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A comparative investigation on the separation of crude oil from marine media with the aid of natural fibres

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The present research examines the adsorption characteristics of different types of natural sorbents for the removal of crude oil from sea water. Sorbent characterisation is done using SEM and FTIR analyses. The effect of different adsorption test factors such as contact time, adsorbent weight and initial concentration of crude oil has been investigated to obtain crude oil removal using arrowroot fibre, talipot palm fibre, newspaper, cocopeat and human hair. The maximum oil sorption capacities are found to be 5990 mg/g, 4650 mg/g, 5830 mg/g, 6710 mg/g and 4980 mg/g for the arrowroot fibre, talipot palm fibre, newspaper, cocopeat and human hair respectively. The maximum percentage oil removed are found to be 94.66%, 68.36%, 97.37%, 99.6% and 90.41% for arrow root fibre, talipot palm fibre, newspaper, cocopeat and human hair respectively. Oil adsorbency and water adsorbency are determined and the recycle study has been performed for all the selected natural adsorbents. The design of experiments has been carried out using Box -Behnken method in response surface methodology using Minitab 19.

Keywords: Adsorption Isotherm, Box-Behnken method, Crudeoil removal, Natural sorbents, Response surface methodology

Introduction

Fuels are complex class of compounds which are necessary for the existence of the world. Among that crude oil is a mixture of hydrocarbons which is explored, extracted, transported and shipped from oil wells. This sequencing process results in large or small oil spill in the oceanic environment. The water resources get polluted with this persistent hydrocarbon pollutant. As a result, the living organisms, environment and ecological balance are disturbed to a greater extent. Several living creatures in the water body partially or fully losses its life, and ultimately it results in depletion of non-renewable sources of energy[1]. Spilt oil undergoes a number of weathering processes such as evaporation, oxidation, emulsification, sedimentation, dispersion and biodegradation[2]. There is chance of deposition of heavy crude constituents underwater, near shorelines whereas lighter constituents are dispersed over upper surface of water due to the action of wind, tide, pressure differences etc. Petroleum products persist in the environment for longer time. Therefore, it is important to protect natural resources from the crude oil spills with the aid of external mechanisms.

Oil spill recovery techniques include physical/ mechanical, chemical or biological[3,4]. Oil remediation by physical or mechanical methods such as booms, skimmers, sorbent materials are eco-friendly and cost-effective method than chemical dispersants[5]. Oil spill remediation can be effectively achieved by the technique of adsorption[6]. Adsorption process with the aid of sorbents could be considered as a cost-effective method since they are readily available, environment friendly, reusable and cheap substances[4]. Among the sorbents used, most of them are biodegradable and constitute to the sustainability of the biosphere. On the basis of oil spill scenario, sorbent selection is carried out so that an effective oil remediation from liquid sources is possible[4]. Biomass based sorbents such as banana peel[2,4], luffa[4,19], natural wool fibre[13], mixed leaf residue[14], mixed saw dust[14], sisal[14], coir fibre[14], sponge guard[14], silk floss[14], water hyacinth fibre[15], empty palm fruit bunch fiber[17], cotton byproduct[18,20], human hair[18], recycled cellulose[18], kapok[20],cattail[20] etc. were used in their raw form for the removal of crude oil constituents from water sources. Since these substances are by-products from many agricultural sources, it does not cause any cost issues. The excellent physical, chemical and structural properties of advanced carbon-based materials in their nanoparticle form ensure them to be used as oil

pollutant removal from water sources[5]. Apart from this, there are a few advanced materials like aerogels, foam membranes, inorganic meshes, and surface modified fabrics for the removal of oil from water mixtures[4]. Despite several advantages of sorbent materials, there are a few concerns regarding the availability of raw material, processing techniques used, expense, reuse, recovery and degradation of newly developed substance. The natural fibres mainly consist of cellulose which is the reason for its hydrophilic property. Water absorption may reduce the ability to capture oil molecules into the structure of these fibres.

Adsorption is a mass transfer operation where substance of low concentration is sorbed by certain solids. Such solids having specific properties are known as sorbents. Sorbed substances remain concentrated on or near the surface of the sorbent in the case of adsorption. When the rate of adsorption equal to rate of desorption, adsorption equilibrium will be established.

Experimental Section

Material collection and pretreatment

Crude oil used in the test was obtained from BPCL Kochi Refinery, Ambalamugal. Density of the crude oil was determined using gravimetric method and kinematic viscosity measurement was conducted in a redwood viscometer before the start of the study. Sea water was tapped from Fort Kochi harbour and was used in all the sorption experiments. Salinity and pH of sea water was determined using electrical conductivity method in an electrical conductivity meter and pH meter, respectively.

The naturally available fibres used as sorbents were arrowroot fibre, talipot palm fibre, waste newspaper, cocopeat and human hair. Arrowroot fibre was obtained from household arrowroot powder extraction, talipot palm (Coryphaum braculifera) fibre procured traditionally from a small-scale unit producing powder from this variety of palm, waste newspaper and cocopeat was locally collected, and human hair of Indian origin was collected from a local salon.

Arrowroot fibre, Talipot palm fibre and Cocopeat were washed thoroughly with deionized water to remove foreign substances, dust and impurities present on it. Then they were drained and dried in hot air oven at 80℃ for 8 h for the removal of water content. Fibres were cut to small pieces of 0.5-1 cm, ground and sieved in 2 mm mesh. Waste newspaper was equally cut to small pieces of 0.5-1 cm in dimension and was used throughout the study. Human hair was washed with deionized water and sun dried. All these sorbents were sealed in zip lock bags for the entire study. The photographs are shown in Fig. S1 (Supplementary Information).

Characterization of sorbent

Surface morphology of the sorbents was captured using Scanning Electron Microscope (HRTEM: Joel/JEM 2100). All the cross sections of the sorbent sample were placed on the sample holder and coated with gold using JFC 1600 Auto fine coater machine. The measurements were done at 20 kV accelerating voltage and at 500x magnification. After 2 min of stabilization, the samples were kept on the sputter machine for 60 s. Beam current required was 80 μA. In order to identify the functional groups, present in the structure of adsorbents, FT-IR image was recorded using Fourier Transform Infrared Spectrophotometer (Model: Thermo Nicolet iS50). For conducting FTIR analysis, all the samples were placed over diamond crystal in ATR attachment. The average number of scans taken was 64 per sample in the wavenumber range 4000-400 cm^{-1}.

Oil and water adsorbency test

For performing dynamic degradation test according to ASTM F726_12, about 3 g of sorbent was weighed and put in a beaker placed on a water bath incubator shaker and 100 mL of sea water was added to it. The beaker was allowed to shake for 15 min at 110 rpm. This mixture was allowed to settle for 2 min. After straining for 30 s, weight of wet sorbent was measured[11].

Water adsorbency,

$$WA = Ww / Wi \quad \text{... (1)}$$

The net water adsorbed,

$$Ww = Wwt - Wi \quad \text{... (2)}$$

where W_i is the initial weight of dry sorbent (g) and W_{wt} is the weight of sorbent at the end of dynamic degradation test (g)[11].

To measure oil adsorbency, initial weight of 4 g of adsorbent was dipped in oil for 15 min. After draining the oil for 30 s, sample weight was recorded[11].

Oil adsorbency, $OA = Wo / Wi$ … (3)

The net oil adsorbed,

$$Wo = Wot - Wi \quad \ldots (4)$$

where W_i is the initial weight of dry sorbent (g) and W_{ot} is the weight of sorbent at the end of oil test (g)[11].

Adsorption experiment

Oil spill was artificially created by pouring 5 mL of crude oil to 50 mL of sea water taken in a 250 mL glass beaker. Five such oil spills were simulated and these beakers were kept on water bath incubator shaker at 60 rpm for the corresponding time intervals of 15, 30, 45, 60, 75, 90, 105 and 120 min.

After shaking, the mixture was filtered using a metallic mesh and drained for a period of 5 min. The wet sorbent containing oil particle was weighed using analytical balance with accuracy of 0.001 g. Batch kinetic study was carried out in triplicate at ambient temperature (30±2°C) and the mean of these results were recorded.

Sorption process is a phenomenon in which sorbate attaches to the surface or interior of a sorbent. Sorption capacity is the amount of sorbate taken up by the sorbent per unit mass of the sorbent and it is expressed in terms of mg of oil /g of sorbent. The quantity of oil sorbed at time t is determined by Eq. 5.

$$\text{Oil sorption capacity, } Sc = \frac{Wt - Wi - Ww}{Wi} \quad \ldots (5)$$

where W_i is the initial weight of dry sorbent, (g)W_t is the weight of wet sorbent containing oil and water after the test (g) and W_w is the quantity of water wetted by the sorbent (g)[6].

The percentage of oil removed is determined by Eq. 6 as given below.

$$\text{Percentage oil removal} = OR(\%) = \left[\frac{Wt - Wi - Ww}{Wo}\right] * 100 \quad \ldots (6)$$

where Wo is the initial weight of oil (g)

Mechanical squeezing method was practiced for the recovery of oil from the wet sorbent and the same could be reused in the successive tests.

Results and Discussion

Analysis of crude oil is shown in Table 1. According to the results of density and viscosity of crude oil, this type of crude oil comes under medium oil type[11]. Table S1 shows the analysis of seawater.

Characterization of sorbents

SEM Analysis

The surface morphologies of the sorbents were studied using SEM as shown in Fig. 1. Arrowroot fiber, talipot palm fiber and cocopeat were observed as a mixture of rough and smooth fibers whereas newspaper and human hair appeared to be a rough fiber.

FTIR Analysis

In the case of arrowroot fibre which is a polysaccharide, the absorption peak at 3289.87 cm^{-1} is due to polymeric O-H stretch as shown in Fig. 2a. The peak found at 2917.83 cm^{-1} shows methylene C-H

Table 1 — Analysis of crude oil

Crude oil Parameter	Values
Density at 30 ± 2 °C	862 kg/m^3
Specific gravity at 30 ± 2 °C	0.865
API gravity	34
Kinematic viscosity at 30 ± 2 °C	332 cP

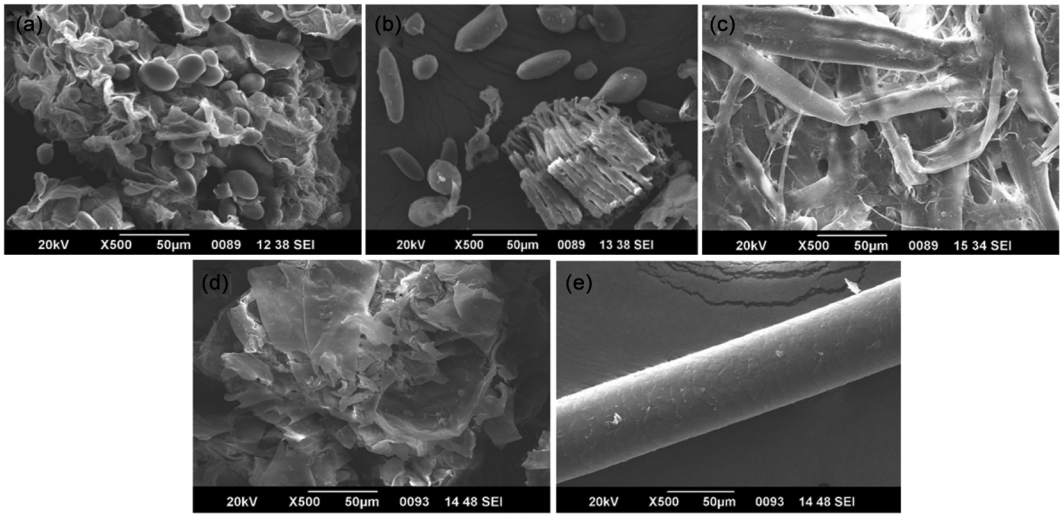

Fig. 1 — SEM images of (a) arrowroot fiber (b) talipot palm fiber and (c) waste newspaper (d) cocopeat and (e) human hair at 50 µm resolution

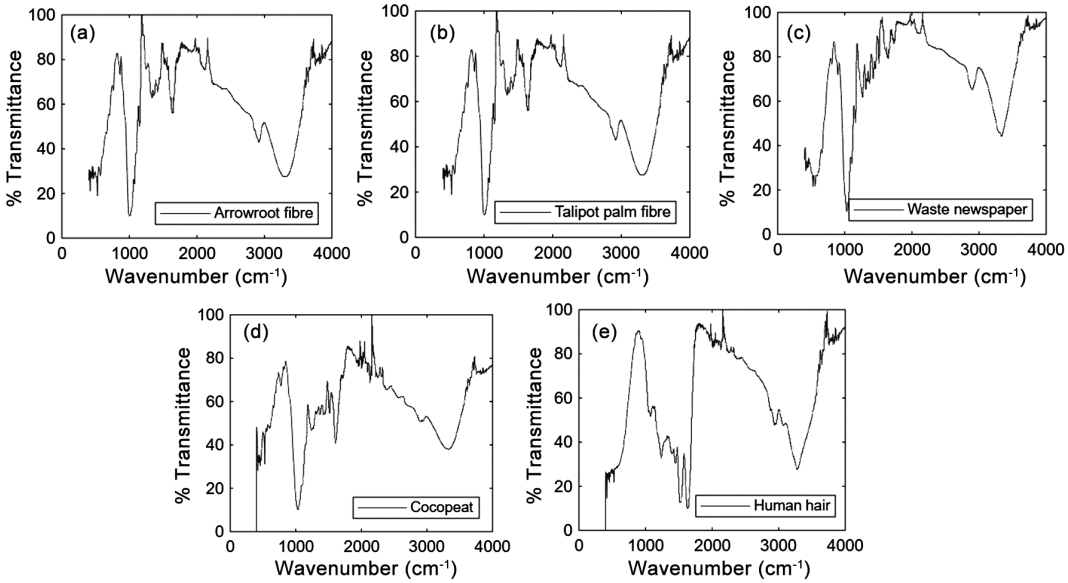

Fig. 2 — FTIR spectra of (a) arrowroot fiber (b) talipot palm fiber (c) waste newspaper (d) cocopeat and (e) human hair

asymmetric stretch whereas alkenyl C-C stretch is observed at 1633.93 cm^{-1}. The peak at 1417.14 cm^{-1} and 1336.77 cm^{-1} are observed as carboxylate group and methyne C-H bend, respectively. The remaining peaks at 1150.47 cm^{-1}, 1000.5 cm^{-1} and 860.2 cm^{-1} correspond to skeletal C-C vibrations[16].

Similarly, for talipot palm fiber (Fig. 2b), due to polymeric OH stretch, there is an absorption peak at 3289.46 cm^{-1} whereas at 2927.76 cm^{-1} and 1634.61 cm^{-1}, there are methylene C-H asymmetric stretch and alkenyl C-C stretch, respectively. The presence of carboxyl functional groups are observed at 1417.77 cm^{-1} and 1374.6 cm^{-1}. Skeletal C-C vibrations are seen at 1242.64 cm^{-1}, 1150.21 cm^{-1}, 1012.78 cm^{-1} and 857.78 cm^{-1}(Ref.16).

FTIR analysis of waste newspaper (Fig. 2c) show absorption peaks at 3335.42 cm^{-1} and 2899.02 cm^{-1} are due to polymeric O-H stretch and methyne C-H stretch, respectively. At 1730.36 cm^{-1} there is absorption peak of aldehyde functional group, at 1646.32 cm^{-1} there is alkenyl C-C stretch and at 1509.62 cm^{-1} there is aromatic ring stretch. The carboxylate functional groups show peaks at 1423.78 cm^{-1} and 1370.33 cm^{-1}. Appearance of peaks at 1263.48 cm^{-1}, 1161.88 cm^{-1}, 1030.64 cm^{-1} and 896.49 cm^{-1} are due to skeletal C-C vibrations[16].

For cocopeat (Fig. 2d), the peak at 3423.6 cm^{-1} is due to hydroxyl group, H bonded O-H stretch and at 2920.63 cm^{-1} is due to methylene C-H asymmetric stretch. Secondary amine, N-H bend is observed at 1617.81 cm^{-1}. Absorption peak for carboxylate is at 1514.16 cm^{-1}. Methyl C-H asymmetric bending is observed at 1451.86 cm^{-1}. At 1112.69 cm^{-1}, 770.27 cm^{-1} and 516.79 cm^{-1} skeletal C-C vibrations are seen[16].

For human hair sample (Fig. 2e), hydroxy group, H bonded O-H stretch is seen at 3277.27 cm^{-1}, methylene C-H asymmetric stretch is observed at 2933.77 cm^{-1}. Secondary amine, N-H bends are recorded at 1633.87 cm^{-1} and 1516.94 cm^{-1}. At 1447.57 cm^{-1} there is an absorption peak of methyl C-H asymmetric bend. Skeletal C-C vibrations are observed at 1235.95 cm^{-1} and 1076.44 cm^{-1}.

Oil and water adsorbency test

The oil adsorbency and water adsorbency of different sorbents are shown in Fig. 3. It is found that cocopeat has the highest oil adsorbency and water adsorbency followed by newspaper, arrowroot fibre, hair and talipot palm fiber. It may be due to higher hydrophobic and oleophilic character for all these sorbents, where cocopeat showed the maximum hydrophobic nature.

Effect of contact time

By using all the selected sorbents, when contact time was varied from 0 to 120 min, oil sorption capacity increases, after which it gradually decreases and becomes stable as shown in Fig. 4. This stable response may be due to the attainment of saturation by the molecules of these sorbents. Oil sorption capacity of 4080 mg/g, 4200 mg/g and 3900 mg/g at 60 min were observed for arrowroot fibre, waste newspaper and human hair, respectively, but at 45 min, talipot palm fibre showed maximum sorption

capacity of 2950 mg/g. The maximum sorption capacity of cocopeat at a contact time of 75 min was found to be 5320 mg/g. Oil removal percentages were determined and it is shown in Table 2.

Effect of sorbent weight

When the sorbent dose increased, there was an immediate rise in the oil sorption capacity and gradually it declines as depicted in Fig. 5. This may be due to the oil molecules occupying the void spaces of porous sorbents leaving behind no more vacant space. As a result, the attachment of oil droplets onto the sorbent surface ceases. With 0.5 g of talipot palm fibre, waste newspaper and cocopeat, the adsorption capacity obtained was 3250 mg/g, 5830 mg/g and 5790 mg/g, respectively. With a sorbent weight of 1 g, the adsorption capacity for arrowroot fibre is 4450 mg/g and for human hair 3900 mg/g.

Effect of initial concentration of crude oil

When the initial concentration of crude oil was varied from 8620 mg/L to 86200 mg/L, the maximum oil sorption capacity for arrowroot fibre and talipot palm fibre was obtained as 5990 mg/g and 4650 mg/g, respectively, at a concentration of 8620 mg/L. For newspaper, cocopeat and human hair, it was obtained as 5630 mg/g, 6710 mg/g and 4980 mg/g, respectively. The results are shown in Fig. 6.

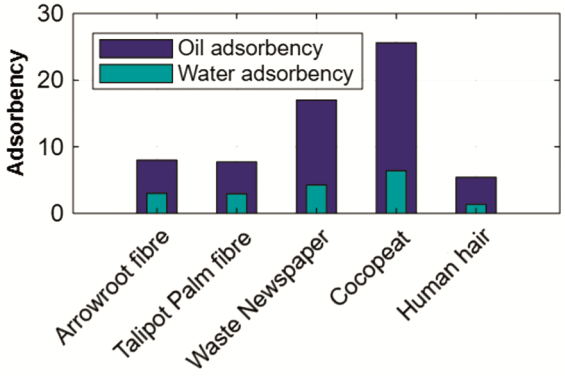

Fig. 3 — Oil adsorbency and water adsorbency

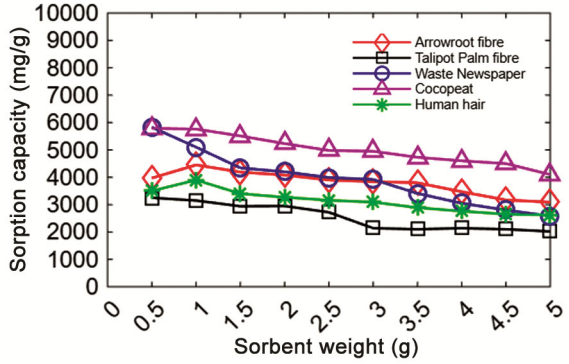

Fig. 5 — Oil sorption capacity *vs* sorbent weight

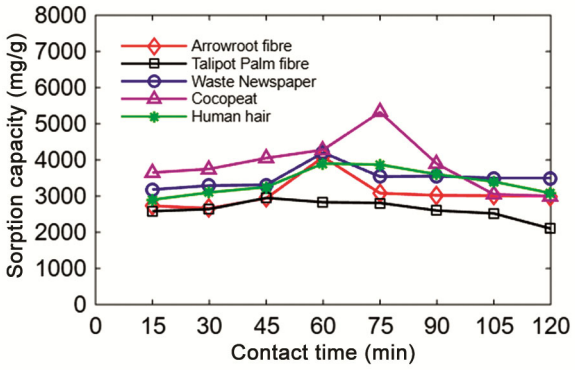

Fig. 4 — Oil sorption capacity *vs* contact time

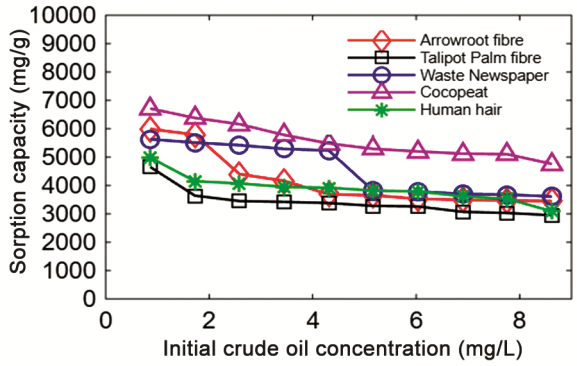

Fig. 6 — Oil sorption capacity *vs* initial concentration of crude oil

Table 2 — Percentage removal of crudeoil using prepared fibres at different contact times

Contact time (min)	Oil removal (%)				
	Arrowroot fiber	Talipot palm fiber	Waste Newspaper	Cocopeat	Human hair
15	63.23	59.77	73.78	84.66	67.24
30	61.75	61.25	76.40	86.90	71.90
45	68.19	68.36	76.86	93.92	75.39
60	94.66	65.66	97.37	99.33	90.41
75	71.53	65.20	82.05	99.60	89.79
90	70.16	60.31	82.32	90.24	83.53
105	69.77	58.47	81.28	70.66	78.89
120	69.64	48.82	81.22	69.61	71.46

Recyclability of sorbent

All the sorbents were tested for their recyclability and reuse by its continuous use for about five successive cycles as shown in Fig. 7. During each cycle of adsorption, desorption was carried out by mechanical pressing for the recovery of crude oil trapped on the surface of the sorbent. Then sorbent was dried before its next use.

Optimization of parameters

In order to prove the established theories, experiments were conducted based on the changes in input and all the outputs are analysed using Box-Behnken method in Response surface methodology using Minitab 19 [Ref.12,21]. The independent variables are contact time, sorbent weight and initial crude oil concentration, while maintaining constant temperature and agitation rate. The maximum and minimum range of variables are 15 min to 120 min for contact time, 0.5 g to 5 g for sorbent weight, and 8620 mg/L to 86200 mg/L for initial crude oil concentration.

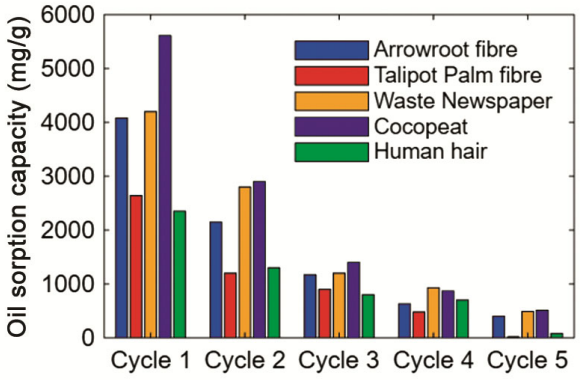

Fig. 7 — Adsorbent recyclability

Surface plots

The surface plots of oil sorption capacities for the selected adsorbents are depicted below in Fig. 8-12. The hold values for the initial crude oil concentration were taken as 47410 mg/L. Surface plots depict how the sorption capacity is related to the varying contact time and sorbent weight by maintaining constant initial crude oil concentration, and then by varying initial crude oil concentration keeping sorbent weight as constant.

Contour plots

Contour plots of these sorbents are shown in Fig. S2-S6 (Supplementary Information). The three continuous variables in contour plots are contact time, sorbent weight and initial crude oil concentration. It depicts the effects of these continuous variables on sorption capacity.

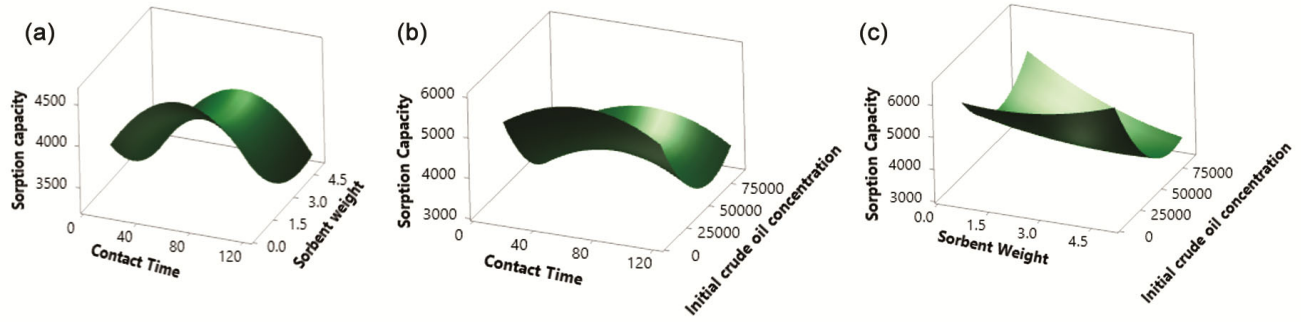

Fig. 8 — Surface plots of Arrowroot Fibre

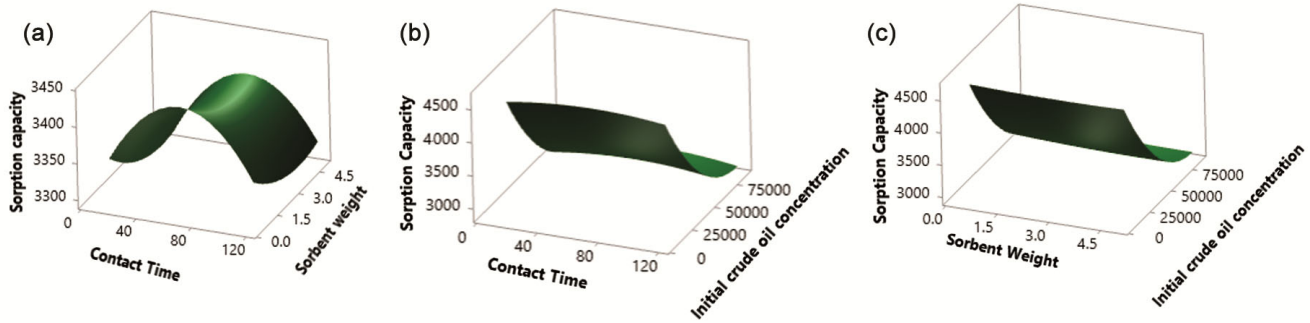

Fig. 9 — Surface plots of Talipot palm fibre

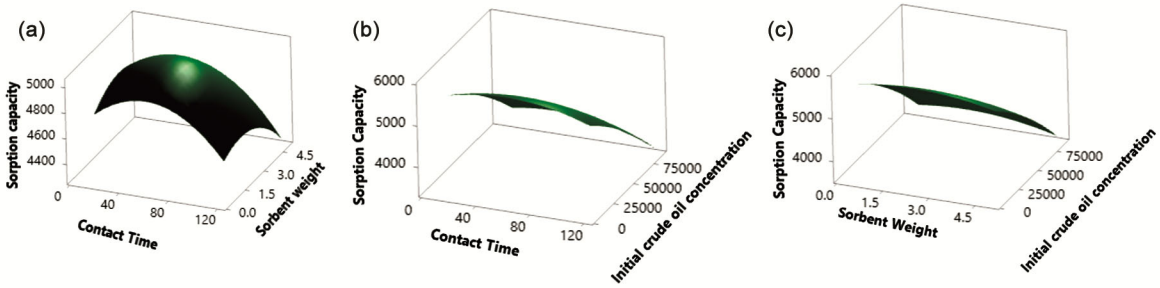

Fig. 10 — Surface plots of Waste newspaper

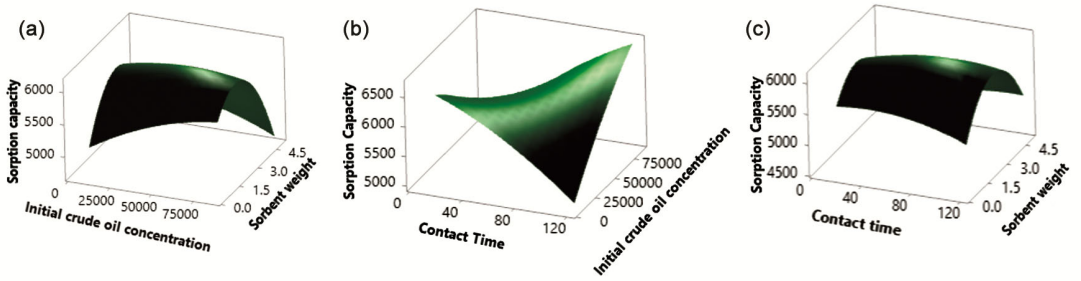

Fig. 11 — Surface plots of Cocopeat

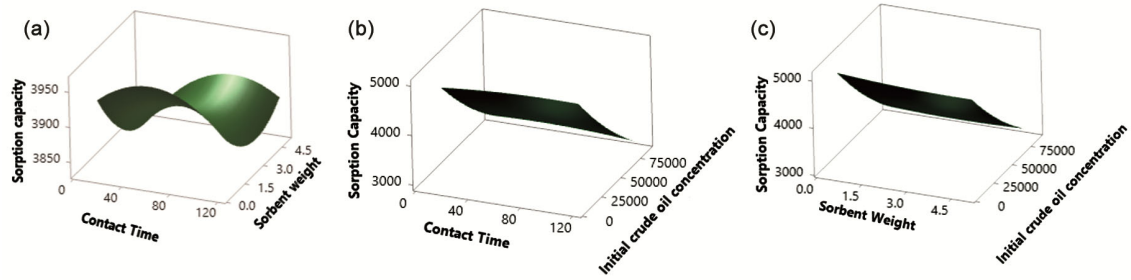

Fig. 12 — Surface plots of Human hair

Conclusion

The present study used raw form of certain fibres as sorbents which are free of chemicals, comparatively easy disposable, and environment friendly. These natural fibres possess hydrophobic property due to the presence of an outer waxy coating. This hydrophobic nature enhances the attraction of oil onto the surface of sorbents. The recycle study confirmed the capability of the adsorbents to be reused for at least five successive cycles. Optimized factors for contact time, sorbent weight and initial crude oil concentration in the case of arrowroot fibre was found to be 68 min, 5g sorbent, and 8620 mg/L, respectively, whereas for talipot palm fibre the corresponding values were found to be 71 min, 0.5 g and 4675 mg/L, respectively. For newspaper, the optimized conditions are 58 min, 2.4 g and 8620 mg/L whereas for cocopeat it was obtained as 120 min, 2.2 g and 86200 mg/L. In the case of human hair adsorbent, the optimum conditions were 89 min, 0.5 g and 8620 mg/L, respectively. The study demonstrates that cocopeat is the best oil adsorbent for marine water among all the selected adsorbents.

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Conflict of Interest

The authors declare that they have no conflict of interest.

Supplementary Information

Supplementary information is available on the website https://nopr.niscpr.res.in/handle/123456789.

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