

DETERMINATIONS OF SELECTED GRAVIMETRIC, FRICTIONAL AND FUNCTIONAL PARAMETERS OF NATURAL CORN STRAW FIBERS AS CRUDE OIL SORBENT ON SURFACE AQUATIC ENVIRONMENT.

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Abstract

Natural and pretreated corn straw fibers (CSF) have been studied and explored as a cheap, readily available and environmentally clean up approach with the sorption of crude oil spills (sediments) on surface water. The proximate compositions with moisture (16.25%), ash content (8.53%), crude fiber (72.12%), cellulose (23.15%), hemicellulose (14.05%) and lignin (45.23%) were determined. The identity of the CSF was ascertained by FT-IR. Five different particle sizes (60,100,150,203, and 305mm) were adopted with the treatment of the selected characteristics. The relationship between the particle size and the bulk density ($R^2=0.9151$), tapped density ($R^2=0.9577$), true density ($R^2=0.9577$), porosity ($R^2=0.0867$), repose angle ($R^2=0.9534$), Carr's index ($R^2=0.0845$), Hausner ratio ($R^2=0.0889$), and OSC ($R^2=0.9963$) were defined. Two paired sample T-tests between the particle size and the bulk density, tapped density, true density,

porosity, Carr's index, Hausner ratio, and OSC retained significant relationships, while the angle of repose was significance in only one direction.

The concluded conditions for the effective adsorption of the CSF were disclosed with particle sizes of 259.1369 to 68.0631mm, 0.4227 to 0.1529g/ml with the bulk density, and 0.5260 to 0.1984g/ml with the tap density, 0.7813 to 0.2935 g/ml with the true density, and 0.4921% with porosity, 1.2067 with Hausner ratio and 19.7003% with the oil sorption capacity.

Keyword: Corn straw fibers, particle size, gravimetric properties, frictional properties, functional properties

1.INTRODUCTION

Crude oil is a natural resource is a bedrock of energies industrially. Meanwhile, there is always an accidental means of polluting both the aquatic and terrestrial ecosystems by the direct discharge of oil spills. The spills must be eliminated and prevented before it becomes emulsified with the water bodies forming sludge. Emulsified oil is exceptionally difficult to clean by ordinary strategies as the oil contains numerous harmful and extremely destructive synthetics to the environment where they can have impacts on living organisms. At present, physical, chemical, and natural treatment techniques are employed for the cleanup and

remediation of contaminated wastewater [2]. Physical adsorption procedures, which connected with hydrophilic materials to expel released oil from water, are considered as the most financial and legitimate technique because of its high retention limit and economy [3]. Meanwhile, the majority of synthetic absorbents usually limited with respect to non-biodegradability, low absorption capacity, poor separation efficiency and high cost of production [4]. Thus, the fabrication of new environmental-friendly oil-absorbing material with a higher absorption ability and lower production cost is required and urgent [4]. Utilizing waste biomass materials as an oil absorbent is of incredible interest for oil-water partition and is turning into a zone of extreme research since they are modest, available, and biodegradable [4]. As of late, super-hydrophobic surfaces have stood out in view of their remarkable properties, including self-cleaning, against staining, waterproof, substance steadiness, and oil recuperation [4]. In principle, development of a natural structure with low surface strength is an imperative parameter for the making of super-hydrophobic surface, which is described by a water contact angle more than 150° [4]. Corn straw fiber, natural and biodegradable biomass from agricultural waste, is typically disposed by incineration within the environments as it raises the level of air pollution. Truth be told, effective use of this waste is indispensable in fathoming and managing the effects of air contamination. Zang et al. (2016) expelled oil from water applying super-hydrophobic/super-oleophobic corn straw yarns with ZnO particles by means of regular impregnation [4]. ZnO particles are deep spheres with a normal measurement of $5 \mu\text{m}$, with hexadecyltrimethoxysilane (HDTMOS) as a hydrophobic modifier. The super hydrophobic/superoleophobic properties of the organized corn straw fiber emerged from the consolidated impacts from the deposition of homogeneous SiO_2 inorganic particles, with normal molecule size around 40–50 nm with sol-gel technique, and the hydrophobic advancement with (Heptadecafluoro-1, 1, 2, 2-tetradecyl) trimethoxysilane [5]. This enables the biomass the capacity to effectively remove oils from water bodies. In view of its intrinsic water-repellency, high absorption capacity, chemical stability, and environmental friendliness, the prepared corn straw fiber float on the surface of the water after absorbing the oil, allowing it

to be easily transported and recycled [4]. However, the gap with natural and intrinsic corn straw fiber particles need to be defined physicochemically prior to modification to be filled. Therefore, this paper established some selected physical and chemical properties of corn straw particles in its natural state comparatively and subsequently with properties enhancement in the treatment of oily wastewater, thereby providing new insight into producing a sustainable high-efficiency oil absorbent from agricultural waste.

2. MATERIALS AND METHOD

2.1. Preparation and pretreatment of the corn straw fibers

Corn straw was shredded and blended to obtain straw fibers for selected proximate compositions, which were then sieved through mesh standard screens to obtain seven uniform graded fibers (60,100,150,203,305,400 and 450mm). The powdered corn straw was then rinsed with equal volumes (50ml) of deionized water and ethanol. Afterward, they were immersed in a beaker with a mixture of 100 ml of 0.5 wt. % sodium hydroxide aqueous solution and 30 % (3.5ml) of H_2O_2 and mixed for 5h at ambient temperature. Moreover, the solution's pH (6.8-7.0) was regulated by HCl acid. After washing off with distilled water several times, the pretreated corn straw was dried at 400°C until its weight remained constant [6].

2.2. Proximate analysis

Moisture content, ash content and crude fiber were determined using AOAC (2000) method [7]. Cellulose, hemicellulose, and lignin were also determined [8]

2.3. Determination of gravimetric properties

2.3.1. Bulk density

Bulk density is the fraction of the sample solids mass to its volume. It was estimated by filling the sample to a known volume with a measuring cylinder. Tapping during the filling was done to acquire a uniform packet and to limit the divider impact. The filled sample was

gauged and the mass thickness was determined with the equation below [9].

$$\text{Bulk density} \left(\frac{\text{g}}{\text{cm}^3} \right) = \frac{M}{V}$$

where, M = mass of the sample (g); V = volume of the filled sample (cm^3).

2.3.2. True density

The true density is characterized as the fraction of the sample mass to its actual volume. It was calculated by the toluene displacement technique so as to prevent water retention during the test. 5g of the sample were taken and soaked into a 100 ml measuring cylinder with 50 ml of toluene [10]. The net volumetric changes were recorded. The equation below was applied.

$$\text{True Density}(\text{g}/\text{cm}^3) = \frac{\text{Weight of seed (g)}}{\text{Rise in toluene level (cm}^3\text{)}}$$

2.3.3. Tap density

Tap density is applied to forecast the movement characteristics, nature and compressibility of solid or powder particles [9]

$$\text{Tap density} \left(\frac{\text{g}}{\text{cm}^3} \right) = \frac{\text{weight of sample}}{\text{volume of tapped sample}}$$

2.3.4. Porosity

Porosity, ϵ (%) shows the measure of pores in the material and was determined from the normal estimations of bulk and true densities. [11]

$$\epsilon(\%) = \left[1 - \frac{\rho_b}{\rho_t} \right] \times 100$$

where, ρ_b = bulk density (g/cm^3); ρ_t = true density (g/cm^3)

2.3.5. Carr's index

The Carr or compressibility index means the compressibility of a powder. It is determined by the equation

$$C = 100 \frac{\rho_T - \rho_B}{\rho_T}$$

Where ρ_B is the unreservedly settled mass thickness of the powder (bulk density), and ρ_T , the tapped mass thickness of the powder (tapped density). In a free moving powder, the mass thickness and tapped thickness would be close in size with the smaller Carr's index. Then again, in a poor moving powder where there are more interparticle relationship, the contrast between the mass and tapped thickness would be more pronounced, hence, the Carr record would be larger. [14] Carr's limit of more than 25 is regarded as an index of bad flowability, and less than 15 is of good flowability

2.3.6. Hausner ratio

The Hausner proportion is a number that is related to the flowability of a powder or granular material. It is determined by the formula:

$$H = \frac{\rho_T}{\rho_B}$$

Where ρ_B is the openly settled mass thickness of the powder, and ρ_T is the tapped mass thickness of the powder.

Hausner ratio of more than 1.25 is considered a sign of poor flowability [15].

2.4. Determination of frictional properties

2.4.1. Angle of repose

The angle of repose is the angle with the level at which the material will stand when heaped. The chamber was set over a plain surface and corn straw powder was filled in. Tapping during filling was done to get uniform pressing and to limit the cylinder impact assuming any. The cylinder was gradually raised over the floor with the aim that the entire material could slide and shape a characteristic incline. The tallness of the mass over the floor and the width of the load at its base were estimated and the edge of rest (Φ) was determined by following condition [12];

$$\Phi = \frac{h}{R}$$

or

$$\Phi = \tan^{-1} \frac{2h}{D}$$

Where, Φ = angle of repose ($^{\circ}$); h = height of the pile (cm); and D = diameter of the pile (cm).

Table I. Repose angle standards.

Flow properties	Repose angle ($^{\circ}$)
Excellent	25-30
Good	31-35
Fair	36-40
Passable	41-45
Poor	46-55
Very poor	56-65
Very very poor	> 66

2.5. Functional properties (Oil absorption capacity)

0.5 g of the samples were blended with 6 ml of crude oil in a pre-gauged axis tube. They were mixed for 1 min with dainty metal wire to distribute the sample in the oil.

After a holding time of 30 min, the cylinders were centrifuged for 25 min at 3000rpm. The isolated oil was then expelled with a pipette and the cylinders were reversed for 25 min to drain the oil. Triplicate decisions were done and the oil sorption limits were communicated as a gram of oil per gram of the dried sample. [13]

$$OSC (\%) = \frac{(\text{Tube weight with drained oil}) - (\text{Tube weight corn straw fiber weight})}{\text{Corn straw fiber weight}}$$

3.RESULTS AND DISCUSSION

Table II. Proximate values of corn straw fiber particles.

Parameters		Value
Proximate Analysis	Moisture content (%)	16.25
	Ash content (%)	8.53
	Crude fiber (%)	72.12
	Cellulose (%)	23.15
	Hemicellulose (%)	14.05
	Lignin (%)	45.23

Table III. Selected Instrumental analysis of unmodified corn straw fibers

Material	Analysis	Value	Remark	Reference
Natural Corn straw	SEM		Smooth fibrous surface	Xu, Y., Yang, H., Zang, D., Zhou, Y., Liu, F., & Huang, X. et al. (2020). Preparation of a new superhydrophobic/superoleophilic corn straw fiber used as an oil absorbent for selective absorption of oil from water.
	FT-IR	700 – 3400cm ⁻¹	A band at the high frequency region at 3337cm ⁻¹ being assigned to –OH group vibration.	Hernandez, C., Ferreira, F., & Rosa, D. (2020). X-ray powder diffraction and other analyses of cellulose Nano crystals obtained from corn straw by chemical treatments.
		2920-2851cm ⁻¹	Asymmetric stretching vibrations of –CH ₂ and –CH ₃ .	
	EDX (Energy-dispersive X-ray spectroscopy)	Carbon, O ₂ and Zn	The peaks with oxygen and carbon were generated with respect to ZnO modified corn straw.	Study_of_Biochar_Properties_by_Scanning_Electron_Microscope_Energy_Dispersive_X-Ray_Spectroscopy_SEM-EDX https://www.researchgate.net/publication/293327931
Chemical stability (Contact angle)	≥ 150°	Slight variations with contact angle of 150° and above.	On_the_characterisation_of_structure_and_properties_of_sorghum_stalks https://www.researchgate.net/publication/271883918	

Corn straw modified ZnO at low magnification	SEM		Rough surface due to insoluble layer of ZnO granules	Xu, Y., Yang, H., Zang, D., Zhou, Y., Liu, F., & Huang, X. et al. (2020). Preparation of a new super hydrophobic/superoleophilic corn straw fiber used as an oil absorbent for selective absorption of oil from water
Corn straw modified ZnO at high magnification	SEM		ZnO particles were hollow spheres with approximately average diameter of 5um	Xu, Y., Yang, H., Zang, D., Zhou, Y., Liu, F., & Huang, X. et al. (2020). Preparation of a new super hydrophobic/superoleophilic corn straw fiber used as an oil absorbent for selective absorption of oil from water
	Super-hydrophobicity		Because of the abundant hydroxyl group (OH ⁻) on the fiber surface.	Xu, Y., Yang, H., Zang, D., Zhou, Y., Liu, F., & Huang, X. et al. (2020). Preparation of a new super hydrophobic/superoleophilic corn straw fiber used as an oil absorbent for selective absorption of oil from water

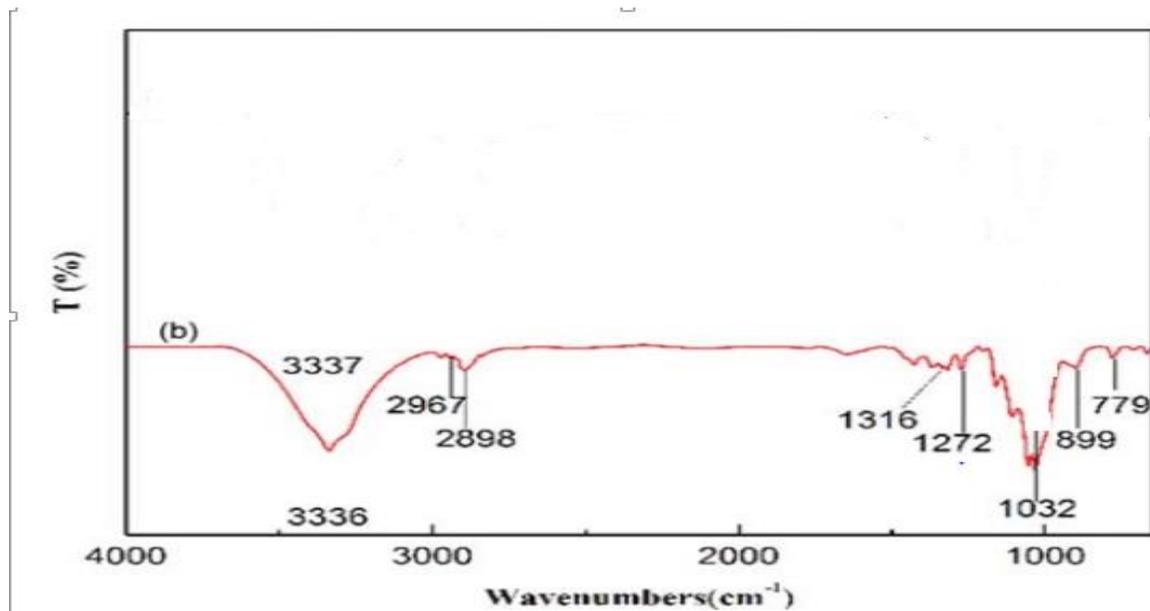


Figure 1. FT-IR of natural corn straw.

Table IV. Selected gravimetric, frictional and functional parameters of corn straw fiber particles

Particle size (mm)	Bulk density (g/ml)	Tap density(g/ml)	True density (g/ml)	Porosity (%)	Carr's index	Repose angle (degree)	Hausner ratio	OSC (%)
60	0.119	0.156	0.229	0.480	23.718	55.3	1.311	4.65
100	0.198	0.259	0.382	0.482	23.552	50.5	1.308	6.85
150	0.280	0.360	0.535	0.477	22.222	45.3	1.286	11.52
203	0.402	0.465	0.700	0.426	13.548	39.6	1.157	14.84
305	0.440	0.571	0.841	0.477	22.942	35.3	1.298	23.50

Table V. Statistical average mean of the selected parameters of pretreated corn straw fiber particles

Parameter	Actual mean value	Highest value	Lowest value
Particle size (mm)	163.6 ± 95.5369	259.1369	68.0631
Bulk density (g/ml)	0.2878 ± 0.1349	0.4227	0.1529
Tap density (g/ml)	0.3622 ± 0.1638	0.5260	0.1984
True density (g/ml)	0.5374 ± 0.2439	0.7813	0.2935
Porosity (%)	0.4682 ± 0.0239	0.4921	0.4443
Carr's index	21.1966 ± 4.3157	25.5123	16.8809
Repose angle (degree)	45.2 ± 8.0542	53.2542	37.1458
Hausner ratio	1.2718 ± 0.0651	1.3369	1.2067
OSC (%)	12.2720 ± 7.4283	19.7003	4.8437

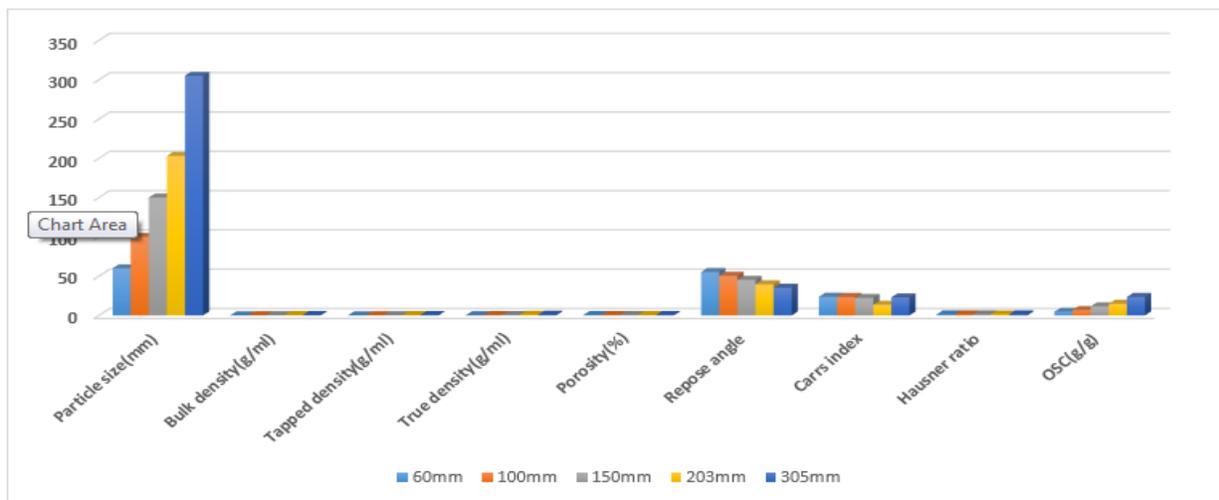


Figure II. 3D column for gravimetric, frictional and functional characteristics of corn straw fiber powder.

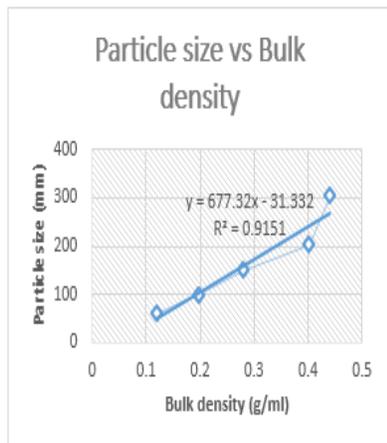


Figure III. Particle size against bulk density

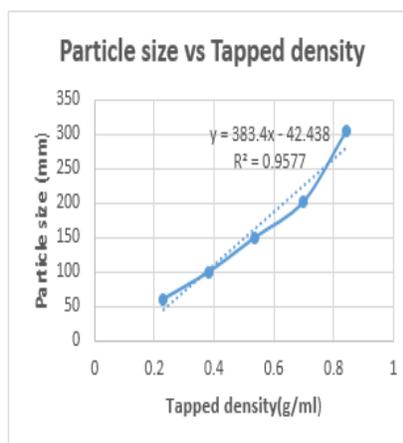


Figure IV. Particle size against tapped density

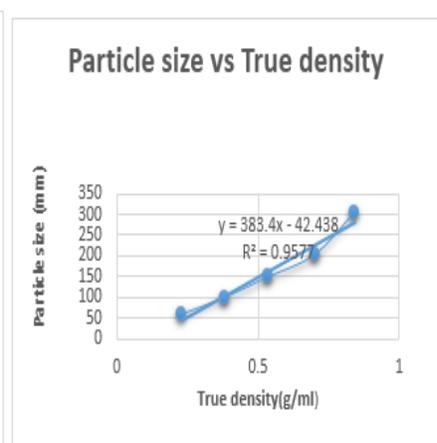


Figure V. Particle size against true density

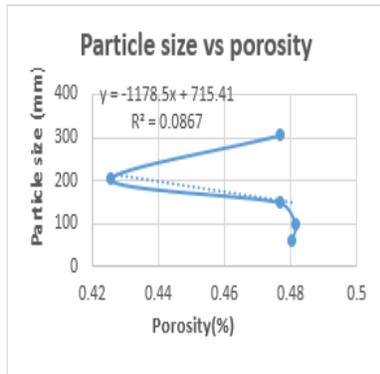


Figure VI. Particle size against porosity

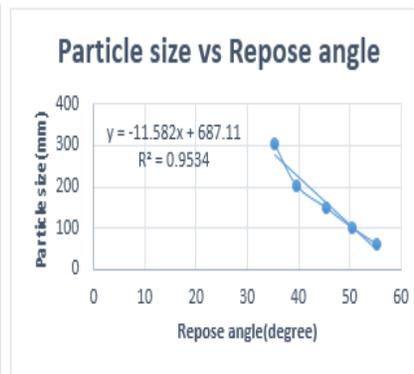


Figure VII. Particle size against repose angle

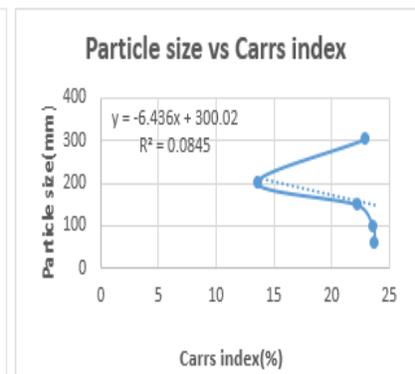


Figure VIII. Particle size against cars index

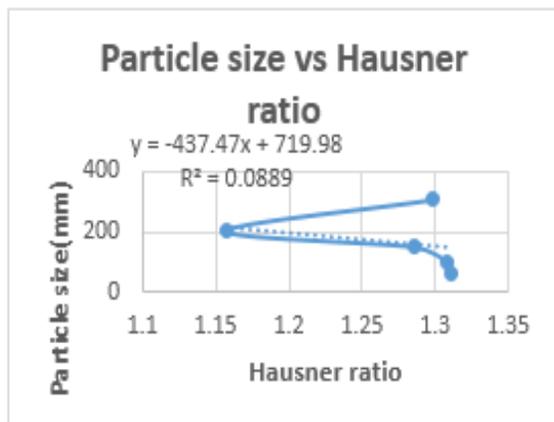


Figure IX. Particle size against hausner ratio

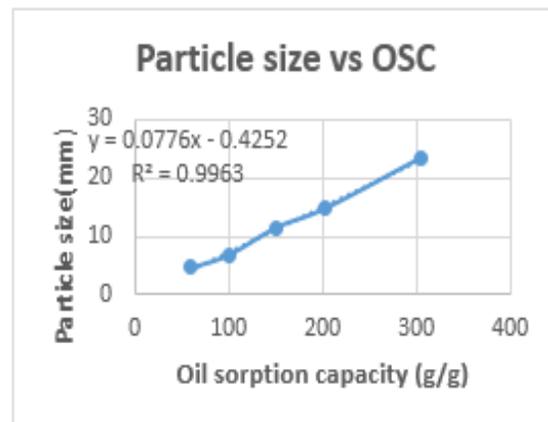


Figure X. Particle size against oil sorption capacity

Table VI. The T-test between particle size and bulk density of corn straw fiber powder.

T Test: Two Paired Samples								
SUMMARY			Alpha	0.05		Hyp Mean Diff	0	
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
Bulk density(g/ml)	5	0.2878	0.134930352					
Difference	5	163.3122	95.40783724	42.66768193	3.82753861	4	1.711727	0.886298
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.009328689	2.131846786			yes			
Two Tail	0.018657377	2.776445105	44.84772336	281.7766766	yes			

Table VII. The T-test between particle size and tapped density of corn straw fiber powder.

T Test: Two Paired Samples								
SUMMARY		Alpha		0.05		Hyp Mean Diff		0
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
Tapped density(g/ml)	5	0.3622	0.163813003					
Difference	5	163.2378	95.37589651	42.6533976	3.827076134	4	1.71152	0.886275
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.009332384	2.131846786			yes			
Two Tail	0.018664768	2.776445105	44.81298301	281.662617	yes			

Table VIII. The T-test between particle size and true density of corn straw fiber powder.

T Test: Two Paired Samples								
SUMMARY		Alpha		0.05		Hyp Mean Diff		0
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
True density(g/ml)	5	0.5374	0.243863281					
Difference	5	163.0626	95.2982611	42.61867799	3.826083015	4	1.711076	0.886225
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.009340326	2.131846786			yes			
Two Tail	0.018680652	2.776445105	44.7341801	281.3910199	yes			

Table IX. The T-test between particle size and porosity of corn straw fiber powder.

T Test: Two Paired Samples								
SUMMARY		Alpha		0.05		Hyp Mean Diff		0
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
Porosity(%)	5	0.468237571	0.023872415					
Difference	5	163.1317624	95.54393645	42.72854735	3.817863526	4	1.7074	0.885816
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.009406366	2.131846786			yes			
Two Tail	0.018812732	2.776445105	44.49829629	281.7652286	yes			

Table X. T-test between particle size and angle of repose of corn straw fiber powder.

T Test: Two Paired Samples								
SUMMARY		Alpha		0.05		Hyp Mean Diff		0
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
Repose angle	5	45.2	8.054191455					
Difference	5	118.4	103.4157628	46.24893512	2.560058944	4	1.144893	0.788031
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.031317084	2.131846786			yes			
Two Tail	0.062634169	2.776445105	-10.0076295	246.8076295	no			

Table XI. The T-test between particle size and Carr's index of corn straw fiber powder

T Test: Two Paired Samples								
SUMMARY		Alpha		0.05		Hyp Mean Diff		0
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
Carrs index	5	21.19657765	4.315666076					
Difference	5	142.4034224	96.87964761	43.32589554	3.286796974	4	1.4699	0.854275
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.015153262	2.131846786			yes			
Two Tail	0.030306525	2.776445105	22.11145176	262.6953929	yes			

Table XII. The T-test between particle size and hausner ratio of corn straw fiber powder.

T Test: Two Paired Samples								
SUMMARY		Alpha		0.05		Hyp Mean Diff		0
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d	Effect r
Particle size(mm)	5	163.6	95.53690386					
Hausner ratio	5	1.271832631	0.065109657					
Difference	5	162.3281674	95.55633583	42.73409252	3.798563578	4	1.698769	0.884846
T TEST								
	p-value	t-crit	lower	upper	sig			
One Tail	0.009563645	2.131846786			yes			
Two Tail	0.01912729	2.776445105	43.67930537	280.9770294	yes			

Table XIII. The T-test between particle size and oil sorption capacity of corn straw fiber powder.

T Test: Two Paired Samples							
SUMMARY		Alpha		0.05		Hyp Mean Diff	
Groups	Count	Mean	Std Dev	Std Err	t	df	Cohen d
Particle size(mm)	5	163.6	95.53690386				
OSC(g/g)	5	12.272	7.428342345				
Difference	5	151.328	88.12331286	39.40994359	3.839843101	4	1.71723
T TEST							
	p-value	t-crit	lower	upper	sig		
One Tail	0.009231008	2.131846786			yes		
Two Tail	0.018462016	2.776445105	41.90845502	260.747545	yes		

Table II is the selected proximate analysis of the pretreated fiber particles at room temperature where the crude fiber as a major parameter responsible for its sorption activity was obtained at 72.12%, while the fraction of cellulose, hemicellulose, ash content, and lignin are at 23.15%, 14.05%, 8.53%, and 45.23% respectively. Figure I reveals the FT-IR spectrum of the pretreated fiber particles with the pronounced peaks at 3336cm⁻¹, 1032cm⁻¹, and 899cm⁻¹ which are assigned to stretching vibrations of –OH, C-O-C and β –glycosidic linkage in the cellulose framework. Similar and previous works on corn straw fibers are depicted in Table III with scanning electronic microscopy (SEM), FT-IR, energy dispersive x-ray spectroscopy (EDX), chemical stability in terms of contact angle with respect to natural and ZnO modified corn straw were summarized. Selected gravimetric characteristics (bulk density, tapped density, true density, porosity, Carr’s index, and hausner ratio), frictional properties (Angle of repose) and functional properties (oil sorption capacity) were estimated with the individual relationship with the particle sizes. Figure II is the 3D histogram of the entire properties of the pretreated corn straw fibers (CSF). Figure III, IV, V, VI, VII, VIII, IX, and X are plots of the particle sizes against bulk density, tapped density, true density, porosity, repose angle, carr’s index, hausner’s ratio, and oil sorption capacity respectively. The plots against the bulk density, tapped density, true density, and oil sorption capacity (OSC) retain a strong positive correlation with the particle size distributions, while repose angle, porosity, Carr’s index, and hausner ratio presented an inverse and

non-linear relationship with the particle size of the pretreated fiber. Table V provides the statistical average mean values for all the selected parameters at the highest and the lowest levels. Two paired sample T-tests were conducted between the particle size and bulk density (Table VI), tapped density (Table VII), true density (Table VIII), porosity (Table IX), repose angle (Table X), carrs index (Table XI), hausner ratio (Table XII) and oil sorption capacity (Table XIII).

4.CONCLUSION

This study has established the nature of pretreated corn straw fibers as a chemically unmodified sorbent for crude oil spills on the aquatic environment with respect to some selected parameters.

Meanwhile, the average quantity of these CSF with the particle size of 163±95.5369 mm, bulk density of 0.2878±0.1349g/ml, tapped density of 0.3622±0.1638g/ml, true density of 0.5374±0.2439g/ml, porosity of 0.4682±0.0239%, carr’s index of 21.1966±4.3157, repose angle of 45.2±8.0542o, hausner ratio of 1.2718±0.0651 and oil sorption capacity of 12.2720±7.4283% were estimated. However, the repose angel was accepted at the level of 37.1458o (Table I). Porosity is enhanced at 0.4921% but will be higher when CSF is chemically treated [R]. Carr’s index at 25.5123%, hausner ratio at 1.2067 for better flowability and 19.7003% with OSC.

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