

REVIEW PAPER

Comparative Study of Natural Fibres as Sustainable Oil Sorbents: Recent Developments in Modification Techniques and Oil Absorption Performance

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Received: 15-04-2026

Revised: 30-05-2026

Accepted: 10-06-2026

ABSTRACT

Oil spills or improper disposal of crude untreated oil, can pose detrimental effects on the environment, living organisms and economy. Industry has invoked many operating and maintenance procedures to reduce accidents that lead to spills. The rate of spillage has decreased in the past 10 years, even with increased oil production, transportation, and consumption. Despite this, spill experts estimate that 30%–50% of oil spills are either directly or indirectly caused by human error, with 20%–40% of all spills caused by equipment failure or malfunction. Emerging spill risks include increased maritime activity in the Arctic, deepwater exploration and development, and the rapid expansion of rail transport of crude oil. In the present work, an attempt is made to provide an efficient, easily deployable method of cleaning up oil spills and recovering of the oil. Driven by the requirement to replace the conventional synthetic fibres to meet this cause, sustainable eco-friendly alternatives are being developed. Natural fibres and by-products of agricultural processes have emerged as promising candidates, due to the low cost, renewability, abundance, biodegradability and suitable absorption characteristics. This study explores the development of high-value oil-absorbing materials from low-cost natural fibres, focusing on jute, banana, okra and mesta, with kapok chestnut-shell fibres, coconut-derived fibres, palm oil fibres, kenaf fibres, and other lignocellulosic agricultural residues. The raw fibres were subjected to several surface modifications procedures like - alkali scouring, bleaching, silane treatment, TEOS-based silica deposition, acetylation and DTES grafting, and the change on their sorption characteristics were analysed. It could be concluded that porosity, fibre morphology, chemical composition, arrangement of surface waxes, strongly influence their oil and water absorption characteristics.

HIGHLIGHTS

- Natural fibres offer sustainable alternatives to synthetic oil sorbents.
- Surface modification improves hydrophobicity, oleophilicity, and oil uptake.
- Kapok and cotton demonstrate superior oil sorption performance.
- Porosity, morphology, waxes, and wettability govern sorption behaviour.
- Greener modifications enable reusable, scalable, biodegradable oil sorbents.

Keywords: Alkali scouring, Bleaching, DTES grafting, Natural fibres, Oil spills, Synthetic fibres, TEOS

How to cite this article: Ray, D.P., Basak, S., Samanta, K.K., Jagadale, M., Jose, N., Debnath, S., Shakyawar, D.B., Bhowmick, S., Adhikary, K. and Mitra, R. (2026). Comparative Study of Natural Fibres as Sustainable Oil Sorbents: Recent Developments in Modification Techniques and Oil Absorption Performance. *Int. J. Bioresource Sci.*, 13(01): 129-145.

Source of Support: None; **Conflict of Interest:** None



Oil is a naturally occurring fossil fuel (Landon, 2002). They serve as the major source of energy in the modern world, and are the raw material for production of synthetic and organic chemicals (Hussein *et al.* 2008). Crude oil or petroleum oil is a mixture of hydrocarbons present in liquid state in the reservoirs and retain their physical state under the atmospheric pressure (Jarmolowicz-Szulc, 2021). It is hydrophobic in nature, thus the two liquids when mixed are found to be immiscible and remain as emulsions (Xia *et al.* 2023). During the extraction, transportation and storage of the crude oil resources, there might be accidental release into the environment, mainly water bodies, that leads to oil spills. Oil spills are man-made calamities that have severe detrimental effects on water ecology and environment (Al-Majed *et al.* 2012b). The oil spills greatly affect marine life, even marine birds face the impact, while shell fishes are the most vulnerable (Dala & Arslan, 2021). They also spoil beaches and shorelines, thus posing a threat to human life as well as the flora and fauna there (Jernelöv, 2010b).

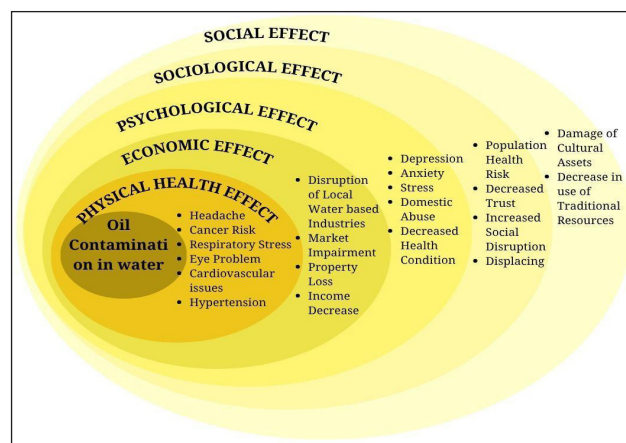


Fig. 1: Harmful effects of Oil Spills

Soon after the oil spills cover the water surface, various processes take place at once, such as - evaporation, emulsification, photo-oxidation, dispersion, sinking, resurfacing and tar-ball formation (Ivshina *et al.* 2015d).

Due to the increased number of oil spills in the present times, the necessity for a low cost sustainable cleanup technology is in high demand. There are different physical, chemical and biological methods for the cleaning of oil spills (Muizis *et al.* 2013).

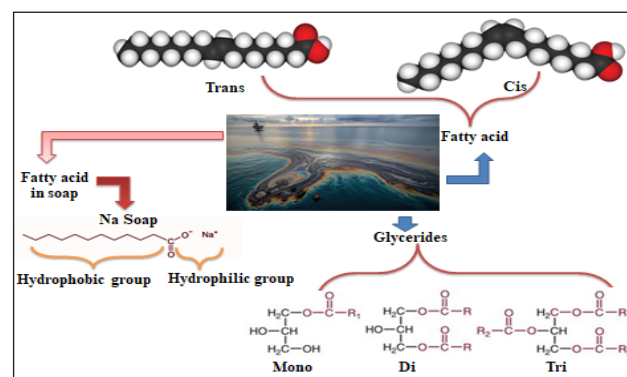


Fig. 2: Major components in FOG (Fat, oil & grease) deposition governing hydrophobic-hydrophilic interactions in oil sorption

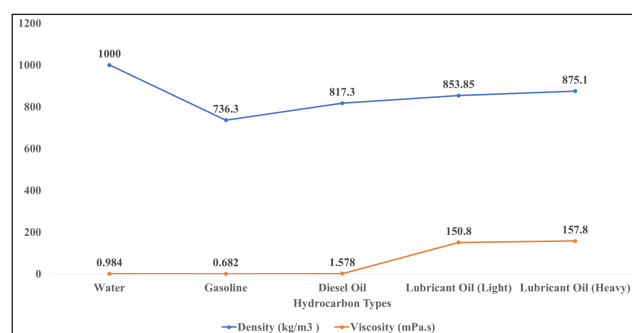
The common ways used are physical cleaning by use of booms, skimmers and sorbents, chemical oil removal by solidifiers or dispersants and biological cleaning by using microorganisms to convert the toxic chemicals to less harmful materials. Out of this physical cleaning by sorption proves to be the most efficient and economical (Prabakaran, *et al.* 2011). The sorbents are different organic, inorganic and synthetic materials (Hussein *et al.* 2009) that soak up liquids by adsorption, absorption, or both. The sorbents transform the oil to a semi-solid phase which is then eliminated from water (Sun *et al.* 2024). Conventionally, water pollution by oil spills is remediated by the use of synthetic sorbents. Among the different synthetic products are polypropylene, and polyurethane foams that are majorly in use (Teas *et al.* 2001). They exhibit high oil uptake capacity and low water absorption, making them suitable for recovery of oil spills. But despite being suitable in oil absorption, the synthetic fibres are not ideal sorbents. The synthetic polymers have poor biodegradability, hence they are either to be disposed of in landfills or incinerated, which adds to environmental pollution (Maja *et al.* 2008). The products are toxic to the environment and are manufactured by energy-intensive procedures. They rely on petroleum reserves for their production, causing the resource to deplete as it is non-renewable (Renuka *et al.* 2016). Therefore, the synthetic fibres incur high costs and are not appropriate for commercialisation or even maintaining an ecological balance. An ideal sorbent should have the properties of hydrophobicity-lipophilicity providing them a high oil uptake capacity, buoyancy, retention of oil over time, easy renewability, recovery of oil and

Table 1: Oil spill remediation techniques

Parameter	Physical	Chemical	Thermal	Biological
Principle	Physical oil removal	Chemical oil alteration	Heat-based removal	Microbial degradation
Advantages	Direct recovery	Rapid large-scale treatment	Fast-oil reduction	Eco-friendly cleanup
Limitations	Weather dependent	Ecotoxicity concerns	Air pollution	Slow process
Best Application	Initial containment	Offshore spills	Thick oil slicks	Long-term restoration
Overall Assessment	Immediate response	Emergency control	Rapid volume reduction	Sustainable remediation

biodegradability (Wei *et al.* 2003). Researchers have found natural fibres as more efficient and effective oil sorbents than the synthetic ones (Ana *et al.* 2004). Natural fibres have fibrous, porous structure (Lim & Huang, 2006c) that allows uptake of liquids, they are easily available from renewable natural sources, are biodegradable, have low density hence buoyant and provide sufficient retention of oil. They are also low-cost. These properties allow them to serve as suitable alternatives to conventional synthetic products. However, they also have certain drawbacks - because they are rich in cellulose they are hydrophilic (Faruk *et al.* 2012). The preference to sorption of water hampers or restricts the oil uptake. The hydrophobic-lipophilic nature of a material is dependent on its surface chemistry including porous structure, hollow lumen and surface waxes. There are different plant-based fibres that have been tested for their oil absorption like cotton fibre, kapok fibre, milkweed fibre, corn silk, jute, banana fibre, and okra fibre (Behnood *et al.* 2013). Several scientists have treated the natural fibres by different surface modification techniques. Alkaline treatment using sodium hydroxide removes surface impurities like waxes, pectins, and lignin keeping only the cellulosic layer (Hafidz *et al.* 2021b). Acetylation processes improves oil affinity by converting the hydrophilic fibres to hydrophobic ones, by replacing their OH⁻ groups with non-polar functional groups (Teli & Valia, 2012b). Moreover, modification by silane treatment, plasma treatment, and sol-gel modification has also been employed to improve the surface of the natural fibres (Cruz & Fanguero, 2016b). Raw cotton absorbs oil by adsorption using its surface waxes, physical trapping by surface morphology or capillary diffusion into its pores. Treatment by organic solvents like trichloroethane and trichloroethylene removes its surface waxes and slightly lowers oil sorption (Hyung-Min *et*

al. 1993). Kapok (*Ceiba pentandra*) is a suitable natural sorbent with great hydrophobic-lipophilic properties. It is lightweight with low packing density and has a hollow lumen, moreover it is sufficiently hydrophobic, making it a suitable oil absorbent (Xiaofeng Huang *et al.* 2005). It has been observed that increased ratio of kapok in nettle-kapok nonwoven fibre raised its oil absorption and lowered the water uptake, compared to just nettle nonwoven fibre (Thilagavathi & Karan, 2018b). Milkweed fibres with high wax content on their surface, demonstrates sufficient amounts of crude oil absorption at room temperature (Chol *et al.* 1992).

**Fig. 3:** Characteristics of different types of oil

Banana fibre is a lignocellulosic fibre with fairly good mechanical properties as sorbents, however they tend to absorb water readily. Esterification of the fibres increases their hydrophobicity-lipophilicity (Korhonen *et al.* 2011b). Despite the rigorous research in this field, there are certain research gaps that prevent its wide-scale acceptance and commercialization. There is a lack of standard comprehensive protocols to measure oil absorption by the fibres. A major gap lies in linking of the fibre structure to their oil sorption behaviour. While some fibres have been extensively studied by multiple researchers, like cotton and kapok, some others, especially agro-residues have only a few laboratory

evaluations with no industrial trials. Although the surface modification techniques are seen to improve lipophilicity of the fibres the parameter for their optimum results have not yet been standardized. Moreover, most experiments have been conducted at laboratory scale, and have not yet been analysed in real life scenarios.

Synthetic Fibres

Synthetic polymeric sorbents make up the most widely used marketed class of oil sorbent materials and have been greatly deployed for oil spill remediation due to their superior oil sorption capacity, chemical stability, buoyancy, and hydrophobic features. These are manufactured by the use of petroleum-derived polymers and are particularly prepared to increase the oil sorption ability. Unlike materials like ash, limestone, clay, perlite or other industrial byproducts which were not specifically designed for spill remediation, but their oil uptake ability was discovered as a secondary property, the synthetic sorbents are especially engineered to have selective oil uptake, high oil retention and low water uptake. They are made to undergo several surface treatment procedures for enhanced performance during marine as well as terrestrial oil spill scenarios. Conventional synthetic sorbents are mainly made of high molecular weight polymers like polystyrene (PS), polypropylene (PP), polyethylene (PE), polyurethane (PU), polyethylene terephthalate (PET), rayon, nylon and multiple copolymeric mixtures. In spite of having these advantages these commercial sorbents have some environmental as well as economic limitations.

Both of these present certain secondary pollution issues. Landfills result in piling up of polymeric wastes in extensive land areas, on the other hand incineration emits toxic fumes and greenhouse gases. Furthermore, the degradation of these sorbents might release microplastics into marine ecosystems adding to the environmental burden. The polymeric sorbent materials can be categorized into fibrous materials and porous or cellular materials. Fibrous sorbents, especially polypropylene nonwovens, depend on capillary channels between fibres and have rapid oil uptake and high buoyancy properties.

Structural Characterization of Synthetic Fibres

The oil absorption capacity strongly relies on

structural parameters such as porosity, fibre diameter, surface morphology, packing density, and overall fibre architecture. These fibres mainly retain oil by capillary action taking place within the inter-fibre spaces. As a result, the arrangement and morphology of the fibrous assembly have a significant role in deciding the sorption efficiency.

The nonwoven polypropylene structure has randomly organized fibres interconnected to form multiple micropores and capillary channels. These pores help quick transport as well as retention of the oil molecules. Scanning electron microscopic analysis have exhibited smooth cylindrical surfaces in polypropylene fibres with comparatively uniform diameters. It has a fibre diameter smaller than kapok and cotton. This small diameter makes up finer capillary channels and raises the number of pores in the arrangement. The smaller capillaries lower gravitational drainage and facilitates more effective filling of the pore spaces thereby providing improved oil retention. The packing density and fibre volume also influences the absorption capacity. The loosely packed fibres have large pore volumes and allow higher oil penetration, unlike densely packed structures that obstruct fluid flow and lower the oil uptake ability. An optimum balance between the packing density and porosity therefore enhances the sorption behaviour.

Cellular materials including polyurethane foams have interconnected open pores that operate as reserves for oil storage. On the other hand, fibrous sorbents depend on the network of inter-fibre capillaries for fluid flow. Both of the structures provide large surface areas and help quick oil uptake.

Natural Fibres

Natural fibres are fibrous materials obtained from renewable biosources via natural processes, and find wide applications in both textile and non-textile sectors, due to their renewability, sustainability and biodegradable nature. Plant-based fibres such as jute, cotton, hemp, flax, kenaf, sisal, coir, ramie, abaca, kapok, and nettle, together with animal-derived fibres including wool and silk, make up an important group of renewable fibres that contribute greatly in the global industries.

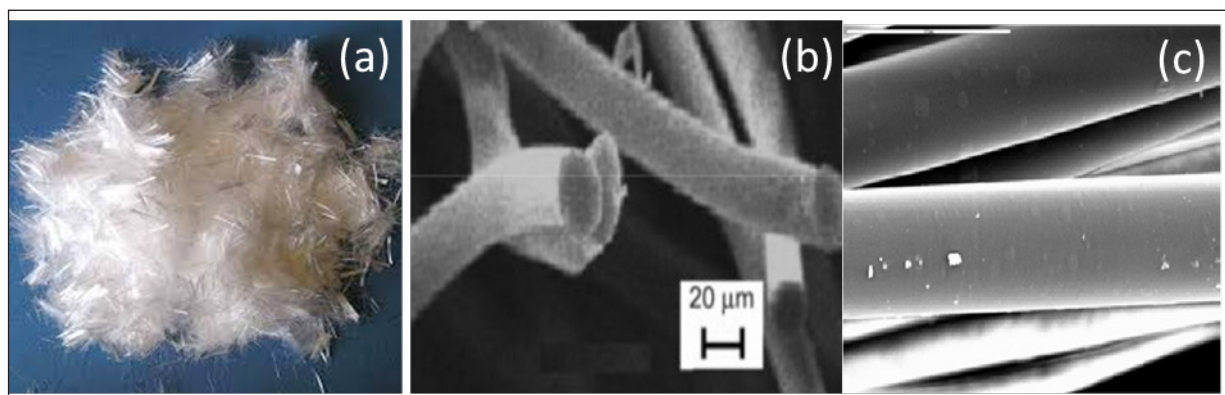


Fig. 4: (a) Polypropylene Fibre, (b) cross-sectional SEM analysis, (c) longitudinal SEM analysis

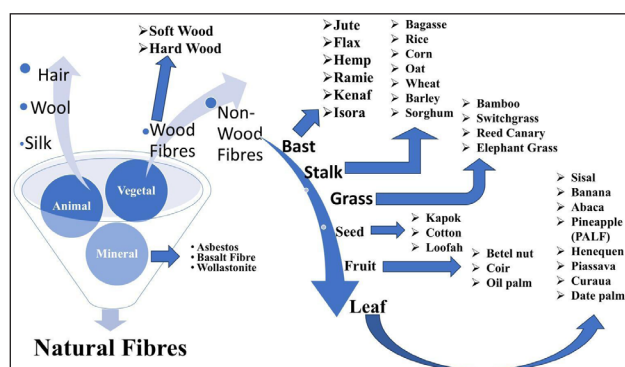


Fig. 5: Classification of natural fibers

Structural Characteristics

Natural fibres due to their porous structures, capillary networks, surface chemistry and low density, serve as suitable materials for oil uptake. Furthermore, materials derived from rice husk, sugarcane bagasse, peanut shells, coconut residues, corn stalks and various fruit wastes have shown varying degrees of oil absorption. Their performance depends on several factors including fibre composition and surface characteristics. The use of these agro-based products not only provides low-cost absorbents but also aids waste valorization, leading towards circular economy approaches. This has directed the use of the fibres beyond the mainstream textile industry towards other composite applications. The structure architecture has a great effect on the ability of oil absorption. The Kapok fiber has large lumen (accounting for ~77% of the fiber volume), outer diameter ~16.5 µm, inner diameter ~14.5 µm and thin cell wall (between ~0.8 – 1.0 µm). It gives very low density fiber of 0.29 g/cm³. The TEM investigation showed that the Kapok fiber has characteristic five layered

walls (compact cuticle, network, two layers of parallel fibrils and inner skin), which provides imperviousness to particles but permeability to oil. The water does not enter the lumen due to negative capillary entry pressure caused by contact angle >90° and high surface tension relative to air. Kapok absorbs ~1.5-2.0 times more oil than polypropylene mats. However, its absorptivity is decreased repeatedly owing to liquid bridge compression between the fibers and oil remains in the dead end lumen. The milkweed fiber (diameter 20-50 µm, wall thickness ~10% of the total diameter, mean 1.27 µm) has non-compressible large lumen and 3% of the surface wax – nearly seven times as much as in the cotton. Sorption of oil by cotton takes place through a variety of means: adsorption by hydrophobic interaction and Van der Waals forces between waxy surfaces and oil, physical retention by its rugged surface morphology, and capillary diffusion through the cuticle and lumen via fiber ends; while the amount of oil absorbed by the hydrophilic cellulose secondary wall is insignificant. The jute fiber possesses an organized bundle form, the banana fiber has an elementary fibril structure within a lignocellulosic matrix, the okra fiber has longitudinal fibril alignment, and the mesta fiber has fine longitudinal fibrils—multicellular structures affecting inter-fiber porosity rather than intra-fiber lumen sorption.

SEM Morphology

SEM micrographs show that bast fibers like jute, banana, okra, mesta share a common multicellular longitudinally aligned fibrillar theme but differ in their surface cleanliness. Jute reveals a comparatively smooth surface with longitudinal ridges, grooves,

and visible cementing material deposits. On the other hand, banana fibre displays surfaces heavily covered with irregular, flaky encrustations and continuous layers along the longitudinal axis, thus generating a bulky, rough appearance that greatly masks the underlying elementary fibrils and grooves. Okra's fibre surface appears relatively compact with some longitudinal ridges and scattered particulate deposits, its fibrillar structure is only partly covered by a slight coating. Mesta fibre is the smoothest out of the bast fibres, presenting a smooth and compact surface with fine longitudinal striations and only a few small adhering particles, and no thick encrustations. Seed fibres like kapok appears smooth, thin, cylindrical with uniform tubular structures and a smooth external cuticle that encloses a large hollow internal lumen, while cotton displayed unconvoluted, smooth and comparatively plump tubular fibers with only the characteristic natural convolutions. Malpura wool, an animal based protein fibre shows a distinct, highly patterned surface made of prominent, sharp and tightly packed overlapping cuticle scales.

Surface Chemistry

The surface composition plays an important role in deciding the oil sorption ability. The bast fibres include jute, banana, okra, and mesta, and they contain high levels of non-cellulosic components. The surface of jute consists mostly of lignin, hemicelluloses, and pectins, banana fiber contains natural waxes, pectins, lignin-hemicellulose matrix, and dirt, okra fiber consists of natural waxes, pectic

compounds, encrustation with lignin, and dirt, while mesta contains thin coatings of natural waxes, pectins, and other non-cellulosic components. In case of cotton, the surface consists mostly of cellulose with small amounts of waxes (0.448%), pectins, and proteins; removal of waxes on the surface using organic solvent like trichloroethane leads to a decrease of 20-30% in oil sorption, which means that only part of oil sorption can be explained with the help of wax-mediated hydrophobic interactions. The surface of kapok fiber is coated with natural plant waxes (wax cutin content is much higher compared to cotton), along with high content of acetyl groups (~13%), making it very hydrophobic. The Malpura wool is covered with thin epicuticles containing lipids, where 18-methyleicosanoic acid dominates.

Surface Wettability

The wettability of the surface determines the selective sorption of oil over water. Kapok and Malpura wool are hydrophobic and oleophilic because of the wax-cutin coat on kapok and high lipids in epicuticles on wool, making them highly efficient for selective oil sorption from aqueous media. Cotton is hydrophilic in nature due to its high cellulose composition but has some oleophilic nature due to the presence of the wax layer on its surface, so raw (grey) cotton has oil-sorbing property but with less selectivity than kapok. The bast fibres, i.e., jute (hydrophilic with lots of -OH group), banana (hydrophilic with cellulose composition and surface impurities), okra (hydrophilic in nature), and mesta (hydrophilic with lignocellulosic cellulose) are mainly hydrophilic in

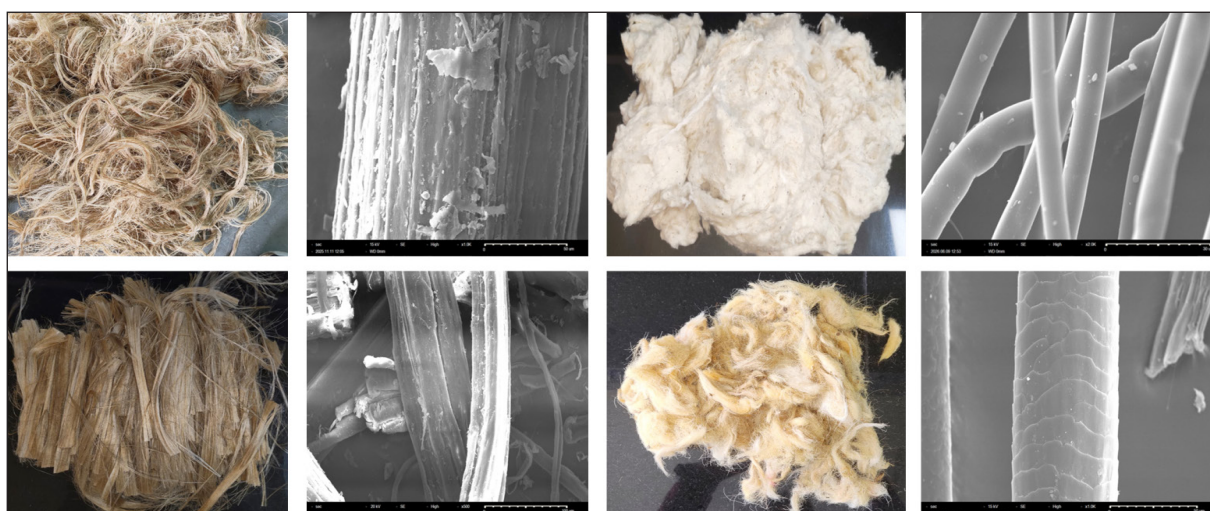


Fig. 6: SEM characterization of natural fibres, (a) Banana fibre, (b) Cotton fibre, (c) Jute fibre, (d) Malpura wool

nature, so they do not have any inherent selectivity towards oil.

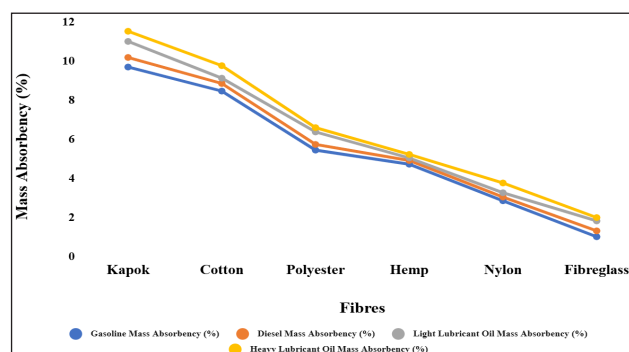


Fig. 7: Comparative oil absorbency of synthetic & natural fibres

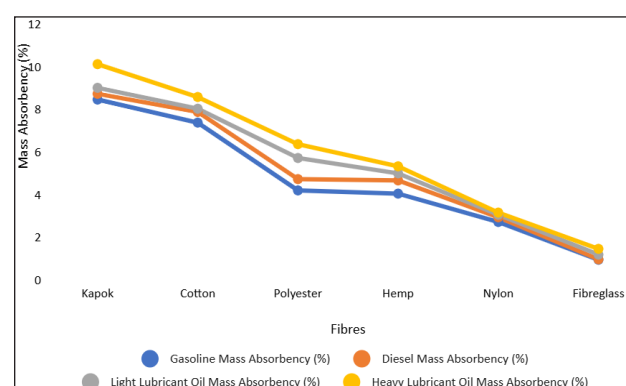


Fig. 8: Comparative oil absorbency of synthetic & natural fibres after recycling

Surface Modifications

The surface modifications have the principal objective of overcoming the internal drawbacks of the natural sorbents by modifying their physico-chemical properties without degrading their fundamental cellular architecture.

(a) Physical Modification

This represents one of the earliest and most environmentally safe procedures deployed to improve the oil uptake behaviour of the natural fibres. In contrast to the chemical treatments that change the molecular structure of the inherent cellulose by covalent interactions, the physical modification mainly alter the external morphology, surface roughness, pore structure, fibre dimensions as well as the internal capillary networks while simultaneously retaining the chemical composition of the fibre material. Consequently, the treatments enhance the accessibility of the oil molecules to the surface of the natural sorbent without adding chemical reagents or producing toxic chemical wastes. Due to the simplicity, comparatively low procedural cost, and minimum environmental damage, this technique of modification has emerged as a suitable sustainable preparation of the oil sorbents. Oil absorption performance of

Table 2: Comparative oil absorbency of synthetic and natural fibres

Fibre Name	Gasoline Mass Absorbency (%)	Diesel Mass Absorbency (%)	Light Lubricant Oil Mass Absorbency (%)	Heavy Lubricant Oil Mass Absorbency (%)
Kapok	9.7	10.19	11.01	11.53
Cotton	8.47	8.86	9.13	9.77
Polyester	5.45	5.74	6.38	6.6
Hemp	4.73	4.92	5.05	5.23
Nylon	2.86	3.05	3.27	3.77
Fibreglass	1.02	1.32	1.83	2

Table 3: Comparative oil absorbency of synthetic & natural fibres after recycling

Fibre Name	Gasoline Mass Absorbency (%)	Diesel Mass Absorbency (%)	Light Lubricant Oil Mass Absorbency (%)	Heavy Lubricant Oil Mass Absorbency (%)
Kapok	8.47	8.74	9.02	10.13
Cotton	7.39	7.89	8.04	8.59
Polyester	4.21	4.74	5.73	6.38
Hemp	4.06	4.68	5.01	5.34
Nylon	2.73	2.95	3.07	3.17
Fibreglass	0.96	0.98	1.2	1.47

the fibres are majorly regulated by their physical architecture. The parameters like fibre diameter, particle size, bulk density, pore distribution, capillary channels, lumen structure and surface roughness collectively decide the extent and rate of oil uptake. The oil enters the fibre mainly via capillary forces and hydrophobic reactions, before they are stored in the interconnected porous network. As a result, improved available surface area, enhanced accessibility of the internal pores can considerably increase the sorption efficiency without the modification of their intrinsic chemistry. The physical treatments have the principal objective to maximize the effective structural properties while maintaining the inherent renewable and biodegradable nature of the sorbent.

One such simple modification strategy is to reduce the particle size of the fibre by milling or grinding method. The reduction of the mechanical size of the natural fibre enhances the specific surface area and exposes a larger number of sorption sites by breaking the larger fibre bundles to finer particles. The small particles have shorter diffusion pathways and better contact with the oil phase, thus aiding quicker absorption kinetics and greater oil uptake. Moreover, grinding disrupts the compact arrangement of the fibres, thereby allowing the oil to penetrate more efficiently into the internal capillary networks and pores that were previously out of reach. The benefits of this mechanical method have been observed in the studies involving agricultural biomass. Sugarcane bagasse of different particle sizes and fractions was investigated and it was concluded that lowering particle size remarkably enhanced oil removal performance (Bayat *et al.*) The small fine bagasse particles offered better interaction between the sorbent material and spilled oil. Studies on corn stalk fibres reached the same conclusion, as finely separated fibres reportedly demonstrated better sorption due to increased surface area accessibility, and increased capillary action (Husseien *et al.*). These researches suggest that this simple physical treatment can substantially enhance oil uptake. Despite size reduction usually improving sorption efficiency, the small particles may also be subjected to certain drawbacks. On excessive grinding the mechanical integrity of the natural sorbent is lost, thereby its recovery after oil sorption is found to be difficult. The fine

powder forms are susceptible to dispersion by the water or wind currents and might need additional containment systems during practical applications. Furthermore, highly compacted fine particles tend to lower inter-particle porosity, obstructing oil flow into the internal structure in spite of their external surface area. Thus an optimum particle size has to be maintained to acquire a balance between high surface area, structural stability, sufficient porosity and recovery.

Another widely used physical alteration includes thermal modification of the fibres. Regulating the heating changed the physical structure of the sorbent biomass by removing moisture, volatile compounds and some low molecular weight impurities while maintaining the overall framework of the fibres. The degree up to which the fibre is modified depends on the treatment temperature, duration of heating, and the surrounding atmosphere. The low temperature thermal modification, primarily includes oven drying that mainly removes the physically adsorbed water and volatile impurities from the surface of the fibre. The water molecules tend to occupy the possible absorption sites and thereby restrict pore openings, the removal of the moisture helps improve the accessibility of the oil molecules on the fibre surface. It was reported through studies that drying does not change the inherent hydrophobic-lipophilic property of the fibre, however it increases oil uptake by eliminating the contaminants on the surface that interfere during the interaction of the oil and fibre interfaces (El Gheriany *et al.*). As a result, the dried fibres demonstrate quicker oil uptake than the untreated ones. Remarkable improvements were noted through high temperature thermal treatments or by the process of pyrolysis. Pyrolysis involves the lignocellulosic fibres undergoing a thermal decomposition under limited-oxygen conditions, thus producing carbon-rich porous architecture with improved surface roughness and notably larger pore volumes. The decomposition of hemicellulose, cellulose, and lignin releases certain volatile materials and leaves behind an interconnected carbonaceous framework that is adorned by multiple micro as well as mesopores. The porous structure considerably enhances oil storage while lowering the hydrophilic character of the original material simultaneously. Researchers have exhibited the effectiveness of pyrolysis in



improving the oil uptake behaviour. Prepared white rice husk ash and brown rice husk ash by pyrolysis, have been observed to have greater oil sorption capacities compared to the untreated ones (Vlaev *et al.*). Rice husk carbonized in the presence of carbon dioxide at 800° showed substantially high oil sorption for crude oil (Kudaybergenov *et al.*). Pyrolyzed rice husk demonstrates varying oil uptake depending on the viscosity, following the order of : heavy crude oil > motor oil > light crude oil > diesel > gasoline. It was observed that oils with greater viscosity were retained more efficiently compared to the less viscous ones, as their slow flow decreased the drainage losses. Thermal treatments have been found effective for agricultural residues as well. Carbonized barley straws showed higher hydrophobicity and greater oil uptake than the conventional propylene sorbents while simultaneously retaining their reusability for two sorption-desorption cycles. These researches exhibit that properly carbonized biomass can compete with the conventional synthetic sorbents.

Plasma treatment is a selective surface modification procedure that allows considerable alterations in surface energy, wettability, roughness, and interfacial behaviour while retaining the lightweight and porous architecture of the fibres. Plasma is an ionized gas that has energetic electrons, radicals, ions and excited species that is capable of interacting with the surface of the fibres. During this treatment, the energetic species bombard natural fibre surfaces, thereby causing cleaning, activation, etching and functionalization of the outer layers. This process can eliminate the surface waxes, impurities, and weak boundary layers while creating micro- and nano-scale roughness at the same time, which increases the effective surface area that is available for oil adsorption. Relying on the type of plasma gas that is used, additional functional groups might also be added to the fibre surface.

(b) Chemical Modification

This is one of the most extensively investigated strategies for enhancing the behaviour of the natural fibres in oil uptake and other hybrid applications. In contrast to the physical treatments, that primarily involve alteration of the surface morphology, the chemical methods cause permanent alterations in the chemical composition and surface functionality

of the cell wall of the fibre. The natural fibres comprise of hemicellulose, cellulose, pectin, lignin, waxes and other such extractives (Kabir *et al.* 2012). The abundance of hydroxyl groups in the hemicellulose and cellulose impart a hydrophilic nature to the fibres, leading to high water absorption, poor compatibility with hydrophobic materials, dimensional instability, and poor affinity to hydrocarbons (Li *et al.* 2007). These in-built characteristics significantly limit the efficiency of the unmodified fibres in selective oil uptake and oil-water separation processes. The chemical modifications are aimed at overcoming these drawbacks by changing or substituting the -OH groups on the surface of the fibre, thus lowering hydrophilicity while improving the lipophilicity and surface compatibility with non-polar substances (Samanth & Bhat, 2023). Relying on the type of treatment that is deployed, chemical treatments can also eliminate amorphous constituents like hemicellulose, pectin, lignin, oils, waxes, and other impurities that layer the fibre surface. The removal of the non-cellulosic components enhances surface roughness, exposes the other active sites, improves accessibility to the pores, enhances fibre wettability to oils and aids stronger interfacial interactions with the hydrophobic materials (Li *et al.* 2007). As a result, the chemically modified fibres usually demonstrate higher oil uptake capacity, enhanced oil storage, higher buoyancy, improved mechanical stability, and better reusability compared to the untreated fibres (Ramachandran *et al.* 2022).

Alkali Treatment (Mercerization)

Alkali Treatment which is more commonly termed as mercerization, is a simple economical and extensively researched modification technique for natural fibres. It includes treating the fibres with an aqueous solution of sodium hydroxide that eliminates the non-cellulosic contents - hemicellulose, pectin, lignin, oils, waxes, and other surface impurities (Goda & Cao, 2007). These constituents add to the hydrophilic nature of the fibres, hence their elimination contributes to the exposure of the cellular microfibrils, and creates a cleaner as well as rougher surface of the fiber (Santos *et al.* 2018; Hassan *et al.* 2014). This method transforms the native crystalline form of the cellulose present in the fibers by their surface

morphology and fiber crystallinity. The elimination of the amorphous substances and impurities leads to increased surface roughness, fibrillation, enhanced pore volume and more accessibility to the internal lumen (Gulati *et al.* 2014). These structural modifications widens the effective surface area for oil uptake and aids capillary penetration of oil within the fiber assembly. The scanning electron microscopy studies have demonstrated that the mercerized fibers have rough, irregular surfaces in comparison to the smooth surfaces of the raw fibers, thus providing more sites for oil storage and stronger physical interlocking (Li *et al.* 2007, Sidik *et al.* 2014). As a result, the alkali-treated fibers in general show enhanced oil affinity and enhanced oil uptake efficiency while lowering water absorption. Scientists have found that alkali treatment improved the crude oil uptake ability of soybean residue, while mercerized cattail fibres acquired high motor oil sorption (Wong *et al.*). Similarly it was reported that NaOH treated rice husk showed substantially improved oil uptake for marine diesel (Bazargan *et al.* 2014). The improved oil remediation behaviour was majorly attributed to lowered bulk density, easier oil diffusion into fiber structure, and improved internal porosity (Li *et al.* 2007). Consequently it could be concluded that alkali treatment has a significant role in improving the oil sorption performance. Apart from improving oil uptake, alkali treatment makes the fibers more compatible with hydrophobic polymers, allowing them to be used together in composite fabrication (Bazargan *et al.* 2014). The elimination of the weak boundary layers and improved surface roughness promotes strong fiber-matrix adhesion and lowers moisture sorption, thus enhancing structural stability and durability of the fiber-based substances (Santos *et al.* 2018). The simplicity, effectiveness, and low cost of this treatment has led to the extensive use of this treatment as a sole surface modification and might sometimes be followed by other alterations, such as acetylation, silane treatment or graft polymerization (Gulati *et al.* 2014).

Silane Treatment

Silane treatment is one of the most effective chemical alteration strategies for enhancing the hydrophobicity and interfacial characteristics of the natural fibers. The silane coupling agents have

bifunctional groups that can react with the -OH groups on the cellulose surface while simultaneously interacting with the surrounding organic matrix or the hydrophobic functional groups. This dual functionality allows the silanes to perform as molecular bridges thus enhancing compatibility of the fiber with non-polar materials and improving lipophilicity (Xie *et al.* 2010, Atiqah *et al.* 2017). An alkali pretreatment is usually deployed before silane treatment to eliminate the surface impurities and expose more reactive -OH groups causing more effective silanization. During silane grafting, the hydrolysable alkoxy functional groups in the silane molecule are converted to silanol groups, that later condense with the -OH groups of cellulose to form the stable Si-O-cellulose covalent bonds (Zhou *et al.* 2013). At the same time, the condensation between the adjacent silanol functional groups generates a cross-linked siloxane network on the surface of the fiber. This chemically bonded coating remarkably lowers the available free -OH groups responsible for water uptake while introducing hydrophobic groups that improves oil affinity (Abdelmouleh *et al.* 2005). Consequently, the silane-treated fibers demonstrated low moisture sorption, enhanced mechanical stability and more resistance to microbial degradation. Research shows that silane treatment considerably enhances oil uptake performance by increasing surface roughness, improving selective oil uptake and lowering polarity (Puglia *et al.* 2013). The modification also enhances fiber-matrix adhesion in polymeric composites, resulting in superior morphological properties and long term durability. The silane-treated fibers usually maintain their structural integrity during repeated oil absorption cycles, producing suitable reusable sorbent materials for oil spill remediation (Atiqah *et al.* 2017). Organosilanes like aminosilanes, methacryloxy silanes, vinyl silanes and long chain alkyl silanes including OTES (Octyltriethoxysilane), and DTES (Dodecyltriethoxysilane) have emerged as suitable silanization agents (Abdelmouleh *et al.* 2005). Since this modification permanently alters fiber surface while keeping the cellulose framework almost intact, it has become a widely used surface modification technology for natural fiber oil sorbents and environmentally safe composite materials (Zhou *et al.* 2013).



Acetylation

Acetylation is another chemical treatment that improves the hydrophobicity of the natural fibers by substituting the hydrophilic -OH groups of the cellulose with acetyl (-COCH₃) functional groups by reaction with acetic anhydride. The replacement of -OH groups lowers the polarity of the surface of the fiber thus lowering water uptake while improving affinity to non-polar hydrocarbons simultaneously (Sun *et al.* 2002). Due to the -COCH₃ groups having less tendency to form H-bonds with water molecules, the acetylated fibers have become relatively more appropriate for oil spill remediation scenarios. Along with altering the fiber chemistry, acetylation also changes the physical properties of the natural fibers (Nawdiogbu *et al.* 2014). The substitution of the -OH groups with bulkier -COCH₃ groups improves the fiber volume, enhances the pore accessibility and increases capillary action in the fiber lumen. These structural alterations lead to rapid oil penetration and more oil storage while retaining buoyancy (Deschamps *et al.* 2003). The extent of alteration has been expressed in terms of weight percent gain (WPG). Higher WPG generally indicates greater hydrophobicity and enhanced sorption behaviour. Studies have reported effectiveness of acetylation in enhancing oil uptake. Acetylated rice straw demonstrated high oil uptake capacities, in comparison to the low values seen in case of the untreated fibers (Li *et al.* 2013). Even in the case of cotton fibers the acetylated fibers exhibited higher oil uptake and reusability. Acetylated corn straw fibers have reached excellent oil sorption for crude oil, diesel oil and vacuum pump oils. The kapok fibers showed significant increase in diesel oil uptake due to the contribution of surface roughness as well as opening of the fiber lumen. Along with improving oil sorption performance, this treatment enhances the dimensional stability, resistance to aqueous moisture and durability of the fibers (Wang *et al.* 2013).

Esterification

Esterification is a well-established chemical surface modification process in which the -OH groups on the cellulose interact with organic acids or their derivatives to form ester linkages. In contrast to acetylation that specifically introduces acetyl groups, esterification includes the integration of

a wide range of functional groups depending on the esterifying agent that is deployed (Abdul *et al.* 2012). This flexibility allows the researchers to alter the surface chemistry of the natural fibers in accordance to their desired use. The formation of the ester bonds lowers the number of free -OH groups available for H-bonding with water thus lowering the fiber hydrophilicity and increasing the hydrophobicity. At the same time, esterification may add long hydrocarbon chains onto the surface of the fiber, substantially improving the oil affinity and helping the selective uptake of petroleum hydrocarbons (Ramachandran *et al.* 2022). The modification might also influence the surface energy, fiber crystallinity, and pore accessibility, all of these contribute to the improved oil uptake performance (Xie *et al.* 2010). Multiple esterification agents including stearic acid, fatty acids, succinic anhydride, maleic anhydride and long chain organic acid derivatives have been studied for altering agricultural fibers. These modifications generate fibers with enhanced oil-water selectivity, improved buoyancy, less moisture absorption and better morphological stability during multiple cycles of use. The integration of hydrophobic carbon chains is especially useful for improving affinity towards crude oil and petroleum products. In contrast to the untreated fiber, esterified fibres, like esterified banana fiber, were observed to show greater durability in aqueous environments and enhanced compatibility with hydrophobic matrices. As a result, esterification has evolved as an effective method for developing environmentally sustainable oil sorbents with long term operational stability and improved oil remediation performance (Xie *et al.* 2010).

Graft Polymerization

Graft polymerization is an advanced chemical modification technology in which synthetic polymer chains are chemically incorporated onto the surface of the natural fibers while retaining the inherent cellulose backbone (Zhou *et al.* 2016). The incorporated polymer adds new functional groups that change the surface chemistry, improves hydrophobicity, and increases compatibility with the hydrophobic liquids like crude oil and petroleum products. In contrast to the surface coating procedure, this grafting method forms

covalent bonds between the polymer chains and cellulose, producing highly stable surface modification (Teli *et al.* 2017; Shi *et al.* 2019). Grafting is generally initiated by free-radicle polymerization using suitable initiators, causing monomers like methyl methacrylate, butyl acrylate, styrene or acrylic acid derivatives to polymerize directly onto the fiber surface. The resultant polymer layer coats the hydrophilic cellulose surface and improves oleophilicity while preserving the fibrous structure required for capillary oil transport (Shi *et al.* 2019). SEM analysis revealed a rough, polymer coated surface with improved fiber diameter and improved surface irregularities both of which are responsible for enhanced oil uptake (Viju *et al.* 2019). Researches have shown substantial enhancements in sorption behaviour after graft polymerization (Said *et al.* 2019; Meng *et al.* 2015). Grafted banana fibers along with grafted coir fibers have developed improved oil sorption capacities. Graft modification also improves physical strength, dimensional stability and resistance to continuous sorption-desorption cycles, resulting in grafted fibers being suitable for reusable oil recovery systems (Teli *et al.* 2016). Despite the graft polymerization usually being more a complex process and expensive than the conventional chemical modifications, it provides great control over fiber surface characteristics (Said *et al.* 2015). As a result, it is extensively being explored for development of high performance natural sorbents and selective oil-water separation.

(c) Biological Modification

This is a field of environmentally suitable and evolving approaches for enhancing the surface properties of natural fibers. In contrast to the chemical modifications that include the utilisation of strong reagents or energy-extensive processing. Biological alterations mainly deal with enzymes, microorganisms, or naturally derived biomaterials to change the surface of the fiber under mild operating conditions. This method is thus regarded as green technology as it lowers the use of chemicals, minimizes secondary pollution and retains the internal biodegradability of the fibers. As a result, the biological treatment has gathered attraction as a developing technique for eco-friendly fiber reinforced hybrids (Li *et al.* 2020). The natural fibers consist of cellulose that is embedded in a matrix

of lignin, hemicellulose, waxes, pectin and other non-cellulosic extractives. Biological modification selectively eliminates the amorphous constituents without leading to any extensive damage to the cellulosic architecture. Enzymes like pectinases, cellulases, laccases, xylanases, and lignin-degrading enzymes are commonly deployed to clean the surface of the fiber, improve surface roughness and expose the additional cellulose microfibrils. In comparison to the conventional modifications, the enzymatic treatments provide more selectivity, provide controlled surface alteration while maintaining structural integrity of the fibers and fiber strength.

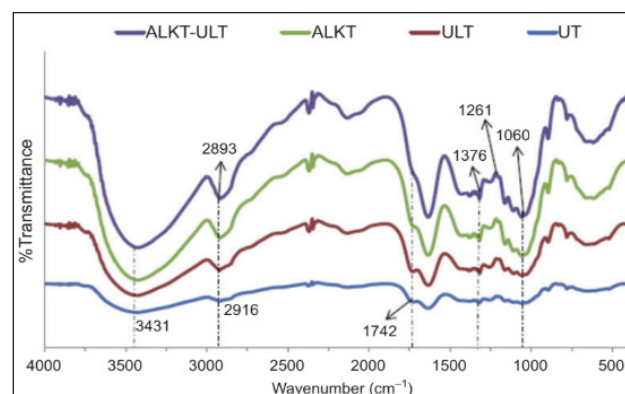


Fig. 9: FTIR spectroscopy of untreated (UT), ultrasound treated (ULT), alkali treated (ALKT) & blend of alkali- and ultrasound-treated (ALKT-ULT) sisal fibres

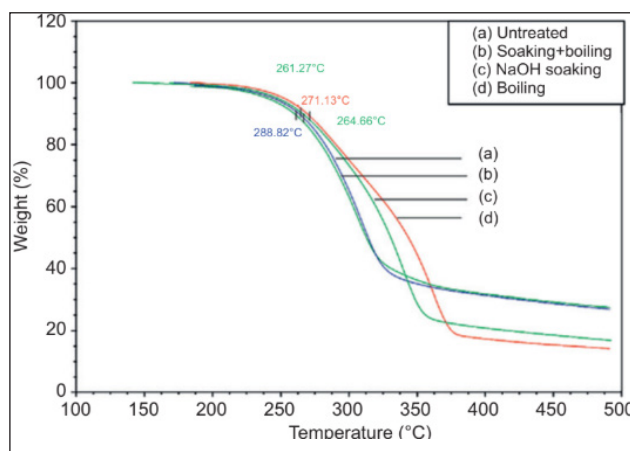


Fig. 10: Thermogravimetric analysis of oil palm fibres, (a) untreated, (b) combination of NaOH and boiling water, (c) NaOH treated, (d) boiling water treated

One of the most widely used biological treatment techniques is the deposition of bacterial cellulose on the surfaces of the natural fibers. Research demonstrated that bacterial cellulose formed during microbial fermentation can be directly deposited onto hemp and sisal fibers, thereby producing a

thin nanofibrillar coating around the fibers (K. Lee *et al.* 2011).

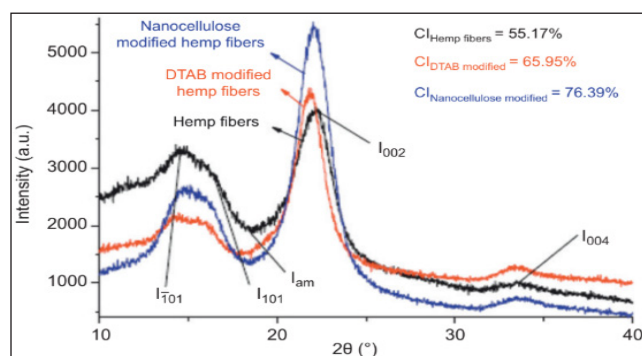


Fig. 11: X-ray diffractogram of unmodified and modified hemp fibres with dodecyltrimethylammonium bromide (DTAB)

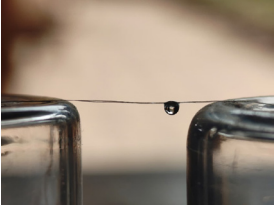
Nearly 5-6% bacterial cellulose deposition substantially improved the interfacial adhesion between fibers and polymer matrices like polylactic acid and cellulose acetate butyrate. The deposited

cellulosic nanofibrils improved the surface roughness and accessible bonding sites while preserving the biodegradable nature of the natural fibers, leading to composites with superior mechanical performance. Other than enhancing composite characteristics, biological treatments also offer advantages of oil uptake applications. Elimination of surface impurities improves fiber porosity and exposes additional adsorption sites, thus helping increased oil penetration via capillary action. The improved surface morphology provides greater interaction between the fiber surface and hydrocarbons while lowering the tendency of fibers to hold excessive amounts of water. Moreover, biological alterations prevent the utilization of harsh chemicals that may degrade the fiber structure or produce hazardous waste. Consequently the process is environmentally sustainable for large-scale production of natural oil sorbents.

Table 4: Surface modifications of natural fibres

Natural fibre	Surface modification	End result	Reference
Cotton	Grafting fluorocarbon monomer	Improvement in hydrophobicity	Ramamoorthy <i>et al.</i> (2013))
Cotton	Coating of ZnO & carboxymethyl chitosan	Higher antibacterial properties & UV resistance	Wang <i>et al.</i> (2016)
Flax	Grafting of nano-TiO ₂ in presence of silane coupling agent	Improvement of thermomechanical/mechanical properties	Wang <i>et al.</i> (2015)
Flax	Grafting of glycidyl methacrylate (GMA)	Enhanced hydrophobicity & mechanical strength	Musaad <i>et al.</i> (2016)
Hemp	Alkali treatment using NaOH and Na ₂ SO ₃ *	Increased tensile strength, crystallinity index & thermal stability	Islam <i>et al.</i> (2011)
Hemp	Carbonization followed by KOH activation	Increased specific surface area & sorption characteristics	Mijailović <i>et al.</i> (2017)
Jute	NaOH treatment	Tensile strength improvement	Roy <i>et al.</i> (2012)
Jute	Pyromellitic dianhydride (PMDA) grafting/modification	Enhanced adsorption capacity	Gao <i>et al.</i> (2015)
Ramie	Amino silicone oil treatment	Increased hydrophobicity	He <i>et al.</i> (2015)
Kenaf	Esterification using citric acid	Greater adsorption capacity	Sajab <i>et al.</i> (2011)
Bamboo	Silane coupling agent	Improvement in mechanical & thermal properties	Zhang <i>et al.</i> (2020)
Coir fibre	Grafting of ethylene glycol dimethacrylate (EGDMA)	Enhanced physicochemical/mechanical properties	Rahman & Khan (2007); Roy <i>et al.</i> (2012)
Oil palm fibre	Treatment with citric acid and PEI individually	Increased adsorption capacity	Sajab <i>et al.</i> (2013)
Sisal	Combined NaOH and clay treatment	Enhanced mechanical, thermal & adhesion properties; water resistance	Mohan & Kanny (2012)
Sisal	Sodium bicarbonate treatment	Increased tensile strength	Sanfilippo <i>et al.</i> (2024)
Silk	Grafting/deposition of nano-Ag	Antibacterial characteristics	Zhang <i>et al.</i> (2014)
Wool	Grafting of chitosan	Antibacterial & antifelting properties	Mohammadi <i>et al.</i>
Wool	Nano-ZrO ₂ coating	Higher thermal stability	Gashti <i>et al.</i> (2013)

Table 5: Hydrophobicity induced in the fibres after surface modification

Material	Fiber Morphology	Droplet Behavior	Estimated Contact Angle	Scientific Analysis
Jute 	Single suspended filament	Symmetric barrel shape completely enveloping the fiber	$< 90^\circ$	Preferential wetting of a 1D cylinder to minimize surface energy; requires inflection point calculations for exact measurement rather than 2D baselines.
Malpura Wool 	Fluffy web	Highly spherical, resting on top of the web	$> 130^\circ$	Highly hydrophobic; droplet sits in a Cassie-Baxter state, suspended by air trapped within the microscopic pockets. Natural lanolin contributes to the high contact angle.
Cotton 	Fluffy web	Highly spherical, resting on top of the web	$> 130^\circ$	Highly hydrophobic; likely raw or unscoured cotton that still retains its natural cuticular waxes, allowing the droplet to remain suspended in a Cassie-Baxter state.

Measurement of Contact Angles

The contact angle (θ) represents the most basic parameter of wettability; it is measured geometrically as the angle formed by the liquid at the three-phase interface.

It is dictated by the thermodynamic balance of three interfacial tensions, explained by **Young's equation** for contact angle:

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cos \theta$$

Where:

γ_{SV} = solid–vapour interfacial energy

γ_{SL} = solid–liquid interfacial energy

γ_{LV} = liquid–vapour interfacial energy (surface tension of the liquid)

θ = contact angle between the liquid droplet and solid surface

Types of Wetting

Hydrophilic ($\theta < 90^\circ$): The liquid spreads over the surface as the solid surface energy is very high.

Hydrophobic ($\theta > 90^\circ$): The liquid forms spheres on the surface since the solid surface energy is low.

Superhydrophobic ($\theta > 150^\circ$): The liquid droplet takes almost spherical shape and rolls down easily.

Future Perspectives

Modified natural fibres provide an excellent opportunity to achieve inexpensive, biodegradable, and sustainable oil spill sorbents. In future, emphasis needs to be put on greener and scalable surface modification techniques including plasma, enzymatic, mild esterification, biodegradable coating, and controlled carbonization to enhance hydrophobicity, oleophilicity, buoyancy, and reusability without affecting biodegradability. Agro-waste materials such as rice husk, bagasse, coir, corn stalk, and fruit residues can be transformed into value-added sorbent materials for circular economy. Further, research efforts are required to explore nonwoven fibrous mat, boom, pad, aerogel and composite materials. Integration of experimental investigations with modeling and life cycle analysis is vital in commercializing such oil sorbents for practical applications.



Future Challenges

Natural fiber sorbents are faced with several challenges despite their promising nature. Natural fiber sorbents have varying characteristics such as composition, morphology, and surface chemistry due to factors such as geographical location and processing techniques; this creates inconsistency in performance. Most natural fibers are hydrophilic in nature; hence, they tend to take in water. Treatment of the natural fibers to enhance oil sorption comes at a higher cost and introduces toxicity. The maintenance of physical integrity and buoyancy when exposed to continuous sorption-desorption is still challenging. Testing under practical conditions including waves, salinity, water temperature, oil viscosity, weathering, and emulsions needs to be done. Testing methods for oil sorption, water absorption, reusability, and biodegradability have yet to be standardized.

CONCLUSION

The use of modified natural fibres is a sustainable and cost-effective replacement for synthetic oil sorbent fibres. The low density, porosity, capillary structure, renewability and biodegradability of modified natural fibres make them applicable for oil spills management. In the list of modified natural fibres studied, kapok and cotton are known for their high oil sorption capacity whereas bast fibres such as jute, banana, hemp and okra need surface modification because of their hydrophilic nature. Sorption of oil in fibres is greatly influenced by fibre structure, presence of surface waxes, fibre lumen accessibility, fibre surface roughness and fibre wettability. Physical, chemical and biological modifications of the fibres will improve fibre hydrophobicity, oleophilicity, floatability, retention capacity and reusability. It is important to find the right balance between the environment, friendliness of modifications, cost-effectiveness and applicability. In future research, there is a need to focus on evaluation, testing of fibres on actual oil spillage sites, re-generation, oil recovery and life cycle assessment.

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