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Improved sorbent for the removal of hydrocarbons spilled in water

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Oil contamination of ecologically sensitive aquifer systems is a major global environmental problem. Depending on their availability, there is a trend to seek sorbent materials based on natural fibers, for their high adsorption capacity and biodegradability. The objective of this work was to obtain a sorbent material, by carbonizing sugarcane bagasse chemically modified to clean the water contaminated by oil. Tests were carried out to determine the sorption capacity of the carbonized material as a type II sorbent according to ASTM method F 726-17. The optimal particle size for which the sorption capacity was greater, was above 2.0 mm. The sorbent obtained was tested on water contaminated with crude oil, and a comparative evaluation was made between this material and a commercially existing one. As a result of the experiments, very similar values were obtained between the properties of both sorbent materials. Thus, it is concluded that the carbonized bagasse material can be used as hydrocarbon sorbent material.

KEYWORDS

hydrocarbon sorption, natural fibers, modified sugarcane bagasse, carbonization, sorption capacity

Introduction

The contamination of ecologically sensitive aquatic systems and soils with oil is a very significant environmental problem. The amount of oil and its derivatives that impact the environment due to spills, leaks, natural outcrops, offshore production, transportation, industrial wastewater, and urban runoff is estimated at more than eight million tons. Only between 2010 and 2013 an estimated of 22,000 tons of oil were released into the marine environment. The affectations on the vital activities of living beings can vary from minimal to severe depending on the location and magnitude of the spills. Contact with hydrocarbons and their consumption through the food chain affects both the biota and human beings (Allan, 2005; Oliveira et al., 2021; Thakur and Koul, 2022).

The spills of oil and its derivatives have caused substantial environmental damage to the ecosystems in the areas where the incidents have occurred, which are generally located near areas of importance—due to their ecological fragilities, such as fishing areas, national parks, wildlife, watersheds, and urban settlements. For this reason, it is necessary to improve the strategies and techniques to face these events and eliminate or

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collect the spilled hydrocarbons, as much as possible. For this purpose, measures have been proposed, which include, among others, mechanical collection or the use of sorbents and dissolution to facilitate their biodegradation (IFC - World Bank Group., 2016; Tan et al., 2021; Zhu et al., 2022).

Great attention is currently being paid to the use of sorbents due to the possibilities they provide for the renewal of the aquatic environment compared to other mechanical and chemical oil spill elimination techniques. The combination of environmental regulatory changes, combined with efforts to introduce sorbents for the elimination of oil spills into the market, has generated an interest in cutting-edge technologies within the field of sorbents. In this sense, the industry aims to reduce and/or eliminate spills with lower costs/benefits, and therefore, attention is paid to materials with wide diffusion in the environment, including natural fibers such as wood chips, tree bark, coconut shells, rice straw, sugarcane bagasse (SCB), and water lily, among others, for their high adsorption capacity and biodegradability and that are discarded in different agro-industrial processes (Crisafully et al., 2008; Kalmykova et al., 2014; León and Díaz, 2019; Zamparas et al., 2020; Maia Cardoso et al., 2021; Oliveira et al., 2021; Nassar et al., 2022).

Among the natural fibers mentioned above, SCB occupies a prominent place due to the volume generated from it, its attractive cost, and its properties as a sorbent derived from its hydrophobicity, high porosity, and silica content (Ortiz González et al., 2006; Brandão et al., 2010; Sarker et al., 2017; Gorgulho et al., 2018; Cavalcante et al., 2019; Ajala et al., 2021). SCB is used both in its original form and modified by some treatments and has been successfully used in laboratory and pilot-scale trials, showing effectiveness comparable to that of commercial products (Armada et al., 2009; Chung et al., 2011; Rincones Poyer et al., 2015; Behnood et al., 2016; Guilharduci et al., 2016; Díaz-Díaz et al., 2018).

One of the ways to transform SCB to use it as a sorbent is its carbonization (Del Angel et al., 2022; Prasannamedha and Senthil Kumar, 2022). The carbonization process of vegetable raw materials is highly demanded and well described in the literature (Azat et al., 2012; Chai et al., 2019; Madhubashani et al., 2021; Pecchi et al., 2022; Seow et al., 2022; Yeletsky et al., 2022). This method is one of the main ones to produce various carbon materials. The compound obtained during the carbonization of plant materials retains its original structure and makes it possible to obtain various compositions of carbon with other substances.

The objective of this work was to obtain a sorbent material, by carbonizing bagasse chemically modified to clean the water contaminated by oil.

Materials and methods

In this work, the methods reported in the literature were used to classify by particle size, to determine the sorption capacity of the material statically and dynamically on the surface as a type II sorbent according to ASTM method F 726-17, defined as loose sorbents that are unconsolidated particulate material and are handled with scoops or similar equipment (ASTM, 2019a), in water and soil, as well as study their buoyancy and ash content (Said et al., 2009; Kudaybergenov et al., 2012; Martinez Nodal et al., 2014).

A sample of SCB was crushed using a homogenizer, then passed through a set of sieves with cell sizes between 0.5 and 3.0 mm, to separate it into fractions and determine the sorption capacity of the material as a type II sorbent for each. The static buoyancy was assessed for the different fractions by placing them in separate containers filled with water and crude oil (31 ^{0}API). The exact time of analysis was set at 12 h. Both determinations were performed according to the above-mentioned ASTM method F 726-17.

The ash content of the SCB was determined as an indicator for carbonizing the material, according to ASTM method D 2584-18 (ASTM, 2019b). The samples (5 g by triplicated) were dried before the test. It was then placed in a muffle oven, where combustion took place at temperatures of 200, 400, and 600°C (Torres-Pérez and Soria-Serna, 2015).

According to method ASTM F 726-17 dynamic conditions test of the absorbent material obtained is conducted in a moving aqueous phase (hydrophilic conditions) containing hydrocarbons (oleophilic conditions). A sorbent sample (5 g) in the presence of oil (3 ml) was placed in a container filled with water (6 replicates), which was agitated with a wave width less than 1/3 of the total capacity. A real wave was modeled, and the dynamic buoyancy of materials was determined. Dynamic buoyancy is an index that allows characterizing the behavior of the sorbent material in real conditions of its use in the aquatic environment.

For the chemical treatment of the SCB sulphuric, hydrochloric, nitric, and phosphoric acids were used. Three concentrations of each acid: 5, 10, and 20% solutions were prepared. In these solutions, the SCB was immersed for 30 min. The SCB samples treated with the acids were further carbonized.

A comparison was made between the physicochemical properties of the sorbent based on SCB with a sorbent based on carbonized rice husk, already recognized and promoted on the market for different uses (Bazargan et al., 2014; Daffalla et al., 2020). The carbonized rice husk was subjected to the same tests as the SCB.

TABLE 1 Fractional composition of SCB.

Fraction composition, mm	Fraction contents, %, mean values	Sorption capacity, g/g
>3.0	31.4	2–3
2.0–3.0	30.6	2–3
1.0–2.0	17	1–2
0.5–1.0	11.8	1–2
<0.5	9.2	1–2

Results and discussion

Sorption capacity for different particle sizes of SCB

The results of the sorption capacity of SCB as a type II sorbent for each of the fractions obtained are shown in Table 1. As seen, the maximum sorption capacity is obtained for fractions of 2.0 to 3.0 mm and those greater than 3.0 mm. In addition, the absorption capacity is between 2 and 3 g/g sorbent, since the crushing cannot achieve an adequate particle size, and the specific surface area is reduced (Machado-Soberanes et al., 2012).

During the experiment, it was observed that the crude adheres to the surface of the particles of the SCB instead of its absorption, qualitatively this is due to the absence of enough pores with an adequate size in the structure of the source material.

Determination of the buoyancy of SCB

Figure 1 shows how the different fractions in the water and hydrocarbon containers behaved during the evaluation of the static buoyancy of SCB over time. The time of analysis was set at exactly 12 h.

Figure 2 shows that the cane bagasse is located below the meniscus of the water surface and at the bottom of the vessel. With a slight movement, more than 10% of the particles sank so the material fails the static buoyancy test. The change in color of the water and a distinctive sweet smell are due to the release of polysaccharides, indicating that the SCB is hydrophilic and does not adhere to the surface of the water.

Given the low absorption capacity concerning other materials and the observed buoyancy of the material, a carbonization process was considered, to improve its initial characteristics.

Ash content

According to the results, 2.5% of the ashes were obtained. It can be said that the entire organic component burns, including cellulose, which has an ignition temperature of 275°C and auto ignition of 420°C·

Chemical treatment of bagasse

From the tests carried out, it was found that the natural bagasse has little absorption capacity for hydrocarbons, which is increased with a previous chemical treatment.

The acid solution was poured into a container and the impregnated bagasse was placed in a dryer. When treated with sulfuric acid, the bagasse took a brown tone, sunk quickly and small bubbles appeared on the surface of the water. During treatment with hydrochloric and nitric acids, no obvious signs of reaction were observed, only a small brown hue appeared in the bagasse. In the treatment with phosphoric acid, no changes were observed and, in both cases, the bagasse was below the meniscus of the liquid in the vessel, with a tendency to subsidence.

During the chemical treatment, it occurs a surface delignification of bagasse and it increases the adsorption capacity by the additional formation of pores on the surface, joint to the inner section of the fibers (oil-bagasse chemical bonds). Delignification produces on the material, accessible residual polysaccharides and it obtains a material most effective for bio disposal.

Charring of bagasse

In the first stage of the carbonization process, which is at 200°C, evaporation of the aqueous acid solution occurred, and the impregnated sample lent itself well to evaporation. This is necessary to create pores, because when the material is in contact with high temperatures, water begins to heat up, then boils and evaporates. After 15 min, the temperature in the chamber increased to 400°C.

In the second stage, at a temperature of 400°C, the outer bagasse particles were carbonized. In 15 min, part of the bagasse was transformed into coal. The third stage, at a temperature of 600°C, was necessary to completely carbonize the remaining organics in the sample. As a result of carbonization in a closed muffle oven, at three temperature conditions, a black material was obtained (Figure 3).

A black or dark gray dispersed poly powder was obtained that resembles silicon-carbon sorbent materials, which externally closely resembles charred rice husk (Figure 4). This is

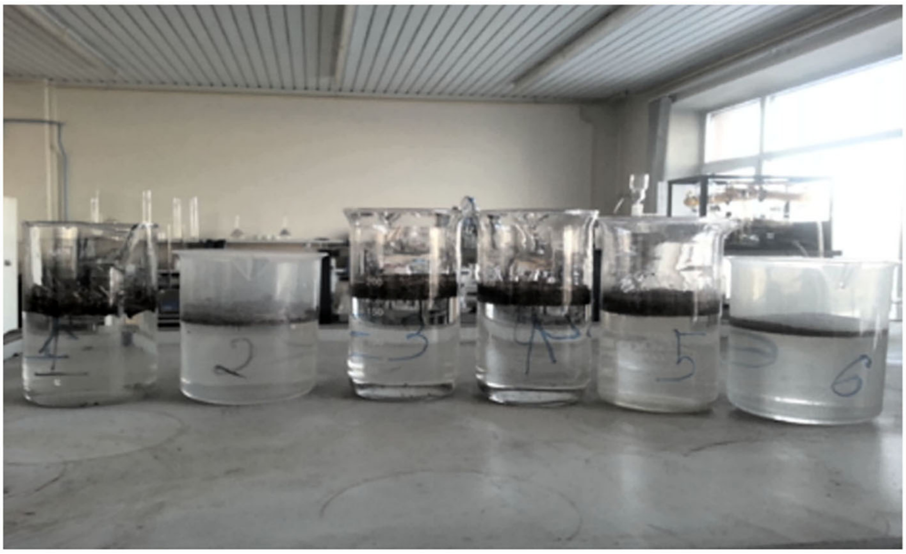

FIGURE 1
Static buoyancy test (start).

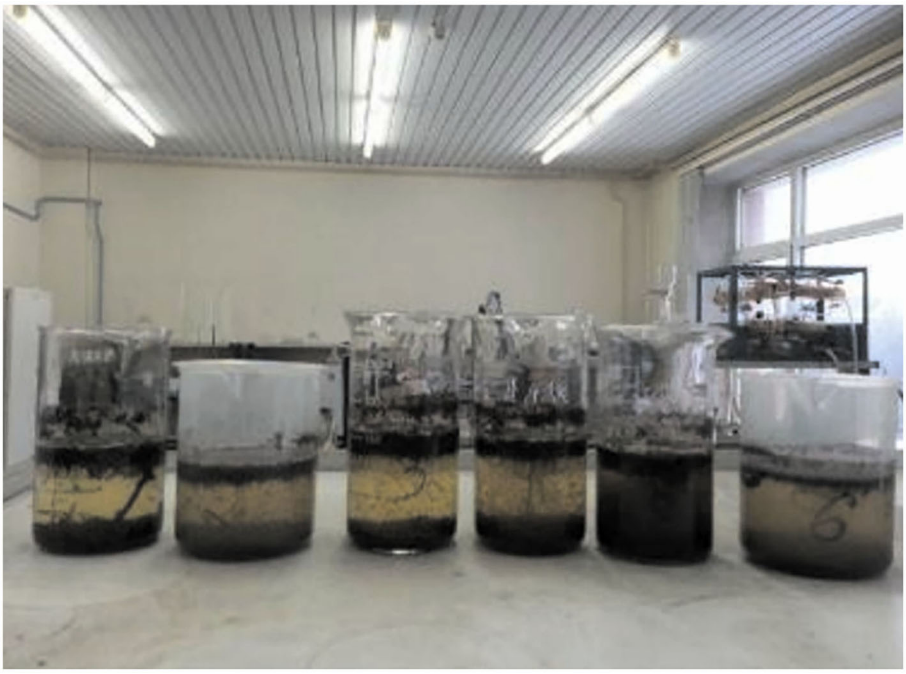

FIGURE 2
Static buoyancy test (12 h).

a sorbent material already tested and marketed, and according to the manufacturer, belongs to the class of organic mineral sorbents, is non-toxic, and is very effective in eliminating oil spills, petroleum products, and other organic substances (Kudaybergenov et al., 2012).

Absorption capacity of carbonized SCB

After carbonization, fractions of less than 0.5 mm, 0.5–1.0 mm, and 1.0–2.0 mm joined to form a single structure that could be removed from the crucible without damage. Fractions

FIGURE 3
Charred bagasse.

FIGURE 4
Charred rice husk.

TABLE 2 Sorption capacity of charred SCB.

Size fraction, mm	Adsorption capacity, g/g
>3.0	8–9
2.0–3.0	8–9
1.0–2.0	4–5
0.5–1.0	2–3
>0.5	1–2

TABLE 3 Sorption capacity of some experimental and commercial sorbents.

Sorbent$^{\text{reference}}$	Oil product	Sorbent capacity g g^{-1}
Raw SCB[a]	Machine oil	8.9
Raw SCB[b]	Crude oil	7.6–8.1
Raw SCB[a]	Synthetic effluent of crude Petroleum	6.65
Acetylated SCB[b]	Crude oil	8.8–9.2
Acetylated SCB[a]	Machine oil	11.4–16.5
Na(OH)/H_2O_2 treated SCB[c]	Light and medium crude oil	5.1–5.9
Biochar from rice husk[d]	Crude oil	3.03–9.2
Biochar from maple wood[d]	Crude oil	3.8–6.2
Saw dust[e]	Oil spill	4.5–8.5
Lessorb-extra[e]	Oil spill	8
Activated carbon AU-3[e]	Oil spill	4.5–10
Sorboyl S8[e]	Oil spill	8–10
Peat[f]	Crude oil	6.7–7.3
Straw[f]	Crude oil	4.6–5.5
Benelftesorb-extra[g]	Crude oil	2.8–4.9
Sphagsorb[g]	Crude oil	4.0–5.6

[a] Gorgulho et al., 2018; [b] Oliveira et al., 2021; [c] Díaz-Díaz et al., 2018; [d] Madhubashani et al., 2021; [e] Galblaub et al., 2016; [f] Paulauskiene et al., 2014; [g] Téllez Carmona, 2007.

larger than 2.0 mm during carbonization did not join and formed a bulk material. According to the methodology F 726-17 (ASTM, 2019a), which was used for the determination of the absorption capacity, data were obtained for each fraction indicated in Table 2 for the SCB treated with phosphoric acid (the other samples, treated with nitric and hydrochloric acids, gave poorer results, data not shown).

The results show that the absorption capacity of hydrocarbons increases by increasing the fraction size but becomes constant after the size of 2 to 3 mm. Thus, the greater hydrocarbon absorption capacity of the carbonized material is obtained with this fraction.

The carbonized SCB fraction of 2 to 3 mm in size was selected for further studies on light petroleum, with a density of ∼0.8367 g/cm^3, and the carbonized rice husk was also evaluated.

The selected fraction of carbonized SCB is effective for the collection of hydrocarbons in water, according to the absorption capacity obtained. Although comparisons are difficult since the activity of each sorbent depends on its grain size, modifications of its surface, buoyancy, etc., in Table 3 the results of the SCB herein obtained are compared with the absorption capacity of some experimental and commercial sorbents.

It can be seen that the effectiveness of the carbonized SCB obtained is in the same order of magnitude as the products shown in Table 3.

Figures 5, 6 show the charred bagasse during harvesting and after being impregnated with oil.

Buoyancy of carbonized SCB

The charred fraction of 2.0 to 3.0 mm was placed in a container of water and left for 12 h in a static position, noting that it did not sink but was below the lower meniscus of the water surface. This indicates that the material swelled with water, but small air bubbles remained inside it, which did not allow it to descend to the bottom, which is of utmost importance for the collection of the impregnated material.

When exposed to water, the charred rice husk behaves in the same way as the charred SCB. The carbonized rice shell sorbent is kept below the lower meniscus of the water surface and does not sink. However, after 12 h the material is partially saturated with water, according to the water absorption relation obtained in early experiences (Díaz-Díaz et al., 2018).

The buoyancy of sorbents is considered one of the most important factors. High-efficiency sorbents must keep their floating capacity even when they are saturated with water or oil. However, some very light sorbents, which could remain on top of spilled oils, could also cause some difficulties in sorbing oil (Hoang et al., 2021).

When the static buoyancy of the material on a hydrocarbon spilled on the surface of the water was tested, the carbonized SCB showed excellent results. The sorbent in the base SCB, which falls on the hydrocarbon, sorbs it from the surface of the water and stays afloat due to its lower density of the same.

Static buoyancy was verified for 7 days, during which time the carbonized SCB, as well as the carbonized rice shell sorbent, remained on the water surface and did not sink (Figure 7). In addition, a solid conglomerate formed on the surface of the oil-water and charred bagasse particles (Figure 8). Such property is indispensable, as it facilitates the collection of hydrocarbons captured by emergency response teams in spills at sea.

FIGURE 5
During oil pickup.

FIGURE 6
Impregnated with crude oil.

FIGURE 7
Charred SCB 1.

Dynamic buoyancy of charred bagasse and rice husk

According to the methodology, the material passes the test if it sinks no more than 10% of the weight originally taken. Upon contact with hydrocarbon, the materials formed a strong conglomerate between the oil particles and the sorbent. The conglomerate remained on the surface of the water during its oscillation and did not sink. Hence, both materials passed this test.

Absorption capacity of carbonized bagasse in water for different hydrocarbons

The carbonized bagasse fraction of 2.0 to 3.0 mm was tested to determine the hydrocarbon absorption capacity at the water surface. Hydrocarbon was poured into water, after which the sorbent was placed on the hydrocarbon (Table 4). In parallel, the absorption capacity of carbonized rice husk was evaluated (Table 5) under similar conditions.

As can be seen in Tables 4, 5, the absorption time depends slightly on the molecular weight of the sorbed petroleum product, while for diesel fuel and low molecular weight gasoline, the absorption time increases slightly. This is explained by the spread of such petroleum products on the surface, partial dissolution in water, and the formation of an unstable stain. Medium, high molecular weight, more viscous oil does not spread over the surface and does not form a continuous stain, floating in separate pieces with an average layer thickness of 3.5 mm.

The high capacity of the sorbent for the average oil is explained by its low fluidity. As a result, the conglomerate is formed with the interaction of the surface of the sorbent with the oil, requires no additional processing, and is easily removed from the water surface.

The interaction of the sorbent with fuel oil was also tested, but the sorption is practically not produced without mixing.

Conclusions

Raw SCB does not show good buoyancy or good sorption capacity for hydrocarbon derivatives. This was also observed for the samples treated with acids so a carbonization stage to obtain the sorbent was necessary.

An improved sorbent material has been obtained by carbonizing SCB after chemical treatment with phosphoric acid. The SCB treated with hydrochloric and nitric acids gave inferior

FIGURE 8
Charred SCB 2.

TABLE 4 Sorption capacity of charred SCB in water surface.

Oil product	Layer thickness, mm	Sorption time, min	Sorption capacity, g/g
Gasoline	0.28	1–2	10–12
Diesel	0.35	1–2	10–12
Lube oil	0.47	2–4	9–10
Light crude	0.40	3–6	8–9
Medium crude	3.35	3–6	7–8

TABLE 5 Sorption capacity of charred rice husk in water surface.

Oil product	Layer thickness, mm	Sorption time, min	Sorption capacity, g/g
Gasoline	0.28	5.0	8–11
Diesel	0.35	4.5	8–10
Lube oil	0.47	4.0	9–10
Light crude	0.40	3.5	7–9
Medium crude	3.35	4.0	6–8

results in terms of sorption capacity. Fractions with 2–3 mm size sorb hydrocarbons are more effective.

The improved sorbent material has a comparable absorption capacity with known commercial sorbents, has a high buoyancy in the presence of hydrocarbons, and has a sufficient apparent density (0.11–0.12 g / cm^3) to apply the material using spray guns.

Data availability statement

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

Author contributions

All authors listed have made a substantial, direct, and intellectual contribution to the work and approved it for publication.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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