Oil absorption and desorption by polypropylene fibers

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ABSTRACT: Oil spill is a threat to the ecosystem, and there is a need for the development of highly efficient oil sorbents for environmental remediation. In this study, four different types of polypropylene fibers were evaluated for their oil absorbency and desorption characteristics. These fibers varied in their fineness and structural characteristics, i.e., hollow or solid. A modified ASTM methodology was used in the study to better represent the oil sorption capacity of a sorbent. To the best of our knowledge, this is the first study to present the effect of fiber fineness and structure on the oil desorption behavior of polypropylene fibers. Results showed that finer fibers had higher oil sorption capacity (g/g) than the coarser fibers. There was no statistical difference in the rate of desorption among the solid fibers; however, the hollow fiber had a statistically higher rate of desorption than the solid fibers.

Application: This study will help with the development of efficient sorbents, which will have high oil sorption capacity and ease of transportation.

Economic growth and an increase in the world's population have caused an increase in the consumption of oil [1]. The increase in the consumption of oil has resulted in the increase in oil spill volumes and instances, with an estimated 224,000 tons of oil being released into the marine environment across the globe from 2000 to 2011 [2]. The 2010 Deepwater Horizon oil spill, for example, was responsible for the release of 4.9 million barrels of oil into the ecosystem, causing an influx of hydrocarbons into the Gulf of Mexico's ecosystem [3,4].

The contamination of water due to oil is caused during production, refining, transport, and general use. Oil spills have proven to take a toll on the ecosystem, and the impact can be understood by the fact that one liter of Benzene can effectively make millions of gallons of water unfit for human consumption [5]. Studies, such as the one conducted by Sabir, point to the transfer of oil compounds along the food chain via oil-contaminated marine food like fish and seaweed [2]. Laboratory testing programs have also shown the effects of oil on soil, including reduction in soil permeability, maximum dry density, optimum water content, and strength due to oil exposure [6]. Hence, there is a need to control the damage due to oil spills in an efficient and economically feasible way.

Existing technologies for the cleanup of oil include in-situ burning, use of chemical dispersants, sorbents, and physical methods [7,8]. Among these, the sorption processes and the use of sorbents provide the lowest economic cost, simple operational procedures, and high oil removal efficiency for the oil spill cleanups [9,10]. There are three classes of sorbents available: synthetic, natural, and inorganic mineral sorbents [11]. Oil sorbents have seen an increasing market share in the recent past, and the consumption of oil sorbents has also been predicted to expand, due to sales growth in allied industries such as automotive and manufacturing [1]. Consequently, there has been a great interest in recent times to develop a cost-effective and efficient sorbent for the absorption of oil in water due to ecological disasters and strict environmental policies worldwide [2]. An ideal sorbent should be economically feasible, readily available, oleophilic and hydrophobic in nature, and have high sorption capacity [8,12]. There has been wide research about the use of both natural and synthetic fibers for potential use in sorbents [2]. Researchers have extensively investigated natural fibers as oil sorbents and have successfully produced novel solutions [8].

The development of natural fiber-based sorbents, as well as introducing functionality onto the natural fibers, has been a great source of academic interest [9,13-16]. More recently, there has been an interest in the use of cotton as an oil sorbent [8,17-19]. A study by Vinit et al. found that low micronaire cotton showed higher sorption values due to higher surface area and increased fineness [17]. Micronaire of cotton fibers is regarded as an indicator of fiber fineness (linear density) and maturity (degree of cell wall development). A follow-up study by Vinit et al. presented a cotton-based novel sorbent with sorption capacity of 50.27 g/g, using low micronaire raw cotton [8]. However, in a recent study, the same group has comprehensively compared the sorption capacity of greige cotton hydroentangled and needle-punched nonwovens versus loose greige cotton fibers. The loose cotton fibers were found to have more oil sorption capacity as compared to the cotton nonwoven substrates [19]. Cortese et al. [20] proposed diamond like carbon (DLC) coated cotton textiles for the oil-water separation application, by the process of plasma-enhanced chemical vapor deposition of carbon, and reported superhydrophobicity, while Ge et al. [21] developed a fabrication method in their study for a hydrophobic fiber for the oil absorption—a graphene coated cotton. Lee et al. [22] fabricated a hydrophobic silicon dioxide (SiO_2) coated cotton fiber for potential application in oil spill cleanup. These methods do provide an insight into the oil absorption research on natural fibers; however, work still needs to be done to make multistep production and manufacturing of these sorbents scalable as well as commercially and economically viable.

Agricultural byproducts and wastes as potential oil sorbents have been worked on lately. Bazargan et al. [23] discussed an oil absorbent made after the treatment of rice husks, an agricultural waste. Suni et al. [24] have compared the oil sorption properties of cotton grass against commercially available synthetic sorbent for absorbing petrol, synthetic oil, diesel oil, mineral oil, and heavy fuel oil. Cotton grass is a major byproduct obtained when peat is excavated from wetlands. The oil sorption capacity (g/g) of cotton grass was observed to be the highest of all the previously-mentioned sorbents.

Synthetic fibers, on the other hand, provide a lower cost, are commercially and economically feasible, and can be produced regardless of the demography—unlike the natural fibers. Researchers have worked extensively on developing a sorbent consisting of polypropylene (PP) as at least one of the constituents. Zahid et al. [25] have investigated the removal of oil from the surface of seawater by the PP matrices. Zhao et al. [26] have used PP as a constituent in their work, presenting a thorough study of three nonwoven materials: PP; PP/poly(butylmethacrylate-co-hydroxyethylmethacrylate) (PBMA-co-HEMA; 6 wt%); and PP/poly(butylmethacrylate-co-hydroxyethylmethacrylate) (PBMA-co-HEMA; 20 wt%). The addition of PBMA-co-HEMA was reported to increase the oil sorption capacity, consequently reducing the strength of the material. The authors pointed out the dependence of oil viscosity on the oil absorption capacity; higher viscosity of crude oil as compared to diesel and toluene resulted in higher absorption of crude oil. Li et al. [27] discussed the fabrication of melt electrospinning based PP fibers, reporting an oil sorption capacity of 129 g/g—significantly higher than that of a PP nonwoven, which is attributed to the finer fiber diameter and porous structure. Lin et al. [28] proposed a composite material consisting of waste tire powder and recycled PP fiber, observing the cumulative oil sorption capacity after repeated use to be higher than that of PP fiber. Zhou et al. [29] introduced a new type of PP sorbent material obtained by UV-radiation induced graft polymerization of octadecyl acrylate monomer on the PP fiber and PP nonwoven matrices. The grafted PP fiber and PP nonwoven both were observed to have a significantly better oil retention capacity, even after 10 cycles. Wei et al. have presented a study with five different types of commercial nonwoven PP sorbents using different (stitchbonded, needle-punched, melt-blown) manufacturing techniques, producing varying properties (processing, GSM, porosity, and fiber diameter) to determine and compare the retention ratio for oil sorption [30].

The wide array of research about PP does point out the importance of the PP fiber as a cornerstone in oil spill cleanup, an ideal sorbent [30], and as a widely used sorbent material for cleanup [8]. Batzargan et al. [23] argued that good oleophilicity and the ability of a sorbent to withhold the oil under gravitational force are desirable characteristics for a good sorbent. Bazargan et al. [31] have highlighted the importance of retention behavior of oil sorbents in the oil spill application, validating their model for sixty sorbent-sorbate systems. One of the factors that determine the suitability of a sorbent for an oil spill application is the retention of oil after the sorption, as long duration of handling and transportation is required.

The ever-increasing demand and potential expansion of the oil sorbent market calls for an in-depth study of the mechanism of the oil sorption by individual PP fibers. More importantly, the need for a cost-effective sorbent to enhance human health and protect the environment necessitates such a study. Accordingly, this study was conducted to better understand the oil sorption capacity, desorption behavior, and the rate of desorption of the PP fibers for oil spill cleanup application. We have used four different types of PP fibers with varying fiber fineness, as it influences the available surface for oil sorption. The dependence of the oil sorption and desorption behavior of PP fiber on the structure (hollow or solid) has also been studied. The sorption capacity of PP fibers was observed to decrease with increase in the fiber fineness, which agrees with the findings of other researchers [12,27]. The desorption performance and the rate of desorption values have been presented and statistically analyzed to better understand the retention capacity of the PP fibers.

MATERIALS AND METHODS

Polypropylene samples

Four different types of PP fibers having fiber fineness 1.5 denier (den), 2.2 den, 4 den, and 7 den were used in this study. The PP fibers were provided by FiberVisions (Covington, GA, USA). **Table I** provides the information about the fibers used in the study. Fibers with 1.5 den, 2.2 den and 4 den were solid in cross section, and the fibers with 7 den were hollow.

Fiber Fineness, den	Cross-section	Staple Length, mm	Crimp, crimps/mm	Surface Finish, TiO_2 Level*
1.5	Round	38.1	0.89	High level
2.2	Round	38.1	0.98	Low level
4	Round	47.62	0.83	Low level
7	Hollow	47.62	0.55	None

* The amount of titanium dioxide (TiO_2) influences (increases) the surface roughness and opacity of the fiber along with the dullness of the fiber surface. The TiO_2 is mixed in the PP resin during the extrusion process.

I. Polypropylene (PP) fiber properties

Type	Viscosity, cP	Density, g/cm³	Surface Tension, mN/m
Non-detergent motor (NDM) oil	139.8	0.925	31.44

II. Oil property [19].

Oil used

A non-detergent motor oil was used as a sorbate for the study. The properties of the non-detergent motor (NDM) oil are presented in **Table II**. According to the ASTM F726-12 "Standard test method for sorbent performance of adsorbents," the density of NDM oil used in the study satisfies the density requirement (0.860 to 0.970 g/cm³) for the medium oil.

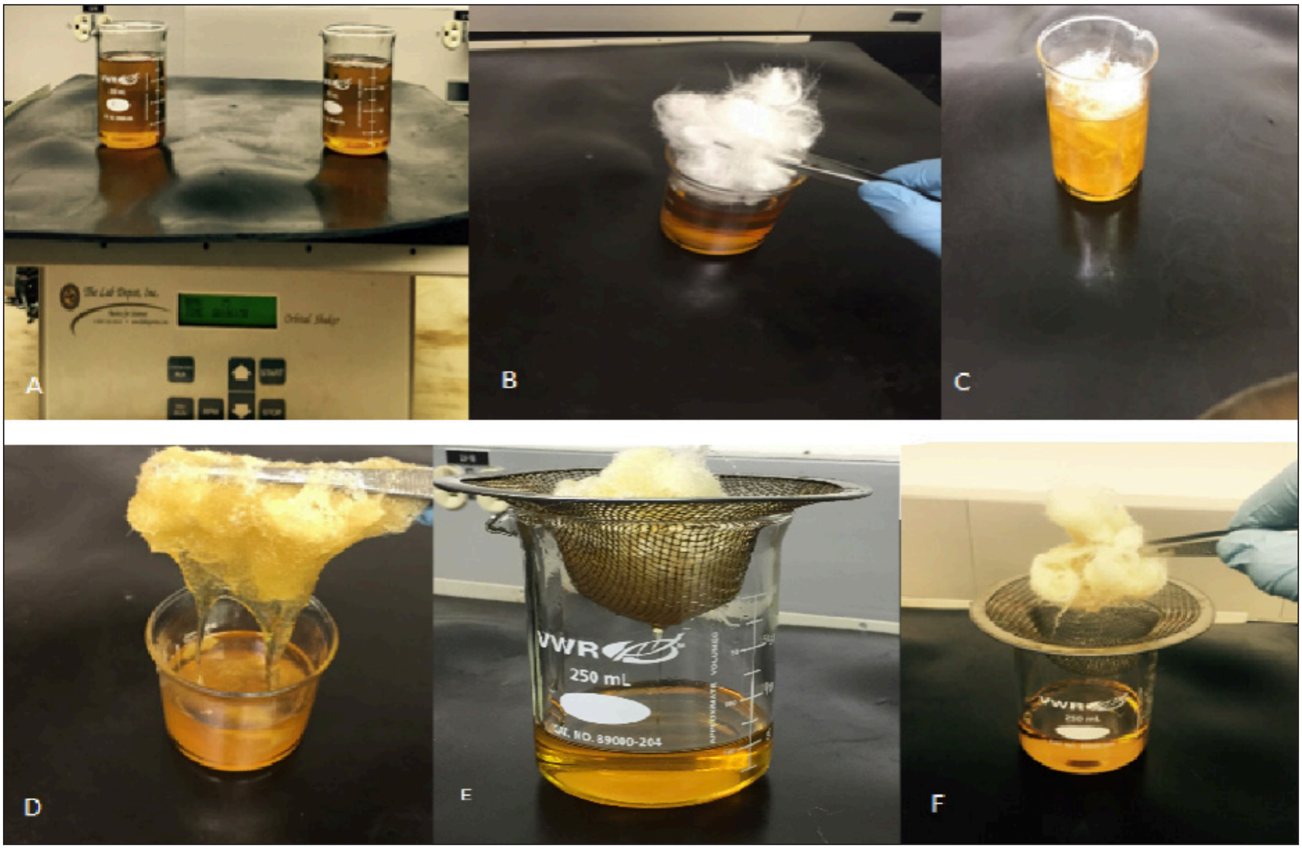
EXPERIMENTAL SETUP

The tests were carried out according to a modified ASTM F726-12 Standard (**Figs. 1A - 1F**). To perform the tests, 250 ml beakers containing 200 ml of NDM oil were used. The PP fiber samples, 5 grams in weight, were dropped in flasks containing oil. The beaker was then installed on the orbital shaker table, which was used to create a dynamic environment set at 75 rpm. The test was run for 15 min ± 3 s. The oil soaked samples were removed from the beaker and subsequently placed in a mesh for drainage. The gravimetric drainage process was used, and the weight of the sorbent was measured every 10 min ± 3 s, until reaching a cumulative time of 60 min ± 3 s. Researchers have observed a high desorption rate for the initial 10 min [8,32]. The values for the sorption capacities (**Fig. 2**) would be much higher if the measurements were taken after draining for 30 s rather than draining for 10 min. However, during an oil spill cleanup, the sorbents are spread onto the oil spill and then collected once the oil is absorbed. Accordingly, citing the high initial desorption, along with the delay in removal of the sorbents from the oil spill, we adopted a method for this study that measures the oil sorption after 10 min of gravimetric dripping, rather than the 30 s mentioned in ASTM F726-12, which to our understanding would reflect the oil sorption values mimicking real world application [8,17,19].

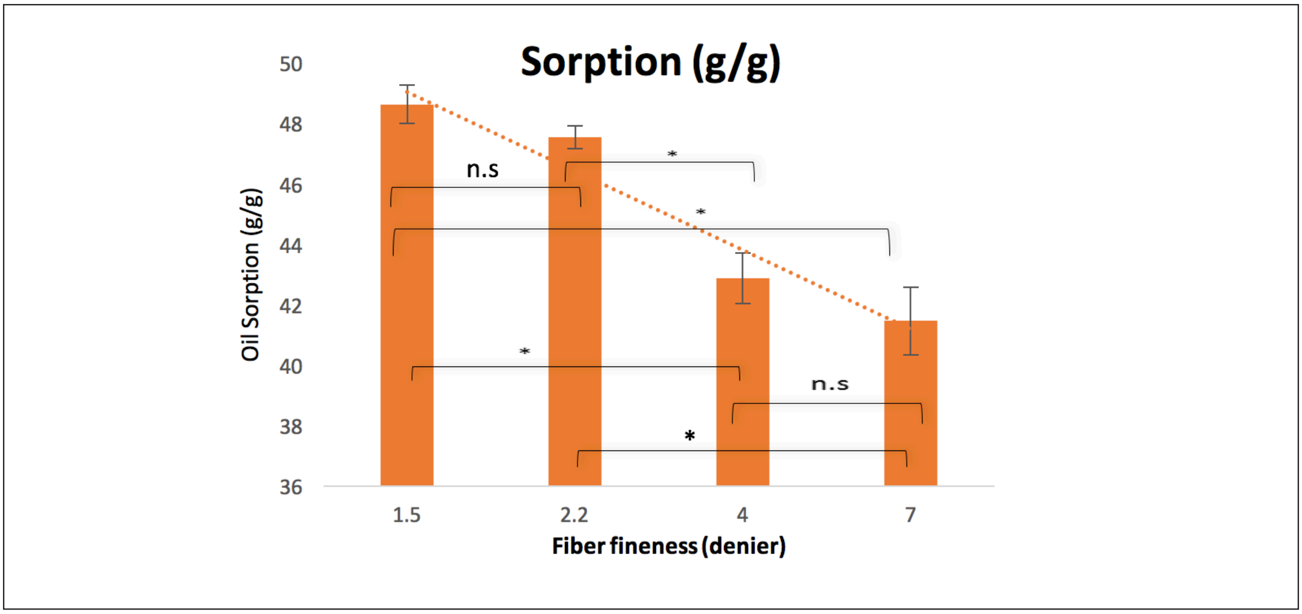
The oil sorption capacity was calculated as shown in Eq. 1:

$$\text{Oil sorption capacity} = (S_{st} - S_o)/S_o = S_s/S_o \quad (1)$$

where S_o = initial dry sorbent weight (g), S_{st} = weight of the oil sorbed sample (g) after 10 min of gravimetric dripping, and $S_s = (S_{st} - S_o)$ = net oil sorbed (g).



1. Modified ASTM F726-12 test standard for oil sorption study. A: Experimental setup for oil sorption study. B: Polypropylene (PP) fiber being immersed in the oil beaker. C: Oil sorption process: fiber getting fully immersed. D: Oil soaked PP fiber during the sorption process. E: Gravimetric oil dripping setup. F: PP fiber after oil draining for 10 min.



2. Sorption capacity (g/g). Statistical analysis was performed using two-sample t-tests for a sample size of 10 for all the possible pairs. Cross bars represent standard error of means, n=10; the region denoted by an asterisk (*) represents significance (P < 0.05), and "n.s" represents non-significance (P > 0.05).

RESULTS AND DISCUSSION

Oil sorption and desorption

The rationale for conducting both oil sorption and desorption study is provided here. There have been a wide range of studies about numerous materials for oil spill cleanup applications. The importance of sorption capacity (g/g) and the oil retention property of the sorbent have been stressed by researchers [16,17,33]. To the best of our knowledge, there has been no prior study on the desorption characteristics of polypropylene fibers and the effect of fiber fineness on the desorption of oil. The desorption study is critical for the design of oil sorbents, as once the oil is taken up, the expectation is that the sorbent does not lose the oil quickly so that it can be transported to a convenient site without causing any additional environmental problems. However, for recovery, after transporting the oil soaked sorbent to a safe environment, the oil must be recovered. The desorption behavior of the PP sorbent was evaluated using the gravimetric dripping process.

As reported in **Table III**, average oil sorption capacity (g/g) for PP fiber with fiber fineness (1.5 den, 2.2 den, 4 den, and 7 den) was found to be 48.65, 47.55, 42.89, and 41.48, respectively. As seen in **Fig. 2**, 1.5 den fiber had statistically higher oil sorption capacity than 4 den and 7 den fibers; however, there was a non-significant difference with 2.2 den fiber. The 2.2 den fiber had significantly higher oil sorption capacity than 4 den and 7 den fibers. There was no statistical difference between the oil sorption values of fibers with 4 den and 7 den.

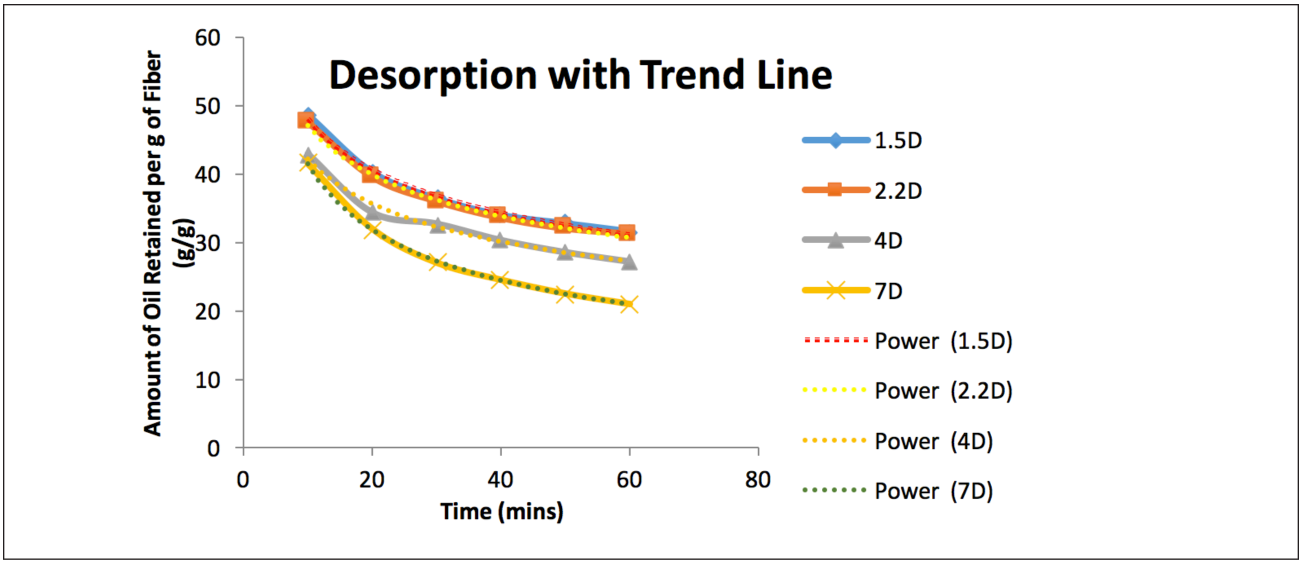
Fiber Fineness, den	Sorption, g/g
1.5	48.65 (0.63)
2.2	47.55 (0.38)
4	42.89 (0.83)
7	41.48 (1.12)

III. Mean sorption values (sample size =10) after gravimetric drainage for time of 10 min with standard error values in parentheses.

The fiber fineness, which represents the thickness of the fiber, was observed to have a significant effect on the oil sorption capacity of PP fibers. The oil sorption by PP fibers can be attributed to adsorption on the fiber surface and absorption by fibers [17]. It was seen, as a general trend from Fig. 2, that as the fiber becomes coarser, its sorption capacity decreased, as is the case with 7 den fiber compared with 1.5 den fiber. This means coarser fibers showed less oil sorption capacity than their finer counterparts. The high oil sorption, due to low fineness, corroborates with the results reported by other researchers [27,34]. Low fineness corresponds to higher specific surface area, which contributes to high oil sorption (g/g) [12,17]. Higher specific surface area would provide more surface for the sorbate to adsorb onto the surface, which is the initial mechanism for the oil sorption process.

Oil retention behavior

To analyze the oil retention behavior of the sorbent, the sorption test was performed in oil medium. As can be seen from the experimental curves (**Fig. 3**), all the fibers followed a similar oil retention behavior. The oil retention ratio (g/g) of the PP sorbent, presented in **Table IV**, underwent three stages. As soon as the sorbent was removed from the test, a high dripping rate was observed, accounting for the loosely



3. Amount of oil retained per gram of fiber (g/g) for PP fiber with fiber fineness (1.5 den, 2.2 den, 4 den, and 7 den) versus time.

adsorbed oil. In the second stage (from 10 to 30 min), a lower dripping rate was observed in all the evaluated fibers. The last stage (from 30 min until the end of the test) signified a stable and low dripping rate region. To further analyze the oil retention behavior, the desorption curve was plotted empirically. The desorption curve fit well with the power equation, as shown in **Table V**, with strong R^2 values. Higher R^2 values (all above 98%) provide evidence for the good fit of the experimental data to the derived power equation.

Rate of desorption

The rate of desorption for PP fibers is presented in **Table VI** with different fiber fineness (1.5 den, 2.2 den, 4 den, and 7 den) and were found to be 0.22, 0.24, 0.27, and 0.38, respectively. Statistical analysis was performed using a two-sample t-test for a sample size of 10 for the possible pairs. Cross bars in **Fig. 4** represent standard error of means, n=10; the region denoted by an asterisk (*) represents $P < 0.05$ and “n.s” represents non-significance.

Fiber Fineness, den	10 min	20 min	30 min	40 min	50 min	60 min
1.5	48.65 (0.62)	40.25 (0.47)	36.4 (0.60)	33.97 (0.57)	32.75 (0.51)	31.57 (0.52)
2.2	47.55 (0.38)	39.54 (0.37)	35.98 (0.31)	33.56 (0.36)	32.02 (0.4)	31.23 (0.26)
4	42.89 (0.83)	34.39 (0.59)	32.74 (0.77)	30.35 (0.63)	28.59 (0.57)	27.23 (0.48)
7	41.48 (1.12)	32.00 (1.09)	27.06 (1.18)	24.55 (1.15)	22.56 (1.03)	21.02 (0.96)

IV. Mean sorption values (g/g) with standard error in parenthesis (sample size =10) for different times under gravimetric dripping.

Fiber Fineness, den	Equation for Desorption Y: desorption, g/g X: time, min	R^2 Value
1.5	$Y = 83.95X^{-0.242}$	0.994
2.2	$Y = 81.355X^{-0.238}$	0.995
4	$Y = 74.07X^{-0.244}$	0.986
7	$Y = 99.332X^{-0.38}$	0.999

V. Desorption equations derived with their R^2 values.

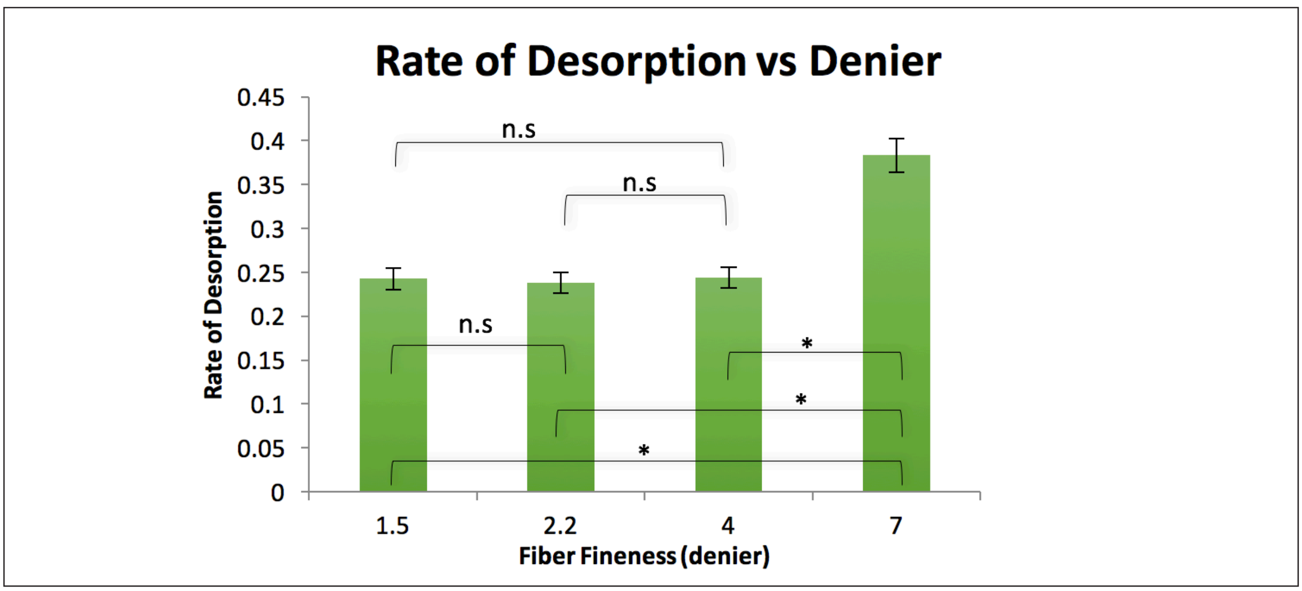
Fiber Fineness, den	Rate of Desorption (S.E.)
1.5	0.243 (0.008)
2.2	0.238 (0.007)
4	0.244 (0.007)
7	0.384 (0.021)

VI. Fineness versus the mean rate of desorption values (sample size =10) with standard error (S.E.) in parentheses.

The rate of desorption was calculated using the power equation for each of the samples used in this study (Fig. 4). The desorption rate, as discussed earlier, would provide a better understanding of the oil retention behavior of the fibers. Low rate of desorption is a desired attribute, as it signifies the tendency of the fibers to hold oil. Structure (i.e., solid or hollow) of the fibers was observed to play a role in the desorption rate. The fiber types with 1.5 den, 2.2 den, and 4 den, which are all solid in cross-section, have a statistically significant difference in rate of desorption with 7 den fiber. This can be explained by the solid structure of 1.5 den, 2.2 den, and 4 den fibers and hollow structure of 7 den fiber. To the best of our understanding, the absence of core in the hollow fibers is responsible for a statistically higher rate of desorption as compared to the solid fibers. As has been discussed by Vinit et al. [17], oil is diffused into the fiber through the micropores on the fiber, although this effect will be relatively less. However, in the case of hollow fibers, in the absence of the core, oil retention by individual fibers will be relatively less compared to regular fibers. In addition, the non-statistical significance of the difference in rate of desorption for fibers (1.5 den, 2.2 den, and 4 den) with solid structure supports this explanation, as fiber fineness does not have any significant effect on the rate of desorption. The higher rate of desorption in hollow fibers and non-dependence on fiber fineness are interesting takeaways from this study, and it would help in the selection of an appropriate fiber with maximum oil sorption and desired desorption.

CONCLUSIONS

A detailed analysis of PP fibers as oil sorbents, including the testing of fibers with fiber fineness 1.5 den, 2.2 den, 4 den, and 7 den, has been presented in this paper. In addition, the dependency of the fiber structure (i.e., hollow or solid) on the oil sorption was also investigated. The oil sorption capacity was observed to decrease (48 g/g to 42 g/g) with an increase in the fiber fineness (from 1.5 den to 7 den). The desorption, which is an important attribute for the oil sorbents, showed a similar trend for all the fiber types—high desorption followed by a decrease in the desorption. Strong statistical evidence suggested power equation as the best fit for time dependent desorption under gravimetric dripping. The rate of desorption was statistically insignificant within the solid fiber types (1.5 den, 2.2 den, and 4 den). However, the rate of desorption was significantly higher for the hollow fiber in comparison with other solid fiber types. In conclusion, the finer the fiber is, the higher is the oil sorption. However, in the case of desorption, the rate of desorption was shown to be influenced by the structure of the fiber rather than the fineness, which needs further investigation. Such a study would involve fibers with different cross-sections, different structure (i.e., solid or hollow), and varying fiber fineness. We hope that the results from this study would be valuable



4. Rate of desorption versus fiber fineness. Statistical analysis was performed using two-sample t-tests for a sample size of 10 for all the possible pairs. Cross bars represent standard error of means, n=10; the region denoted by an asterisk (*) represents significance (P < 0.05), and "n.s" represents non-significance (P > 0.05).

for designing a suitable oil sorbent with necessary oil sorption and desorption capabilities. **TJ**

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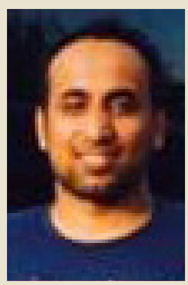
ABOUT THE AUTHORS

We studied this topic to find remedial solutions for environmental pollution and contamination using fibers and nonwovens. This topic furthers the research being done in our laboratory that focuses on understanding the use of a common fiber from the nonwovens industry for applications in oil spill remediation.The most difficult aspect of this study was to establish a reliable test protocol, as we had to use a small sample size, so it was important to focus on basic science that can lead to applications. Our most interesting finding was that fibers in loose form are the best candidates for use in oil spill remediation.

Toxic chemical and oil spills are common in many manufacturing industries, in addition to being a major issue in oil transport. So, our research highlights the use of fibers and nonwovens in saving the environment. The next step is to transfer these results so that practical products can be developed.

Yegya Raman



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