

Specific Physiochemical Characterization of Corn Straw Fibers as Potential Agro-sorbent for Crude Oil Spills on Surface Water.

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Abstract: Oil to water pollution remains an international menace with terrible environmental and socio-economic implications. However, diverse control methods and techniques such as agricultural biomaterials as fibers are been designed for the management of the crude spills across the water bodies. Most plant fibers have been reported to be good sorbents of oil. However, despite the excellent capacity of most synthetic sorbents, natural agro-based resources are still perceived to be of good performance when critically evaluated. Natural corn straw fibers have been selectively identified as a readily available, viable, and environmentally oriented approach with the remediation potential of water bodies. The proximate compositions, such as the moisture at 16.25 percent, ash content of 8.53 percent, crude fiber of 72.12 percent, cellulose of 23.15 percent, hemicelluloses of 14.05percent, and lignin content of 45.23percent were determined. Identifications by FT-IR justify the availabilities of the stretching vibrations of -OH, C-O-C, and alpha-glycosidic bonds respectively. Five different particle sizes (60,100,150,203, and 305mm) were adopted in the treatment of the selected characteristics. Thus, the individual particle sizes (PS) were interacted and modeled with the bulk densities (Bd)[$PS=677.3(Bd) - 31.33$]; tapped densities(Td)[$PS=573.2(Td) - 44.02$], true density(TrueD)[$PS= 383.4(TrueD) - 42.43$], porosity(p) [$PS= - 3236(p) + 1652$], repose angle(Ra)[$PS= -119.2(Ra) + 2889$], carr's index(CI)[$PS= -11.58(CI) + 687.1$], hausner ratio(Hr)[$PS= -2619(Hr) + 3520$], and the spill sorption capacity (COSC)[$PS= 12.83(COSC) + 6.057$]. The significance of these relationships was ascertained with the statistical T-tests. The least particle size of 60mm retained the sorption ability of 4.65% with the initial fiber weight of 0.5g with a very poor frictional property of 55.3^o, while the highest particle size of 305mm resulted in 23.50% sorption capacity with an appreciable frictional property of 35.3^o. These developments concerning the poor crude oil sorption performance have declared a rationale for the improvement of corn straw fiber as an agro-sorbent through chemical modifications for optimum performance.

Keywords – water pollution, crude oil spills, agricultural biomass, biodegradable sorbent, and particle size.

1. INTRODUCTION

Crude oil remains the fraction of natural fossil resource with the substratum of energies industrially. Unfortunately, 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 [1]. Emulsified oil is exceptionally difficult to clean by ordinary strategies as the oil contains numerous harmful and extremely destructive synthetics components to the environment where they can have impacts on living organisms [2]. At present, physical, chemical, and natural treatment techniques were applied in cleaning up and remediating contaminated water systems. Physical adsorption procedures, which are connected with hydrophilic materials to expel released oil from water, are considered the most financially and legitimate technique because of their high retention limit and economy [3]. Meanwhile, the majority of synthetic absorbents are usually limited as non-biodegradability, low absorption capacity, poor separation efficiency, and high cost of production

[4]. In this direction, the development of a novel ecological and oleophilic material with a higher sorption potential with cost-effectiveness is extensively required. Typically, waste agro-based biomass with oil retentiveness is of amazing significance for oil-water interface with the scientific investigation to the advantage of its availability, and biodegradability [5]. As of late, very active hydrophobic layers have stood out because of their remarkable properties, including self-cleaning, against staying, waterproof, substance steadiness, and oil recuperation [6] By standard, the development of a natural sorption structure with low surface activity is an imperative parameter for the making of the active hydrophobic surface, which is described by the angle of contact above 150° [7]. Cellulose is the major strength block for the plants and in maximum fibers. The amount of cellulose is around 40 to 50 % in the majority of the natural fiber. Cellulose has a translucent framework when contrasted with different constituents present in the regular fiber. In the crystallinity formation of cellulose, the particles equipped with hydrogen bonds invigorate the fiber during stacking conditions [8]. Hemi

cellulose is the most available fraction accessible in fiber after cellulose. The content is co-joined with the fibrils of cellulose with hydrogen bond connecting up with the fibrils support and functions as a network material for restricting the cellulose fibrils with lignin. Hemi cellulose that is making out of glucose, arabinose, sugar, xylose, galactose, and mannose as building blocks in common fiber allows the expansion with the hydrophilic and thermal characteristics. Eventually, the overabundance measure of hemicellulose content in common fiber inhibits the hydrophobicity of the fibers at higher temperatures. [9]. Also, the oleophilic characteristic of natural fiber depends upon the hemicellulose concentration in fiber as they are generally adhered to the cellulose microfibrils and in fiber in form of a short branch crudely crystalline chain for

embedding the microfibrils [10]. Lignin is also a binding matrix for cellulose microfibrils it will attach with the cellulose with the help of hemicellulose content. It gives rigidity to plant fiber by bind up the microfibrils, resisting microbial attack and hydrolysis with any acid. Lignin has an amorphous structure of cross-linked molecules and acts as a glue between fibrils [11]. Pectin remains a heteropolysaccharide in the essential cells of plant fiber. The adaptability of plant and fiber is technically on the pectin components [12]. Hemicelluloses and cellulose contents are susceptible to heat before the lignin. In other words, the thermal activity of most plant fibers is because of the lignin component as this development presents a protection layer of lignocellulosic fiber for subsequent deactivation [13].

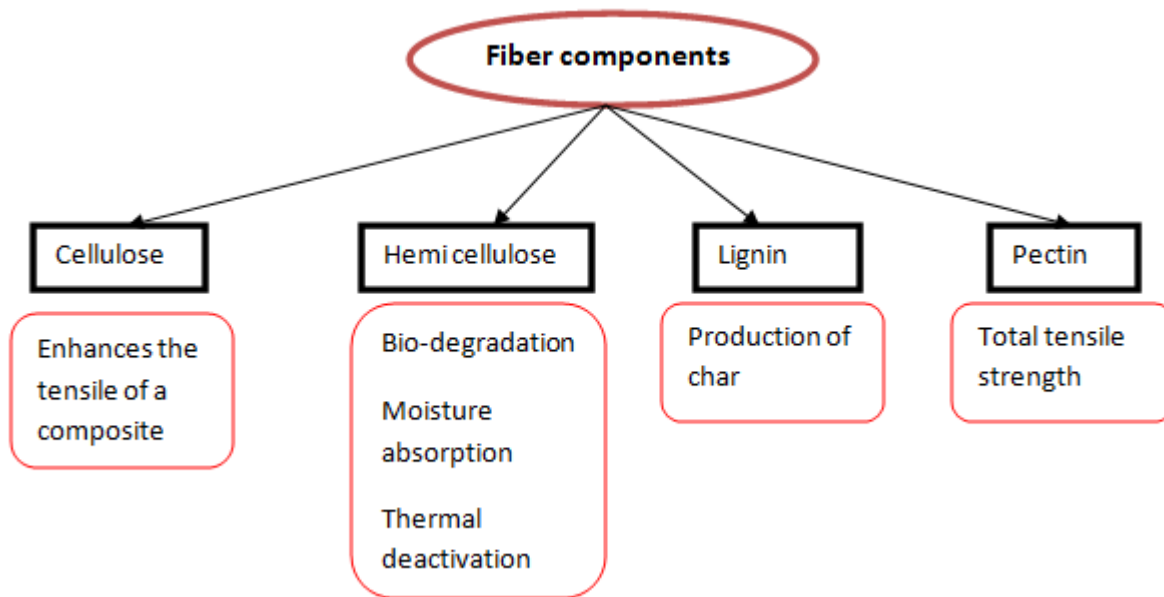


Figure1. Fiber compositions with composite characteristics [14, 15]

Corn straw fiber, natural and biodegradable biomass from agricultural waste, is typically disposed of 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 expelled oil from water applying super-hydrophobic/super-oleo phobic corn straw yarns with ZnO particles through regular impregnation [16]. ZnO particles are deep spheres with a normal measurement of 5 µm and a hydrophobic hexadecyltrimethoxysilane modifier. The superhydrophobic/superoleophobic properties of the organized corn straw fiber emerged from the consolidated impacts from the deposition of homogeneous SiO₂ 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[16]. This enables the biomass the capacity to effectively remove oils from water bodies. Given 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[17]. Therefore, this study recognized some selected physicochemical indices of corn straw particles in their natural state comparatively and subsequently with properties enhancement with the remediation of oily wastewater, thereby providing new insight into the development of highly efficient and biogenic oil sorbent from plant resources.

2.0 EXPERIMENTAL

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 H₂O₂ 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 40°C until its weight remained constant [18].



Figure 2. Natural Corn straw fibers particles.

2.2. Proximate analysis

Moisture content, ash content, and crude fiber were determined using AOAC (2000) method [19]. Cellulose, hemicelluloses, and lignin were also determined [20].

2.3. Determination of gravimetric properties

2.3.1. The bulk density

This 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 [21].

$$\text{Bulk density} = \frac{\text{Sample Mass (g)}}{\text{Volume of the filled sample (cm}^3\text{/ml)}}$$

2.3.2. Tap density
 Tap density is applied to forecast the movement characteristics, nature, and compressibility of solid or powder particles [21].

$$\text{Tap density} = \frac{\text{Sample weight (g)}}{\text{Volume of the tapped sample (cm}^3\text{/ml)}}$$

2.3.3. True density

The true density is characterized as the fraction of the sample mass to its actual volume. This was determined by the toluene displacement strategy to forestall water maintenance with the

test. 5g of the example were taken and drenched into a 100 ml estimating chamber with 50 ml of toluene [22]. The net volumetric changes were recorded [22].

$$\text{True density} = \frac{\text{Sample Weight (g)}}{\text{Rise in Toluene level (cm}^3\text{/ml)}}$$

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. [23].

$$\epsilon (\%) = 1 - \frac{\text{Bulk density}}{\text{True density}} \times 100$$

2.3.5. Carr's index

The Carr or compressibility index means how compressible a powder can be.

$$\text{CI} = 100 \left(\frac{\text{Tapped density} - \text{Bulk density}}{\text{Tapped density}} \right)$$

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 relationships, the contrast between the mass and tapped thickness would be more pronounced, hence, the Carr record would be larger. Carr's limit of more than 25 is regarded as an index of bad flowability and less than 15 is of good flowability [24]

2.3.6. Hausner ratio

This is a factor that is identified with the flowability of a powder or granular material [24].

$$HR = \frac{\text{Tapped density}}{\text{Bulk density}}$$

2.4. Determination of frictional properties

2.4.1. Angle of repose

This expresses 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 angle of rest (Φ) was determined by the following condition [24];

$$\phi = \frac{h}{R} \quad \text{OR} \quad \phi = \text{Tan}^{-1} \frac{2h}{D}$$

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

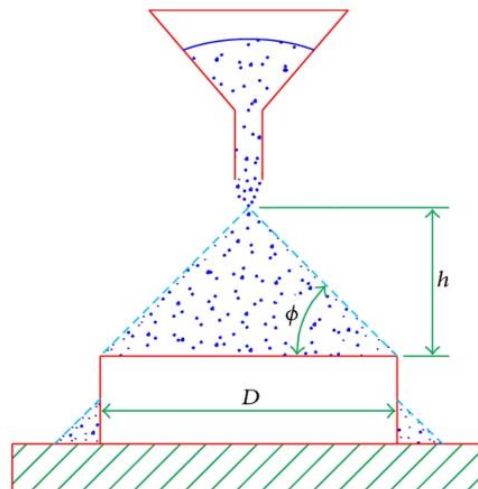


Figure 3. Repose angle experimental set up

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. [25]

2.6 FT-IR Analysis

The powder was carefully homogenized with potassium bromide and carved into pellets for analysis in the range of 400-4000 cm^{-1} [26].

3. RESULTS AND DISCUSSION

Table I. Proximate Evaluation of natural corn straw fibers.

Parameter	Value
Moisture Content (%)	0.20
Ash content (%)	8.53
Crude fiber (%)	72.12
Cellulose (%)	23.15
Hemi cellulose (%)	14.05
Lignin (%)	45.23

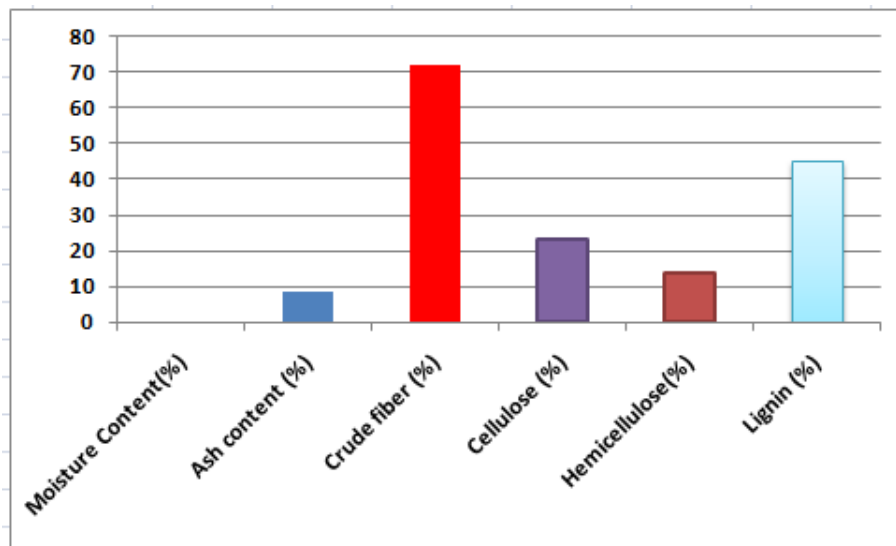


Figure 4. 2D chart for the proximate compositions of natural corn straw fibers.

Natural fibers derived from plants mainly consist of cellulose, hemicellulose, lignin, pectin, and other waxy substances. Cellulose remains a profoundly crystalline framework with about 80 percent crystalline structures [13]. Hemicellulose is comprised of highly structured polysaccharides appended to the depectinized cellulose. Lignin in its formless state hardens the cell boundaries and is a defensive mechanism for cellulose. The impact of various constituents in terms of the characteristics of various common fibers rests on the levels of fiber in them. In a

nutshell, cellulose is just solely responsible for the intrinsic strength of the fiber due to its structural frameworks. Technically, the hydrophilic status of fibrous hemicellulose is simply accountable for its hydrophilicity, as the geometry of the microfibrils attached with the cellulose is completely linked with the lignin and the hemicelluloses activity. Hence, the estimated level of hemicelluloses in corn straw fiber is an indication of unmodified and natural properties.

Table 2. Repose angle of powders [34].

Flow property	Angle of repose (degree)
Excellent	25 –30
Good	31 –35
Fair	36–40
Passable	41–45
Poor	46–55
Very poor	56–65
Very very poor	>66

Table 3. Selected gravimetric, frictional and functional parameters of corn straw fibers.

Particle size (mm)	Gravimetric properties						Frictional property	Functional property
	Bulk density (g/ml)	Tap density (g/ml)	True density (g/ml)	Porosity (%)	Carr's index	Hausner ratio	Repose angle(degree)	OSC (%)
60	0.119	0.156	0.229	0.488	23.718	1.321	55.3 (VP)	4.65
100	0.198	0.259	0.382	0.482	23.552	1.308	50.5(VP)	6.85
150	0.280	0.360	0.535	0.470	22.570	1.286	45.3(P)	11.52
203	0.402	0.465	0.700	0.439	22.330	1.250	39.6(F)	14.84
305	0.440	0.571	0.841	0.421	22.100	1.242	35.3(G)	23.50

VP = Very poor, P= Passable, F= Fair, G= Good

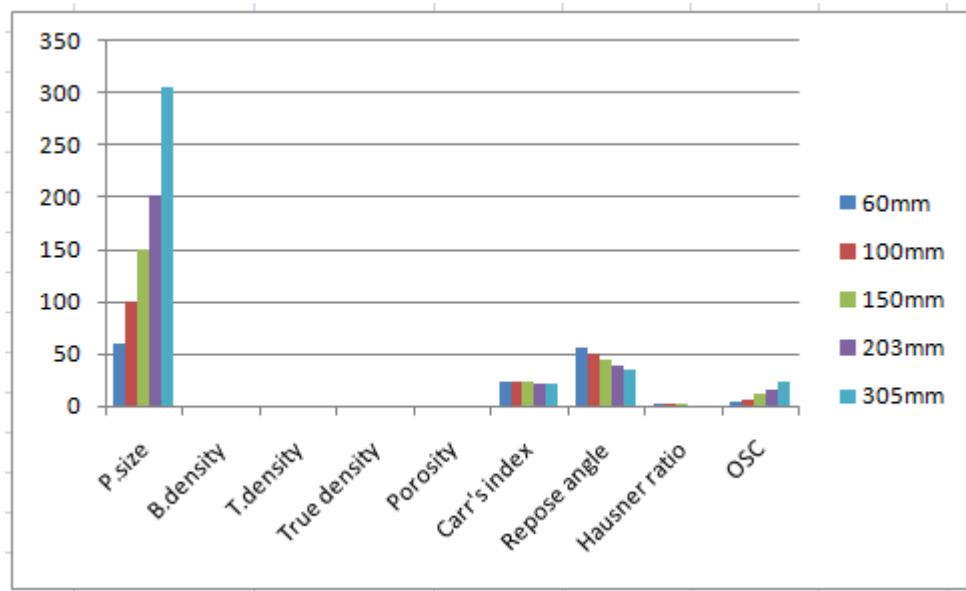


Figure 5. 2D charts for the gravimetric, frictional and functional characteristics of corn straw fibers.

The selected gravimetric characteristics, frictional properties, and functional properties as oil sorption capacity were estimated against each particle size. The particle sizes were the function of the gravimetric, frictional, and sorption capacities of the fiber material. It can be deduced from Table 3, Figure 4, and Figure 5 that the gravimetric characteristics increase with the bulk, tapped, and true densities. The carrs index and the hasner ratio in the

same settings diminish significantly with the trend in the particle sizes. Frictional properties with repose angle decrease with the increase in the particle sizes. The best point is achieved at 35.3° with the implication of averagely good flowing potential. In other words, the frictional level of natural corn straw fibers was ascertained to be a very poor flowing material at the least particle size of 60 and 100mm

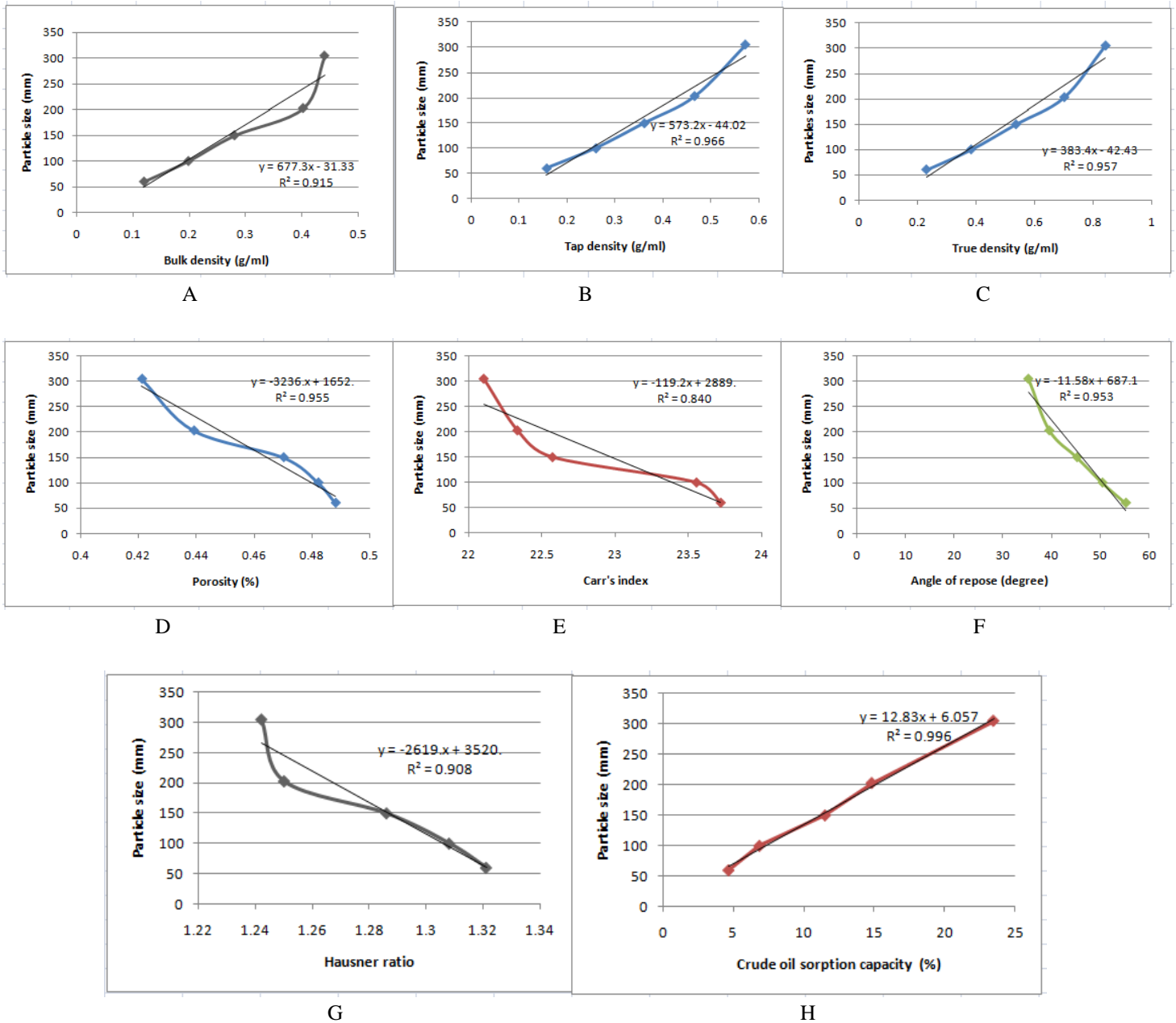


Figure 6. Particle size with the selected properties plots

Table 4. The T-test between particle size and bulk density of corn straw fiber powder ($\alpha = 0.05$); (n=5).

Group	T- test	P value	T-Critical	Upper value	Lower value	Significance	Model	R ²
Particle size	1	0.0093	2.1319	-	-	Yes	P= 677.3(Bd) – 31.33	0.9150
Bulk density	2	0.0187	2.7765	281.7767	44.8477	Yes		
Particle size	1	0.0093	2.1319	-	-	Yes	P= 573.2(Td) – 44.02	0.9660
Tapped density	2	0.0187	2.7765	281.6626	44.8130	Yes		
Particle size	1	0.0093	2.1319	-	-	Yes	P= 383.4(TrueD) – 42.43	0.9570
True density	2	0.0187	2.7765	281.3910	44.7342	Yes		
Particle size	1	0.0094	2.1319	-	-	Yes	P= -3236(porosity) + 1652	0.9550
Porosity	2	0.0188	2.7765	281.7652	44.4983	Yes		
Particle size	1	0.0313	2.1319	-	-	Yes	P= -119.2(CI) + 2889	0.8400
Repose angle	2	0.0626	2.7765	246.8076	10.0076	Yes		
Particle size	1	0.0152	2.1319	-	-	Yes	P= -11.58(RA) + 687.1	0.9530
Carrs index	2	0.0303	2.7765	262.6954	22.1115	Yes		
Particle size	1	0.0096	2.1319	-	-	Yes	P= - 2619(HR)+3520	0.9080
Hausner ratio	2	0.0191	2.7765	280.9770	43.6793	Yes		
Particle size	1	0.0092	2.1319	-	-	Yes	P= 12.83(OSC)+6.057	0.9960
C.oil sorption capacity	2	0.0185	2.7765	260.7476	41.9085	Yes		

Figure 6 are plots of the particle sizes against the bulk densities, tapped densities, true densities, porosities, repose angles, carr's indices, hausner's ratios, and oil sorption capacities. They are as well the representations of the mathematical models as computed by the statistical T-test between the fiber's particle sizes and the rest of parameters. (Figure 5 and Table 4). It is apparent that the correlation with the particle sizes/bulk densities [(y= 677.3x – 31.33) at R² of 0.9150]; particle sizes/tap densities [(y= 573.2x – 44.02) at R² of 0.9660]; particle sizes/true densities [(y= 383.4x

– 42.43) at R² of 0.9570] and particle sizes/oil sorption capacity [(y= 12.83x + 6.057) at R² of 0.9960] are in direct proportionalities. As particle sizes/porosities [(y= -3236x + 1652) at R² of 0.9550]; particle sizes/carr indices [(y= -11.58x + 687.1) at R² of 0.9530]; particle sizes/repose angles [(y= -119.2x + 2889) at R² of 0.8400] and the particle sizes/hausner ratio [(y= -2619x + 3520) at R² of 0.9080] in inverse relationships.

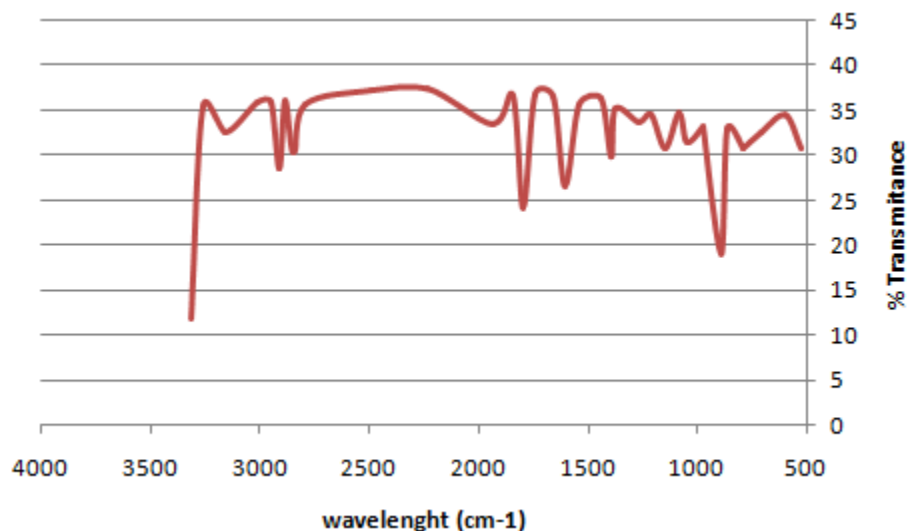


Figure 7. FT-IR of natural corn straw fibers.

In Figure 7 above, the broad peak at 3312 cm⁻¹ was ascribed to -OH stretching vibration in cellulose, hemicelluloses, and lignin molecules. The stretching vibrations of the C-H bonds of -CH₂

moiety in cellulose and hemicellulose molecules were observed at 2905 cm⁻¹. The peak at 1791 cm⁻¹ corresponded with the vibration stretching peak of nonconjugated carbonyl groups in

lignin molecules. The peaks at 1603 and 1401 cm^{-1} were assigned to the vibrational stretching of the C=C portion of the aromatic rings. The peak around 1393 cm^{-1} was assigned to the vibrational

stretching of the glycosidic bonds on cellulose and hemicellulose, affirming that the corn straw contains a lot of lignin, cellulose, and hemicellulose.

Table 5. Instrumental characterization of natural 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 – 3400 cm^{-1}	A band at the high frequency region at 3337 cm^{-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.
		2920-2851 cm^{-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)	$\geq 150^\circ$	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 5 μm	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

4. CONCLUSION

The selected and identified natural qualities of corn straw strands as a potential sorbent for crude spills of oil on water surfaces have been disclosed. The outcomes propose that replacement of manufactured oil sorbents in oil slick cleanup is conceivable by horticultural or phytochemical buildup which could be valuable by consolidating different benefits like biodegradability and their environmental advantages. Furthermore, an active chemical modification will be required in the enhancement of corn straw fiber sorption capacity, hydrophobicity, and buoyancy in an aqueous system.

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