil), vaseline (Vetec, Rio de Janeiro, Brazil) and n-hexane (Merck, Darmstadt, Germany), were of analytical grade reagent quality with a 99% purity. Water used for the experiments was from the municipal water supply. The nonionic surfactant nonaethylene glycol monododecyl ether (Alkonat-L90) used in the microemulsion systems was supplied by Oxiteno (São Paulo, Brazil).

Mandacaru preparation

The Mandacaru stems were cut into uniform pieces by removing only the ribs, and were then dried in an oven (105°C) for 24 hours. The pieces were subsequently ground in a knife mill and sieved on a set of Tyler-series sieves (Natal, Brazil) (500, 150, 75, 63, and 45 µm). The granulometric range of 500 to 150 µm was selected as the standard granulometry for the sorption tests because of its high mass percentage.

Subsequently, the Mandacaru (CJDC) was modified with microemulsion by soaking 8 of CJDC in 100.0 g of microemulsion, which was the required quantity for soaking the entire material. The suspension was mixed with a mechanical stirrer (715, Fisatom, São Paulo, Brazil) at 500 rpm and 25°C for 3 min. After that, the modified Mandacaru was dried in an oven at 60°C for 24 hours, rotulated, and stored in high-density polyethylene recipients.

The microemulsion region was obtained by means of the titration methodology (Dantas, Dantas Neto, Moura, & Oliveira, 2017). Inside borosilicate test tubes (15 mL), different proportions between the oil phase (n-hexane) and cosurfactant/surfactant phase (butan-1-ol/Alkonat-L90 = 0.5) were weighed. In the sequence, each of these mixtures was titrated with the aqueous phase (distilled water), until a turbidity in the solution appeared. The micellar compositions were chosen in the microemulsion region oil/water (O W⁻¹, µE1) for promoting Mandacaru modification.

Table 1. S-10 Diesel oil specifications.

Parameter	Method	Value
Visual Aspect	D 4176	Clean
Color ASTM (none)	D 1500	0.5
Total Sulfur (mg kg ⁻¹)	D 5453	7.2
Specific Mass at 20°C (kg m ⁻³)	D 4052	827.9
Kinematic Viscosity at 40°C (mm ² s ⁻¹)	D 445	2.789
Waters and Sediments (volume, %)	D 2709	< 0.050
Flashpoint (°C)	D 93	69.0
Electrical Conductivity at 28°C (pS m ⁻¹)	D 2624	132

Characterization

Functional group analyses of the CJDC and of the modified CJDC- μ E1 were performed with attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy using a Shimadzu IRAffinity FT-IR spectrometer (Shimadzu Corp., Kyoto, Japan), over the scanning range of 700 to 4000 cm^{-1} and at a resolution of 4 cm^{-1} .

Thermogravimetric (TG) and differential thermogravimetric analyses (DTG) measurements were done with a simultaneous thermal analyzer system (TA Instruments, New Castle, USA), under a nitrogen atmosphere (50 mL min^{-1}). Seven milligrams of each sample were heated from 30 to 900°C at a heating rate of 10°C min^{-1} .

The elemental composition (C, H, N, and O) of the Mandacaru was determined using a 2400 Series II CHNS/O element analyzer (Perkin Elmer, Waltham, USA). The oxygen content was determined by subtracting the percentages of the C, H, and N from 100%.

The surface morphologies of the samples were examined using scanning electron microscopy (SEM) (Leo Evo 40XVP, Carl Zeiss, Oberkochen, Germany) at an acceleration voltage of 15 kV.

Sorption experiments

For the sorption experiments followed the method ASTM F716-09 (American Society for Testing and Materials [ASTM], 2009) and ASTM F726-12 (ASTM, 2012). Initially, investigate the effect of granulometry on sorption in Mandacaru *in natura*, and for the 63-75; 150-500 and > 500 μm granulometries, for 60 min.

Typically, 0.5 g fiber was placed in 5 mL of diesel oil at room temperature. After sorption measurement at 5, 20, 40, 60, and 1440 min. (24 hours), the excess oil drained for 5 min. in nylon filter. All

of the experiments were performed in triplicate. Although a simple technique, the results of this method are reliable and the measurements have low standard deviations. The same technique has been used by other researchers (Haussard et al., 2003; Annunciado, Sydenstricker, & Amico, 2005; Lim & Huang, 2007; Karan, Rengasamy, & Das, 2011; Bazargan, Tan, & McKay, 2015; Ngaini, Noh, & Wari, 2018).

The sorption was calculated as the ratio of sorbed material to dry sorbent mass (S_0), sorption = $(S_t - S_0)/S_0$, where S_t is the total mass of sorbed samples. Thus, sorption is given in unities of g oil/g dry sorbent, being the sorbent the mandacaru and mandacaru modified.

Statistical analysis

The experimental data were submitted to analysis of variance (ANOVA) with significance level of 5%. The software Past and graphical plot were used in the statistical analysis.

Results and discussion

Granulometry

Figure 1 shows the results of the granulometric classification, which demonstrated a heterogeneity profile in the distribution of the granulometry. The granulometry range 500-150 μm obtained the largest percentage of the Mandacaru particulate. Therefore, this was the granulometry range chosen as the standard one, being used in the sorption tests. Nevertheless, preliminary sorption tests were carried out to investigate the effect of granulometry on sorption.

The sorption analysis in the three granulometries 75-63; 500-150 and > 500 μm , showed that the mean sorption of the diesel oil for the Mandacaru sorbents, for each granulometry, presented a significant difference in the sorbent versus granulometry interaction ($F = 1284.7$; $p < 0.05$). Table 2 presents the results regarding different granulometric ranges that were obtained over a period of 60 min., where one can notice that a reduction of granulometry on the Mandacaru particulate caused an increase in the oil sorption capacity of 78%.

Therefore, these results confirmed the importance of controlling the granulometry, as it has been reported by Oliveira, Gomes, Oliveira, and Leão (2016), Karan et al. (2011), and Annunciado et al. (2005) that sorption is directly dependent on the available surface area per gram of sorbent.

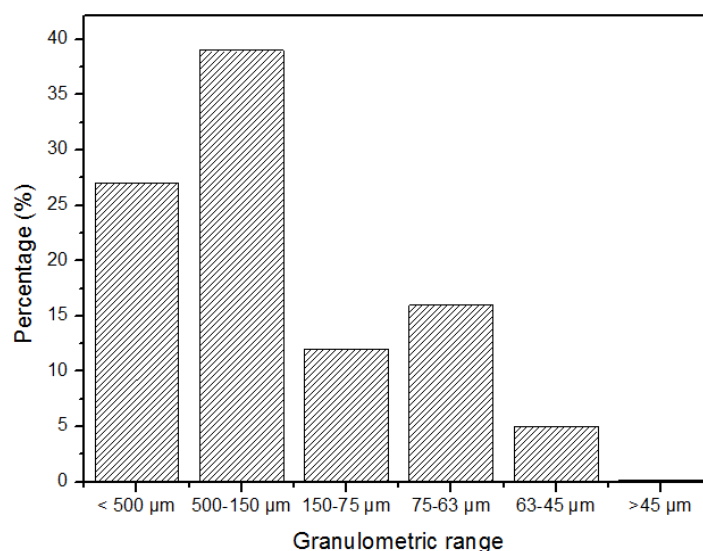


Figure 1. Granulometric distribution of the mandacaru particulate.

Table 2. S-10 Diesel Oil Sorption for the Different Granulometric Ranges of Mandacaru.

Granulometry	S (g g ⁻¹)	Average S (g g ⁻¹)	RSD (%)
75 to 63 μm	1.26	1.25 ± 0.02	1.65
	1.26		
	1.22		
500 to 150 μm	1.62	1.61 ± 0.06	0.68
	1.60		
	1.61		
> 500 μm	0.89	0.88 ± 0.02	2.12
	0.89		
	0.86		

RSD – Relative Standard Deviation.

ATR-FTIR analysis

The ATR-FTIR spectra of the unmodified (CJDC) and modified Mandacaru CJDC-μE1 are presented in Figure 2. The FT-IR spectrum of the CJDC had a peak at 3376 cm⁻¹ that was assigned to O-H stretching vibration of the adsorbed water molecules on the surface of the material (Boni et al., 2016; Lv, Xia, Tang, & Pu, 2017). The peaks at 2926 and 2854 cm⁻¹ corresponded to C-H vibrations, specifically aliphatic CH₂ and CH₃ (Likon, Remškar, Ducman, & Švegl, 2013; Boni et al., 2016; Lv et al., 2017), while the peaks at 1630 and 1734 cm⁻¹ were characteristic of the presence of conjugated and unconjugated carbonyl groups (C = O), respectively. These peaks likely originated from carboxylic acids and ketones from hemicellulose and lignin (Adebajo & Frost, 2004; Barka, Ouzaouit, Abdennouri, & Makhfouk, 2013; Zhang, Yang, Wu, Yuan, & Sun, 2014). The band appearing at 1000 to 1050 cm⁻¹ was attributed mainly to carbohydrates in the cellulose and lignin, including C–O–C, C–O, and C–O–(H) stretching vibrations, groups from glycosidic linkages, lignin precursor monomers, guaiacyl groups, and syringyl groups (Barka et al., 2013).

After modification of the Mandacaru with microemulsion, the intensity of the peaks at 2854 and 2926 cm⁻¹, corresponding to C–H stretching vibrations, were enhanced. This confirmed the interaction of the tensioactive surfactant (ethoxylated lauryl alcohol – Alkonat-L90) with the Mandacaru surface.

Elemental (CHNO) analysis

The elemental analysis of the Mandacaru *in natura* (CJDC) revealed the following composition: 34.63 ± 1.56% carbon, 5.00 ± 0.17% hydrogen, 1.80 ± 0.04% nitrogen, and 58.58 ± 1.70% oxygen. The high carbon content in the biosorbents increased the hydrophobicity and consequently the affinity for the organic phase (Ribeiro, Smith, & Rubio, 2000; Ribeiro, Rubio, & Smith, 2003; Annunciado et al., 2005; Li et al., 2009).

SEM analysis

Figure 3 shows SEM micrographs of the unmodified CJDC and after modification with microemulsion, under different magnifications. The morphological analyses of the unmodified CJDC by SEM revealed rough

and uneven surfaces (Figure 4c and d). However, the SEM images of CJDC- μ E1 demonstrated noticeable alterations because of the modification by microemulsion, and showed a porous structure with round pores on the surface.

TG and DTG analysis

Thermogravimetric curves (TG/DTG) for the CJDC and CJDC- μ E1 are presented in Figure 5. Six weight loss events were observed in the TG/DTG curves for the unmodified CJDC. The first thermal event was mainly mass loss from free water elimination (humidity), while the second one corresponded to the mass loss of linked water because of the hydrophilic property of lignocellulosic materials (Manfredi, Rodríguez, Wladyka-Przybylak, & Vázquez, 2006; Yao, Wu, Lei, Guo, & Xu, 2008). The third and fourth thermal events showed accentuated weight loss and were attributed to the thermal degradation of chemical structures in the Mandacaru, such as hemicellulose ($T_{\max} = 271.75^{\circ}\text{C}$) and cellulose ($T_{\max} = 314.47^{\circ}\text{C}$) [Manfredi et al., 2006; Yao et al., 2008; Sebío-Puñal, Naya, López-Beceiro, Tarrío-Saavedra, & Artiaga, 2012; Boni et al., 2016]. The fifth thermal event ($T_{\max} = 460.29^{\circ}\text{C}$) was attributed to the thermal degradation of some functional groups in the cellulose residue and the burning of more resistant substances (Boni et al., 2016; Wong, McGowan, Bajwa, & Bajwa, 2016). In the sixth event ($T_{\max} = 650.58^{\circ}\text{C}$), carbonization of most of the substances had already occurred, with only inorganic chemical elements remaining in the ash.

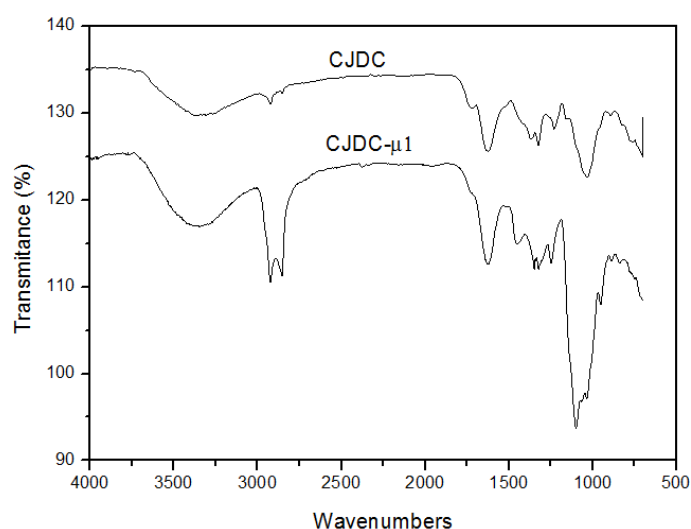


Figure 2. FT-IR spectra of the CJDC and CJDC- μ E1.

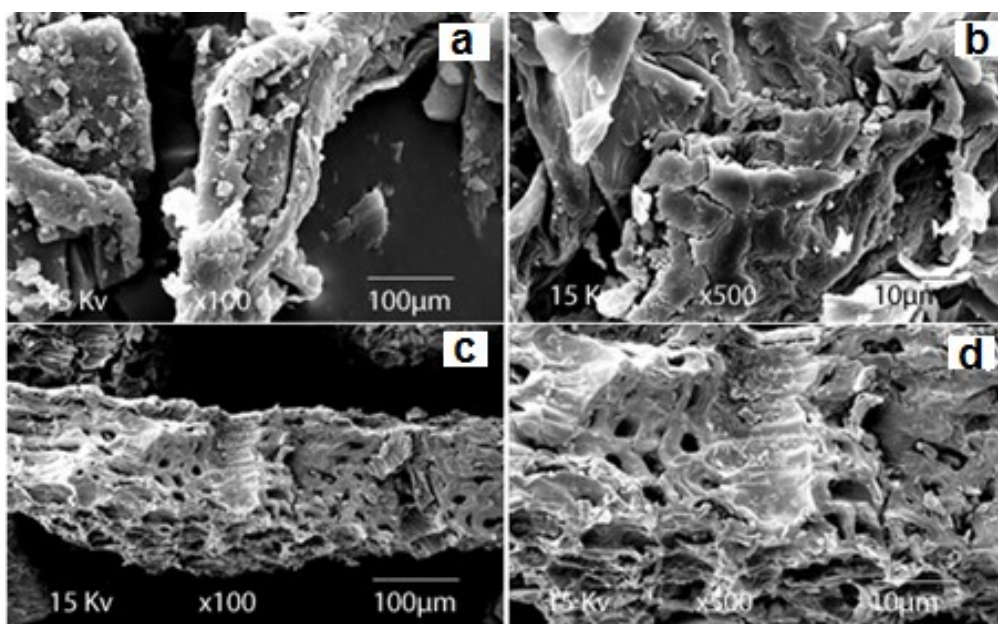


Figure 3. SEM images of the (a) CJDC at 100x, (b) CJDC at 500x, (c) CJDC- μ E1 at 100x, and (d) CJDC- μ E1 at 500x.

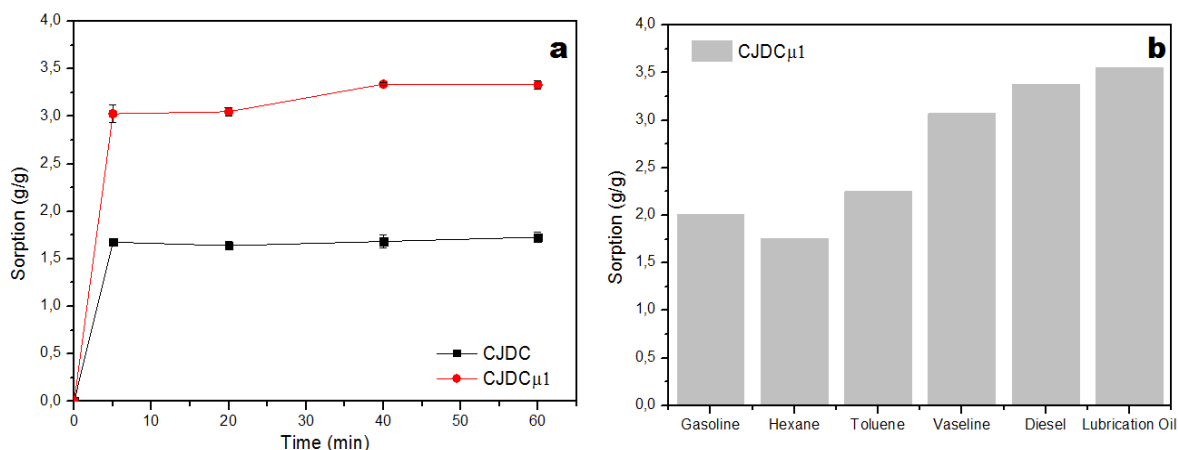


Figure 4. (a) Sorption capacity of CJDC and CJDC-μ1 for S-10 diesel oil under dry conditions. (b) Sorption efficiency of CJDC-μ1 in various oils for 60 min.

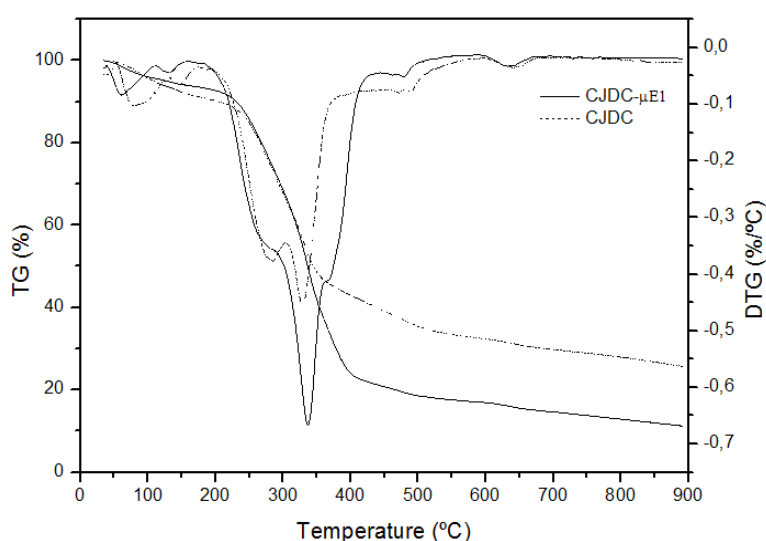


Figure 5. (a) TG and (b) DTG curves for CJDC and CJDC-μE1.

The DTG curves (Figure 4) for the CJDC-μE1 showed seven thermal weight loss events. The first and second thermal events were related to water loss; the first one corresponded to free water (humidity) and the second one corresponded to more strongly linked water (adsorbed water), as was observed for the unmodified CJDC. Similar for CJDC, the third event corresponded to the start of holocellulose degradation, but the curves were differentiated because of the modification of the Mandacaru surface with the microemulsion. This was evidenced by the presence of peak near 283.3°C, which corresponded to weight loss of 19.1%. It was also possible to observe in these TG/DTG curves over the temperature range of approximately 150 to 450°C, increases in the weight loss of 72.8% for CJDC-μE1, while a weight loss of only 49% was noted for the unmodified CJDC. The difference in weight loss in this region may have been related to the mass of the Alkonat-L90 impregnated on the surface of the Mandacaru, which showed efficiency in the process of modification and/or interaction with the surface.

The analysis of the TG curves indicated a decrease in the final residue weight at 900°C for the Mandacaru modified with the microemulsion, with values of 11% (CJDC-μE1). These values were smaller than that for Mandacaru *in natura* (28%). The decrease in the final weight percentage may have been related to the removal of inorganic compounds during treatment of the CJDC with the microemulsions, which resulted in a lower ash content.

Oil sorption capacity

The method of testing sorption capacity of sorbents in pure oil medium is useful to determine the maximum amount of oil sorbed by a particular sorbent. The results of the study shown in Figure 5a the sorption capacity of the CJDC and CJDC-μE1 obtained in pure diesel oil medium and with varying sorption

times. The oil sorption capacity had small variations over time, from 5 to 1440 min., with an average sorption value of $1.61 \pm 0.06 \text{ g g}^{-1}$ (CJDC) and $3.38 \pm 0.09 \text{ g g}^{-1}$ (CJDC- μ E1). After 5 min. of contact, the CJDC and CJDC- μ E1 showed 97 and 87% of the maximum sorption capacity, which indicated that most of the sorption occurred in the first few minutes. Oils with a lower viscosity tend to migrate rapidly to the interior or surface, which may have been an explanation for the observed behavior. In a study with sisal fiber, Annunciado et al. (2005) reported a behavior similar to that observed with the CJDC and CJDC- μ E1 and reached 80% of the maximum sorption capacity in 5 min.

The maximum sorption capacity value of CJDC- μ E1 was $3.54 \pm 0.08 \text{ g g}^{-1}$. This increase may have been related to the increase in the surface area and porosity, as was previously observed in the SEM micrographs of CJDC- μ E1 (Figure 5c and d). According to Karan et al. (2011), the porosity affects the oil diffusion rate and oil retention in the sorbent material. When the porosity is higher, the oil sorption capacity is greater.

Table 3 shows that the microemulsion promoted the greatest increase in the sorption capacity of the Mandacaru. After modification of the CJDC, a major increase in its sorption capacity was observed, with an increase of 111% when compared with that of the unmodified CJDC. Also investigated was a sorption capacity in gasoline, hexane, toluene, vaseline e lubricat oil, during 60 min. of contact with the modified Mandacaru CJDC- μ E1. The results obtained are presented in Figure 5, where it was verified that the sorption capacity of the CJDC- μ E1 decreases for the oils of low viscosity and increases for the oils of higher viscosity. Being the lubricant oil with the highest sorption capacity for CJDC- μ E1.

A comparative study

In general, it is difficult to make a direct comparison about the oil absorbing capacities between different oil sorbents, because of different model oils and weight of an oil sorbent used. However, a comparison with studies by Oliveira et al. (2016) and Oliveira, Leão, Caraschi, Oliveira, and Gonçalves (2011) with diesel oil, as summarized in Table 4, gave an indication that the Mandacaru modified had a superior or comparable oil-absorbing capacity to most of oil sorbents.

The sorption results obtained for the CJDC- μ E1 with microemulsion were considered satisfactory because they were comparable to those obtained by the study on the sorption capacity for *Salvinia* sp. in petroleum jelly (3.6 g g^{-1}) (Ribeiro et al., 2003) and commercially peat (PeatSorb®), especially in the case of the maximum sorption capacity obtained for the CJDC- μ E1 ($3.54 \pm 0.08 \text{ g g}^{-1}$). Ribeiro et al. (2003) tested the sorption capacity of a commercially available peat (PeatSorb®) that is used by the company Petrobras to remediate oil spills, and obtained a value of $2.7 \pm 0.2 \text{ g g}^{-1}$ with an oil viscosity of 237 cP. In this study the sorption capacity of CJDC- μ E1 was observed to be 29% higher than that of the PeatSorb®. It is worth noting that the sorption capacities of the PeatSorb® and *Salvinia* sp. were tested using oils with higher viscosities than the oil used in this work (2.789 cP). The oil sorption inside of the sorbent is inversely proportional to the viscosity; an increase in the viscosity causes an increase in the sorption capacity because the oil remains inside of the pores longer and does not leak during draining. Therefore, it was expected that the PeatSorb® and *Salvinia* sp. would have a lower sorption capacity for diesel.

Table 3. Sorption Capacity of CJDC- μ E1.

Time (min.)	Sorption (g g^{-1})	RSD (%)	Sorption Increase (%)
5	3.08 ± 0.10	3.13	80
20	3.10 ± 0.04	1.37	82
40	3.39 ± 0.01	0.41	99
60	3.38 ± 0.05	1.34	98
1440	3.54 ± 0.08	2.21	111

RSD – Relative Standard Deviation.

Table 4. S-10 Diesel Oil Sorption for the Different Granulometric Ranges of Mandacaru.

Biosorvente	Sorption (g g^{-1})					Média
	5 min.	20 min.	40 min.	60 min	1440 min.	
CJDC ¹	$1,63 \pm 0,04$	$1,59 \pm 0,04$	$1,63 \pm 0,07$	$1,68 \pm 0,04$	$1,53 \pm 0,01$	$1,61 \pm 0,06$
CJDC- μ E1 ¹	$3,08 \pm 0,09$	$3,10 \pm 0,04$	$3,39 \pm 0,01$	$3,38 \pm 0,04$	$3,54 \pm 0,09$	$3,38 \pm 0,09$
Turfa ^{2,3}	1,26	1,17	1,21	1,26	1,28	1,23
Curauá ²	1,10	1,18	1,27	1,32	0,74	1,12
Coco ³	$0,68 \pm 0,22$	$0,66 \pm 0,08$	$0,70 \pm 0,09$	$0,73 \pm 0,08$	$0,98 \pm 0,16$	$0,76 \pm 0,16$

¹This study; ²Oliveira et al. (2016); ³Oliveira et al. (2011).

Conclusion

The results of the CJDC- μ E1, indicated this material is promising for applications as a sorbent for diesel oil. Under the experimental conditions, it was observed that 1 g of CJDC- μ E1 can sorb 3.54 g of diesel oil. The microemulsion under study showed efficiency in the process of modification of Mandacaru and increased the porosity and surface area. The FTIR evaluated the incorporation of surfactants, which was determined by the increase in the degree of C-H and C-O bonds. Concerning the droplet the SEM results showed that the CJDC- μ E1 exhibited morphological alteration on the Mandacaru surface, which improved the oil/biosorbent interaction.

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