Impact of Fiber Treatment on the Oil Absorption Characteristics of Plant Fibers

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Most plant fibers are good sorbents of oil; however, synthetic sorbents have a much higher sorption capacity (SC) than plant fibers. This study evaluated the effect of fiber treatments, specifically hot-water treatment and mercerization, on the absorption characteristics of selected plant fibers. Five common plant fibers—corn residues, soybean residues, cotton burr and stem (CBS), cattail, and oak—were evaluated for their absorption characteristics in crude oil, motor oil, deionized (DO) water, and a 80:20 mix of DO water. The fiber treatments included ground fiber (control), hot-water treatment at 80 °C for 4 h and 125 °C for 4 h, mercerization at room temp for 48 h, and mercerization at 300 °C for 1 h. The absorption capacity (AC) varied with fiber type, absorption medium, and fiber treatment. Mercerization at 300 °C increased the water absorption of soybean residue up to 8 g/g. Mercerization at room temperature and the hot-water treatment at 125 °C increased the crude oil absorption capacity. After certain treatments, the crude oil absorption capacity of CBS and corn fibers increased over 5 g/g, and the motor oil absorption capacity of cattail, corn, and soybean also increased to 4 to 5 g/g.

Keywords: Plant fiber; Mercerization; Hot water treatment; Absorption; Oil; Water

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INTRODUCTION

The agricultural industry is a major consumer of lignocellulosic residues that are available for use in value-added products. The global production of agricultural residues from wheat straw, corn straw, rice straw, and sugarcane bagasse is estimated at 1.4 billion tons (Saini *et al.* 2015). In the U.S., approximately 244 million dry tons of agricultural biomass residues and wastes are available for bioenergy and byproduct applications every year (DOE 2011). There is a vast potential for developing new products and applications for these agricultural residues. Lignocellulosic fibers are good sorbents of oil, and the application of plant fibers as sorbent materials has great potential.

In the last decade, there have been many major oil spills that have resulted in considerable environmental damage despite clean-up efforts. The recent incidents of shale oil spills from the Bakken formation of North Dakota have created many problems for oil clean-up (Inforum 2015). Currently, materials such as absorbents, dispersants, solidifiers, booms, and skimmers are being used for oil spill remediation (Nguyen *et al.* 2013). Absorbents function by completely absorbing the oil, thereby facilitating its removal from the medium (typically water) or spill area. In contrast, dispersants break down oil into smaller particles for its subsequent removal. The most commonly used absorbents include polypropylene, polyethylene, and polyurethane foams, although cellulosic materials and other natural materials, such as minerals, are also good sorbents (Deschamps *et al*. 2003;

Hubbe *et al.* 2013). The oil sorption capacity (SC) of polypropylene is reported as 4.5 to 10 times its weight, and that of urethane polymer is reported as 34.4% (Adebajo *et al.* 2003). Despite favorable oil absorption characteristics, synthetic polymers are controversial because they are not renewable or biodegradable. Also, heavy oils are shown to degrade 5 to 7 times faster in the presence of natural sorbents (Setti *et al.* 1999). A partial list of organic fibers, along with a few polypropylene, polyvinyl chloride, and carbon aerogel examples of sorbent materials and their SCs in grams of sorption medium per gram of fiber (g/g) are listed in Table 1.

Table 1. Primarily Organic Sorbent Materials and their Sorption Capacity (SC)

Sorbent Material	Sorption capacity (g/g)	Form	Sorption Medium	Reference
Polypropylene	3-4.5	Nonwoven web	Light cycle oil light gas oil Iranian Crude Oil	Teas *et al.* 2001
Cellulosic fiber	2-5	Wood chips		
Kapok	10-50	Packed fibers	Diesel, engine oil	Abdullah *et al.* 2010
Recycled wool		Nonwoven mat	Base oil SN150, Diesel, Crude oil	Radetic *et al.* 2003
Milkweed fiber	10-45	Fiber	No. 2 fuel oil, light crude oil, No. 6 fuel oil	Choi *et al.* 1992
Cotton fiber	10-30	Fiber		
Cotton grass	20	Fiber mat	Crude oil	Suni *et al.* 2004
Polypropylene	5-15	Polymeric and cellulose fiber	Liquid hydrocarbons	Wiegand *et al.* 1978
Kenaf	4-5	Fiber, woody core	Crude oil	Choi *et al.* 1992
Sisal	3-6.4	Granular	Crude oil	Annunciado *et al.* 2005
Saw dust	4.1-6.4	Granular	Crude oil	
Silk floss	74-85	Floss fiber	Crude oil	
Cotton, treated	20	Flat cake	Oil, petroleum, fuel	Deschamps *et al.* 2003
Polyvinyl chloride/polystyrene	38-146	Electrospun fibers	Motor oil, diesel, peanut oil, glycol	Zhu *et al.* 2011
Rice husks	20	Husks	Diesel Fuel	Bazargan *et al.* 2014
Populus seed fibers	55-182	Seed hair fibers	Motor oil, Diesel fuel	Likon *et al.* 2013
Cellulose aerogel	18-24	Porous sheets	Crude oil	Nguyen *et al.* 2013

The sorption process involves the absorption and adsorption of liquid medium by fibers. Absorption refers to the dissipation of the medium throughout the bulk of the solid, whereas adsorption refers to the accumulation of atoms, ions, or molecules of the medium to the surface of the sorbent material. Several organic and mineral sorbents have been reported and characterized for their SCs (Table 1). Plant fibers, such as cotton and milkweed, have high sorption capacities of 30 to 40 g/g, compared with polypropylene, with a SC of 10 g/g (Choi *et al.* 1992). The oil sorption of other plant fibers, such as sisal,

leaf residues, saw dust, coir fiber, and sponge gourd, varies from 2.7 to 6.4 g/g at a fiber granularity of 0.85 to 1.70 mm, while silk floss shows the highest sorption capacity of 85 g/g (Annunciado *et al.* 2005).

Plant fibers have many free hydroxyl groups at the molecular level that easily bond with oil or water (Bazargan *et al.* 2014). Therefore, they generally exhibit a good affinity for both oil and water. The oil SC of untreated plant fibers is generally less than polymers used in oil booms. The treatment of fiber is generally expected to enhance the affinity of fibers for oil. Fiber treatment usually refers to the physical, chemical, or thermal treatment of fibers, resulting in a modification of their characteristics. Oil sorption by plant fibers depends on many fiber-related factors, such as the size, shape, structure/arrangement (loose *vs*. mesh), treatment, and the conditions in which fibers are exposed to the oil.

Pretreatment processes can enhance the surface characteristics of natural fibers to enhance their SC. Fiber treatment methods, such as mercerization, chloroform treatment, acetylation, silane treatment, and hot water treatment, modify the fiber surface, composition, structure, dimensions, morphology, and mechanical properties (Rong *et al.* 2001). Mercerization in particular refers to the alkalization of cellulosic fibers with a sodium hydroxide solution (NaOH) (Han and Rowell 1997; Li *et al.* 2007; Amiri *et al.* 2015). Alkalization increases the surface roughness, thereby increasing the surface area of the cellulosic fiber. This aids in increasing the attachment sites for the sorption medium. Alkali treatment partially dissolves and removes hemicellulose in the fiber and slightly reduces the crystallinity, depending on the concentration and type of alkali treatment temperature and treatment time (Rong *et al.* 2001). The preferred oil sorbent should be highly hydrophobic and oleophilic, and various fiber treatments can alter these properties. Although raw cotton has a higher SC than treated cotton, the hydrophobicity of treated cotton is a major advantage in oil clean-up applications (Deschamps *et al.* 2003). A novel, high-capacity oil sorbent containing polyvinyl chloride (PVC)/polystyrene (PS) fibers shows sorption capacities for motor oil, peanut oil, diesel, and ethylene glycol of 146, 119, 38, and 81 g/g, respectively (Zhu *et al.* 2011). Highly porous cellulose aerogel prepared from paper waste cellulose fibers exhibits absorption capacities of 18.4 to 24.4 g/g (Nguyen *et al.* 2013).

The specific objective of this research was to evaluate the effect of two different fiber treatments, *i.e.*, hot-water treatment and mercerization, on the oil absorption characteristics of selected plant fibers. The evaluated hypothesis was that the sorption capacities of some fibers would be enhanced by the simple treatments to levels comparable to polyolefin based oil sorbents. The novelty of this work lies in the value addition of agricultural residue. Selective mild chemical and hot water treatments can improve the absorption capacity of fibers resulting in oil spill abatement applications. The results will further help to understand underlying mechanisms responsible for such variations.

EXPERIMENTAL

Materials

Fibers

The fibers used in this study were primarily of agricultural origin and included oak wood supplied by South Wood Services (Macon, GA), corn residue, soybean residue, and cattails were collected locally (Fargo, ND), and cotton burr and stem (CBS) were supplied by Cotton Production and Processing Research U nit, (Lubbock, TX). Oak fiber was used

as a control fiber. Corn residue included the stem, leaves, husks, and cobs after harvesting the corn grains. Soybean residues included the biomass remaining after harvesting the soybeans. The CBS is part of the waste stream from the cotton ginning operation, consisting of cotton carpels and stems. Cattail is an invasive plant found in wetlands and other wet areas and is often a nuisance to agriculture and ecosystems.

Table 2. Fiber Size and Bulk Density of the Plant Fibers (Han and Rowell 1997)

Fiber Source	Fiber Length (μm)	Bulk Density (kg/m^3)	Fiber Type
Oak	250 to 400	348.9	Woody
Cattail	250 to 875	89	Grassy
CBS	250 to 841	216	Woody-leaf
Corn	250 to 595	168	Grassy
Soybean	250 to 354	191	Woody-leaf

Methods

Fiber preparation

The fibers were first processed in a Wiley mill (Thomas-Wiley Mill Model 4, Philadelphia, PA) to produce a uniform size distribution of 0.400 to 0.841 mm (20 to 40 mesh). The fibers were gravimetrically tested to determine the moisture content and oven-dried at 105 °C for 24 h before treatments. After the treatment, the fibers were oven-dried to remove excess moisture and then they underwent oil adsorption testing. The physical property assessment showed that cattail fibers exhibited the lowest bulk density of 89 kg/m^3, which was one fourth the density of oak fibers. The bulk density was measured following ASTM D4781 protocol (Table 2).

Hot-water treatment

This study explored two different fiber treatment methods, namely hot water treatment and mercerization. The hot water treatment process removed volatile compounds, waxy coatings, and extractives from cellulosic fibers, making them more accessible for the absorption medium. Fibers were subjected to two hot-water treatment conditions at either 80 °C for 24 h or 125 °C for 24 h. Temperature conditions were selected based on existing knowledge that tannins, gums, sugars and starches dissolve in water at this temperature range. For hot-water treatment, dried fibers were immersed in deionized (DO) water in a shaker. After the 24-h immersion, the fibers were drained and dried at 105 °C for 24 h.

Mercerization

In this experiment, the mercerization treatments involved immersing and shaking fibers in 5% NaOH solution at the two treatment levels of 25 °C for 48 h and at 300 °C for 1 h. After the alkali treatment, the fibers were drained and oven-dried at 105 °C for 24 h.

The five types of fiber were subjected to four treatment mediums, *i.e.*, crude oil, motor oil, deionized (DO) water, and a mixture of 80% DO water and 20% crude oil, to determine the absorption characteristics. These mediums were selected to represent a range of oil properties and environments (Table 3). Three to five replicates were conducted to result in a total of 462 observations.

Characterization of absorption capacity (AC)

The ASTM F716-81 (2001) standard was used for characterizing the sorption characteristics of the fibers. The protocol was modified to quantify the absorption capacity (AC) of the fibers. Based on the standard, 1.0 g (W_1) of dried fiber was soaked in 20 mL of medium at 21 to 23 °C on a stirring hot-plate set at 98 rpm for 10 min. The fibers were removed from the medium on a sustainer and set aside for 15 min, followed by blotting to remove excess oil and subsequent weighing (W_2). The weight gain of the fiber (as a percent of initial) after soaking in the medium was calculated and represented the absorption capacity. The fibers that were treated with a mixture of oil and water underwent additional oven-drying to remove excess moisture, and they were weighed again to calculate the medium-specific AC of the fiber.

Data analysis

The absorption data were analyzed using JMP software (Version 11.0.2, SAS Institute, Cary, NC), and an analysis of variance (ANOVA) model was performed to evaluate the effects of fiber, treatment, and medium on the AC. The treatment means were compared using an unpaired *t*-test, and the means were separated using the Tukey-Kramer method of pairwise comparison. Significance was accepted at $P < 0.05$.

RESULTS AND DISCUSSION

In general, the plant fibers exhibited a high affinity for oil and water. The oil absorption of the fiber samples ranged from 0.32 g/g to 9.0 g/g, with an average oil absorption capacity across all fibers, treatments, and mediums of 3.01 ± 1.56 g/g.

Absorption characteristics of different fibers under different mediums

The average oil AC of corn, soybean, and cattail were similar (Fig. 1). As shown in Table 3, oak fibers exhibited the lowest oil absorption capacity. The average oil AC of corn, soybean, and cattail were 3.73 g/g (373%), 3.40 g/g (340%), and 3.43 g/g (343%), respectively. Oak and CBS fibers showed considerably lower oil absorption capacity of 1.68 g/g (168%) and 2.77 g/g (277%), respectively.

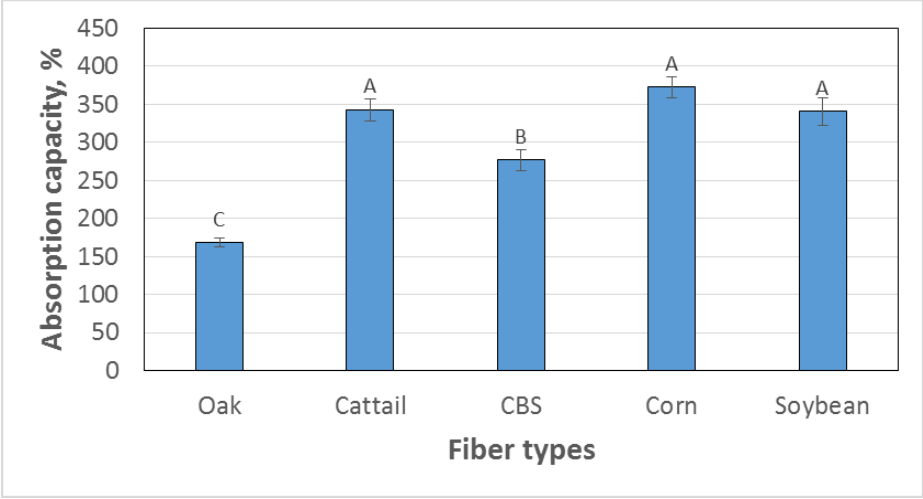

Fig. 1. Average absorption capacity (AC) of five types of plant fibers (n =100). Error bars show the standard error of the mean. Means with different letters are significantly different ($P < 0.05$).

All five types of fibers exhibited different bulk densities for the loose fiber structure, with oak showing the highest bulk density. Usually, a lower fiber density means the fiber has a higher volume per unit mass, indicating higher surface area for reaction with the absorption medium (Toyoda and Inagaki 2000; Eichhorn and Sampson 2010). The media exhibited different average absorption capacity by the plant fibers (Fig. 2). The oil mediums resulted in lower absorption capacity than the deionized (DO) water medium. The lower density and viscosity of water compared to the oil could explain the slightly lower absorption of the oil media. As shown in Table 3, the mixture of oil and water (80% DO water and 20% crude) demonstrated a similar absorption capacity for crude oil and motor oil.

Table 3. Average Absorption Capacity of Plant Fibers According to Fiber Type, Absorption Medium, and Fiber Treatments

Treatment	Levels	Absorption capacity (%) Mean ± Std. dev.
Fiber type	Oak	168.3^{C} ± 64.1
	Cattail	343.0^{A} ± 147.4
	CBS	276.8^{B} ± 134.0
	Corn	372.6^{A} ± 140.5
	Soybean	340.6^{A} ± 181.5
Fiber treatment	Control	234.1^{C} ± 143.4
	Heat125	334.6^{A} ± 105.8
	Heat80	287.8^{B} ± 90.8
	Mer25	359.0^{A} ± 136.9
	Mer300	372.6^{A} ± 222.9
Absorption medium	Motor Oil	288.4^{A} ± 124.1
	Crude Oil	270.2^{A} ± 118.0
	DO Water	385.7^{B} ± 184.9
	DO-Crude	258.9^{A} ± 157.5

Values are shown as the mean ± standard deviation
Heat80: hot water treatment at 80 °C for 24 h; Heat125: hot water treatment at 125 °C for 24 h); Merc25: mercerization at 25 °C for 24 h; Merc300: and mercerization at 300 °C for 1h
^{ABC} Means with different letter are significantly different ($P < 0.05$)

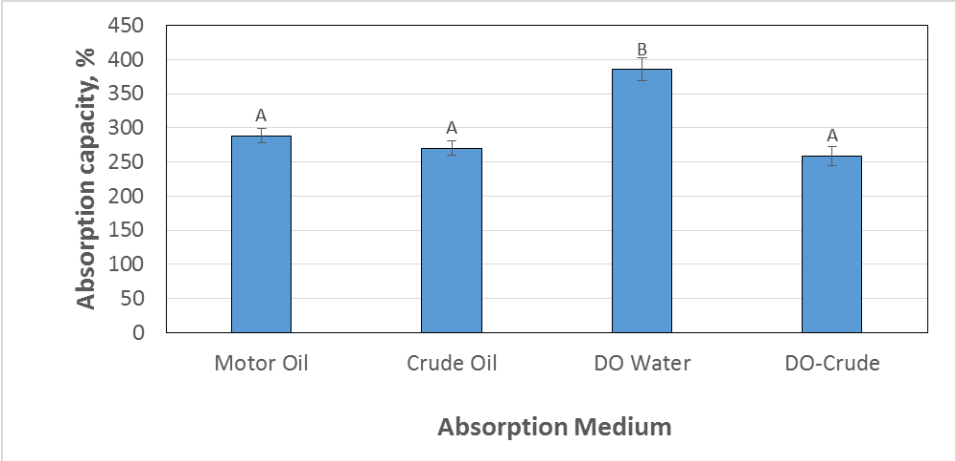

Fig. 2. The average absorption capacity of fibers grouped according to the absorption medium (n =100). The error bars show the standard error of the mean. Means with different letters are significantly different ($P < 0.05$).

The AC of fibers was compared according to the absorption medium. Because of the different physical and chemical characteristics of the fibers and media, it was expected that the different fibers would show different affinities towards each medium. All of the fibers (excluding oak) exhibited distinguishable differences in ACs for different media (Fig. 3). Corn, soybean, and cattail fibers exhibited the highest ACs; however the AC for DO water ranged from 4.4 to 4.7 g/g. The second largest absorption was exhibited by corn/motor oil and cattail/motor oil (3.83 g/g and 3.5 g/g, respectively), corn/crude oil (3.3 g/g), and soybean/crude oil/DO (3.3 g/g). Oak and CBS showed the lowest AC with all of the media. The combination of the bulk density of the fiber and density of the medium were regarded as responsible for the differences observed in the ACs.

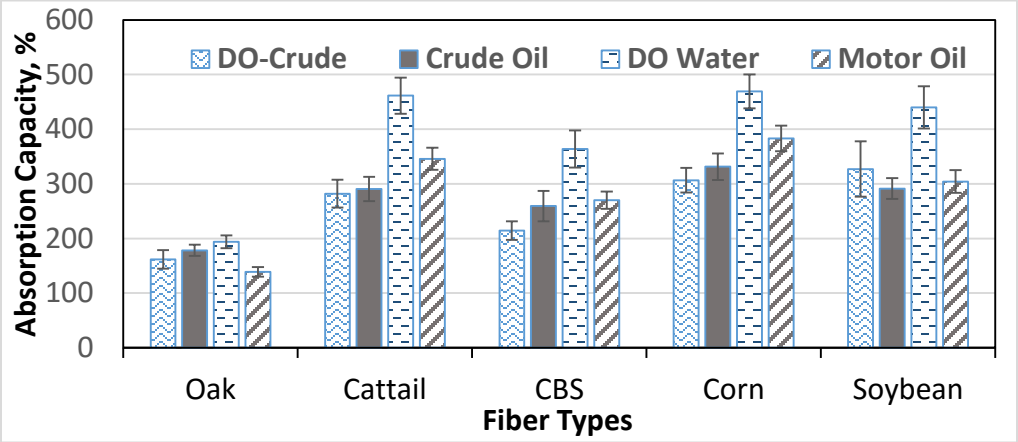

Fig. 3. The absorption characteristics of plant fibers according to the absorption medium. The error bars indicate the standard error of the mean.

Effect of fiber treatment on its absorption characteristics

Both heating and mercerization enhanced the absorption characteristics of the fibers (Fig. 4). However, the two mercerization treatments and the hot water treatment at 125 °C were the more effective at increasing the AC.

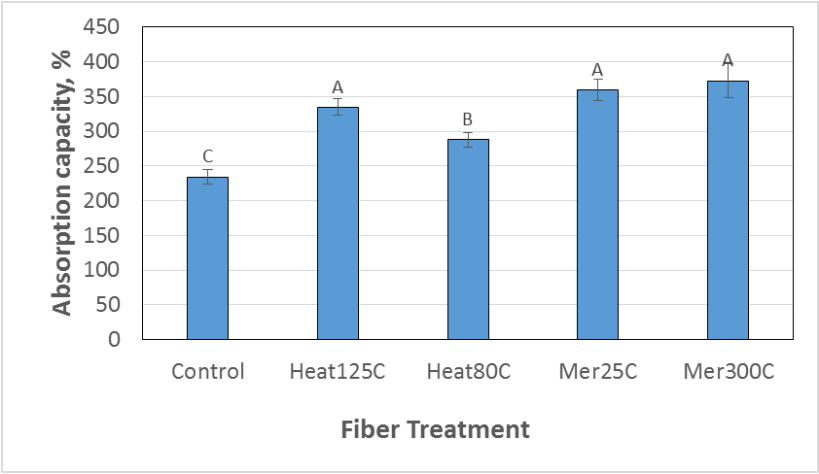

Fig. 4. Effect of fiber treatments on the absorption capacity of plant fibers averaged over different absorption media and fiber types (n =100). Note: treatment conditions: Control (no treatment); hot water treatment at 80 °C for 24 h (Heat80); hot water treatment at 125 °C for 24 h (Heat125); mercerization at 25 °C for 24 h (Merc25); and mercerization at 300 °C for 1h (Merc300). Means with different letters are significantly different ($P < 0.05$)

The hot water treatment process generally removes volatile compounds and the waxy coating on plant fibers, making the cellulosic fibers more accessible to the medium (Bajwa *et al.* 2015). Perhaps the hot water treatment at 80 °C was not hot enough to effectively remove the waxy coating from the fiber and limited its exposure to the media.

There were varying responses to the different treatment methods per fiber type. For example, mercerization at 300 °C more than doubled the ACs of soybean and CBS fibers, while this increase was not as pronounced in the other three fiber types (Fig. 5). Mercerization at room temperature and hot water treatment at 125 °C exhibited the highest ACs in corn and cattail fibers, resulting in a similar value for both fibers. Mercerization at room temperature was also very effective for soybean fibers. Even after fiber treatment, the oil AC of oak fiber was less than half that of the best performance of the other four fiber types. The chemical composition of the fibers and the structural changes due to treatments may have contributed to their difference in performance per treatment type. For example, CBS and soybean stalk are more fibrous materials than cattail and corn.

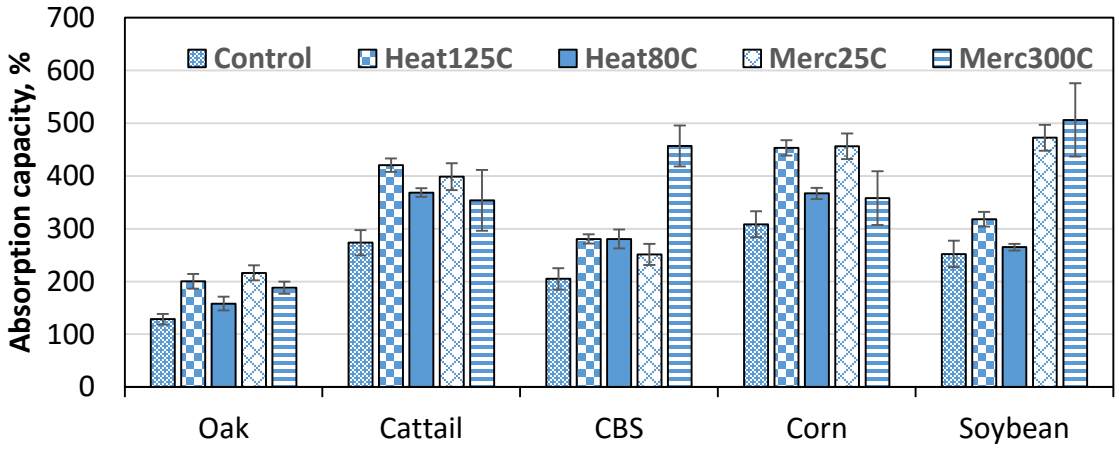

Fig. 5. The absorption characteristics of the different plant fibers according to the treatment type. The error bars indicate the standard error of the mean.

Interaction of fiber type, fiber treatment, and medium

An analysis of the interaction between fiber type, fiber treatment, and medium resulted in the most effective relationship between fiber type and fiber treatment suitable for a specific medium. Mercerization at 300 °C resulted in a distinguishable increase in the DO water absorption over 6 g/g for all fiber types (excluding oak). The largest AC of over 8 g/g was exhibited by soybean fiber mercerized at 300 °C for 1 h when exposed to crude oil/DO (Fig. 6).

For crude oil absorption, the hot water treatment of fibers at 125 °C was the most effective across all plant fibers (excluding CBS). The CBS fibers showed the highest crude oil absorption when mercerized at 300 °C for 48 h. For motor oil absorption, there was an interaction between plant fiber type and fiber treatment type. For example, soybean and corn fibers showed the best motor oil absorption when mercerized at room temperature for 48 h. Overall the corn and soybean fibers exhibited the best absorption capacity with both mercerization treatments as compared to oak. For all fibers hot water treatments at 125 °C was more effective than 80 °C. Mercerization at 300 °C for 1 h increased the AC of the soybean residue in DO-crude media up to 8 g/g (800%).

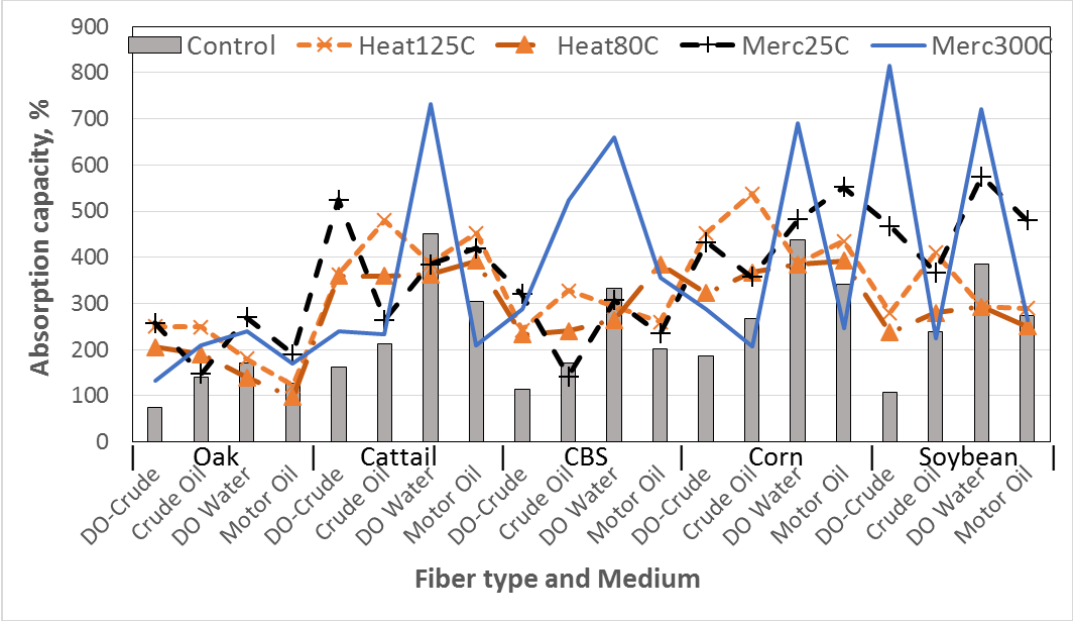

Fig. 6. Absorption characteristics of different plant fibers, fiber treatments, and absorption media

All plant fibers exhibited higher absorption capacities than oak fiber. The low AC of oak fiber can be attributed to its high bulk density compared with the other four plant fibers (Table 2). Since per unit weight of a fiber with a higher bulk density will have less volume, the surface area for reaction with the absorption media will be lower. This finding is consistent with previously reports on the relationship between bulk density and water absorption of various plant fibers, including oak and CBS (Bajwa *et al.* 2011). Overall, the affinity of the natural fibers for water was stronger than for oils (Fig. 2). The low viscosity of water, combined with the ability of water molecules to bind with the free OH groups in the cellulosic fiber, explains this selective affinity.

A comparison of fiber treatments showed that both mercerization at 25 °C and the hot water treatment at 125 °C were equally effective in enhancing the absorption characteristics of the fibers (Figs. 4 and 6). Mercerization increased the surface roughness of fibers, resulting in an increased surface area that led to improved absorption characteristics for all fibers in majority of the media. This was especially apparent for the mercerization at 300 °C. The effect of alkali on the cellulose component of fiber causes swelling, therefore, the natural crystalline structure of the cellulose I relaxes (Weyenberg *et al*. 2006). The type of alkali and its concentration influences the degree of swelling and the degree of lattice transformation into cellulose II. Sodium oxide is commonly used because Na^+ widens the smallest pores between the lattice planes for effective penetration. After removing the excess NaOH, a new Na-cellulose I lattice is formed. Meanwhile, the –OH groups of the cellulose are converted into ONa– groups. Rinsing with water removes the linked Na ions and converts the cellulose into a new crystalline structure, cellulose II, containing a lattice that is more stable than cellulose I (Weyenberg *et al.* 2006).

Although mercerization at 300 °C significantly increased the affinity of all fibers for DO water, other fiber treatments did not have the same profound effect on water absorption. For example, other fiber treatments showed a larger impact on the absorption of the oil medium than that of the water medium. The strong interaction between fiber type,

fiber treatment, and absorption medium is based on the different biochemistry, physical properties, and fiber structures/arrangements.

The results of this research demonstrate the potential of agricultural residues and cattails in oil spill abatement. Simple hot water and mild alkali treatments can alter the surface characteristics of plant fibers and improve absorption capacity. The findings can encourage the use of natural fiber as an alternative to polymeric materials.

CONCLUSIONS

1. The different plant fibers showed varying affinity for different media. Fibers from less fibrous materials showed higher absorption capacities than fibers from woody materials.
2. The four fiber treatments evaluated in this study increased the AC of the plant fibers.
3. The lower bulk density of materials tended to improve the absorption capacity.
4. Mercerization at 25 °C and hot water treatment at 125 °C were equally effective in increasing the absorptive capacity of the materials. Overall, mercerization at 300 °C resulted in a greater affinity for water than oil; however, all of the other treatments/fibers showed a similarity in increasing affinity for oil.

ACKNOWLEDGMENTS

This project was partially supported by the Office of Equity, Diversity, and Global Outreach, and by the North Dakota Agricultural Experiment Station. The authors thank Dr. Greg Holt, USDA-ARS, Lubbock, for providing the cotton burr and stem, and Dr. Guojie Wang, NDSU, for providing cattail fibers.

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Article submitted: March 14, 2016; Peer review completed: May 9, 2016; Revised version received: May 19, 2016; Accepted: March 24, 2016; Published: June 16, 2016.
DOI: 10.15376/biores.11.3.6452-6463