

Sujit Kumar Sinha¹, P. Kanagasabapathi¹, Subhankar Maity²

¹Dr. B. R. Ambedkar National Institute of Technology, Department of Textile Technology, Jalandhar 144011, Punjab, India

²Uttar Pradesh Textile Technology Institute, Department of Textile Technology, Kanpur, 208001, India

Performance of Natural Fibre Nonwoven for Oil Sorption from Sea Water

Zmogljivost vlaknovin iz naravnih vlaken za sorpcijo olj iz morske vode

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Abstract

This work deals with the study of the oil sorption behaviour of needlepunched nonwoven fabrics produced from natural fibres such as cotton, cotton flat waste, cotton/kapok blend, and nettle fibres. Polypropylene nonwoven fabric, which is used as a commercial oil sorbent, was also prepared using the same needling parameters for comparison purposes. The effect of the type of fibre, oil, and fabric parameters on oil sorption and retention capacities was investigated. All of the fabrics displayed higher oil sorption capacities for engine oil (high viscosity) than diesel oil (low viscosity). Among natural fibre nonwovens, cotton and cotton/kapok nonwovens displayed higher oil sorption capacities than that of polypropylene nonwovens, while nettle fibre nonwoven fabric displayed poor oil sorption capacity. An increase in kapok content in cotton/kapok nonwovens led to an increase in oil sorption behaviour. More than 95% of the diesel oils adsorbed by the nonwoven fabrics could be recovered by simple compression. Oil sorption capacity of the nonwovens were reduced significantly during repetitive cycles of use due to higher thickness loss. This study indicated that cotton and cotton/kapok nonwovens displayed better oil sorption behaviour than polypropylene, and may be used as an alternative natural oil sorbent material.

Keywords: biodegradable oil sorbent, recovery of sea-water oil, sustainable textile, oleophilicity, oil spill

Izvleček

V članku je predstavljena študija učinkovitosti iglanih vlaknovin za sorpcijo olj, izdelanih iz naravnih vlaken, tj. bombaža, bombažnih odpadkov iz predilnic, mešanice bombaž/kapok in iz vlaken koprive. Pri enakih pogojih je bila za primerjavo izdelana tudi polipropilenska vlaknovina, saj so polipropilenske vlaknovine komercialni oljni sorbenti. Preučevan je bil vpliv vrste vlaken in olj ter parametri vlaknovin na sorpcijo in sposobnost zadrževanja olja. Vse vlaknovine so absorbirale večje količine motornega (visoka viskoznost) kot dizelskega olja (nizka viskoznost). Med vlaknovinami iz naravnih vlaken so večjo sorpcijo olja dosegle bombažne vlaknovine in vlaknovine iz mešanice bombaž/kapok kot polipropilenska vlaknovina. Pri tem pa je vlaknovina iz vlaken koprive dosegla najnižjo sorpcijo olja. Povečanje vsebnosti kapoka v vlaknovinah iz mešanice bombaž/kapok je vplivalo na povečanje sorpcije olja. Več kot 95 % dizelskih olj, ki so jih absorbirale vlaknovine, je bilo ponovno pridobljeno s preprostim stiskanjem. Pri ponovni uporabi vlaknovin so se zaradi zmanjšanja debeline njihove sorpcijske zmogljivosti zelo poslabšale. Raziskava dokazuje, da imajo vlaknovine iz bombaža in mešanice bombaž/kapok boljše sposobnosti sorpcije olja kot vlaknovina iz polipropilena in bi se zato lahko uporabljale kot alternativni naravni sorbenti.

Ključne besede: biorazgradljivi oljni sorbenti, zbiranje olja iz morske vode, hitrost sorpcije olja, trajnostni tekstil, oleofilnost, razlitje olja

Corresponding author/Korespondenčni avtor:

Subhankar Maity

E-mail: maity.textile@gmail.com

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1 Introduction

Oil spills generally occur on the ocean's surface and also in nearby land areas due to tanker disasters, wars, operational failure, equipment failure, accidents and natural disasters during the production, transportation, storage and use of oil [1–3]. It is a serious problem that causes environmental and ecological imbalance, as well as financial loss. The immediate and effective decontamination and clean-up of spilled oils are necessary in order to protect the environment and human health [4]. Various methods are available for oil spill clean-up, such as mechanical recovery, dispersants, burning, etc. However, not a single system has been found to be completely effective. Oil spill clean-up through oil sorption using sorbents is one of the most efficient and economical methods [5–6].

Commercially, polypropylene is most widely used as oil sorbents due to its oleophilic and hydrophobic characteristics, but it is non-biodegradable [7–8]. This presents a great challenge in the disposal of the sorbent after usage [9]. The use of natural fibres such as milkweed, kapok, cotton, wool, flax, ramie, etc., as oil sorbents has been reported [10–16]. Cotton fibre was studied for oil sorption behaviour and reported to have higher oil sorption capacity than polypropylene fibre [17]. The crude oil sorption capacity of low-micronaire cotton is also significantly higher than that of high-micronaire cotton because it contains a higher number immature fibres [4, 18]. Milkweed and kapok fibres were reported to exhibit better oil sorption behaviour than the rest of the above-mentioned fibres. Higher surface waxes and non-collapsing lumens are believed to be the reason [9, 17]. Kapok/polypropylene blend needlepunched nonwovens were investigated as oil sorbents. It was reported that a 50/50 blend ratio of kapok and polypropylene demonstrated higher oil sorption [19]. Choi, Kwon, and Moreau investigated cotton/polypropylene blend needlepunched nonwovens as oil sorbents, and reported that an increase in cotton content increases oil sorption capacity [17]. Nettle fibre was also tested as another alternative material due to its hollow structure and the presence of surface waxes [20].

Loose fibres demonstrated a higher oil sorption capacity than structured fibrous assemblies due to a less effective or accessible fibre surface area [21]. The collection of fibres in loose form from a spill

area after use has been found to be a challenge. Hence, the nonwoven form is the best choice where the accessible fibre surface area is closer to loose fibres due to structural openness and the easy collection of nonwovens after use [9]. The oil sorbent characteristics of stitch-bonded, needlepunched and spunlaced nonwovens based on polypropylene were investigated. It was determined that porosity, pore size, and fibre fineness are important parameters for oil sorption [6]. However, the oil sorption behaviour of nonwovens does not depend on web forming technologies such as carding and air-laid techniques [9].

Based on literature review it is understood that as natural resources, immature cotton, kapok and nettle fibres may have great potential of oil sorption from seawater. Cotton flat waste has immature fibres in majority and may be a potential candidate for oil sorption. But it is not explored for this application. There is lack of information in literature on oil sorption capacity of needlepunched nonwovens made of cotton flat waste, cotton/kapok blend and nettle fibres.

Therefore, in this work, needlepunched nonwovens were produced from cotton, cotton flat waste, cotton/kapok blend of three different proportions, nettle and polypropylene fibres using the same needling parameters. Oil sorption behaviour, mechanical properties, and the re-usability of all these nonwoven specimens were tested and compared with polypropylene nonwoven to find a sustainable alternative of the same.

2 Experimental

2.1 Materials

The raw materials used for this work were cotton, cotton flat waste (collected from carding machine during spinning of the cotton fibre), kapok, nettle (*Girardinia diversifolia*) and polypropylene fibres. The African variety of cotton, its flat waste and virgin polypropylene fibres (3.33 dtex, 50 mm cut length) were collected from local industrial producers in Punjab, India. Properties of cotton, cotton flat waste and kapok were measured using a high volume instrument (HVI) and advanced fibre information system (AFIS) (Table 1). The nettle fibre (fineness 1.4 dtex and 50 mm cut length) was purchased from the Uttaranchal Bamboo and Fibre Development Board, India. The kapok was collected from

Table 1: Fibre properties measured by AFIS/HVI

Fibre type	Fibre properties (Measured by AFIS/HVI)					
	5% L(N) ^{a)} (mm)	SFC (N) ^{b)} (%)	SFC (W) ^{c)} (%)	Fineness (mtex)	Maturity (%)	IFC ^{d)} (%)
Cotton	33.6	20.8	7.1	165	91	6.5
Cotton flat waste	27.7	67.8	37.0	144	74	16.4
Kapok	19.7	67.1	45.8	125	78	12.6

^{a)} 5% AFIS fibre length, ^{b)} short fibre content by number, ^{c)} short fibre content by weight, ^{d)} immature fibre content

industrial producers in Coimbatore, India. Engine oil (high viscosity) and diesel (low viscosity) were used to conduct oil sorption testing. The specifications of the oils are given in Table 2.

Table 2: Specification of oils

Oil type	Viscosity at 40 °C (mm ² /s)	Density at 15 °C (kg/dm ³)
Engine oil	121	0.95
Diesel	2.5	0.82

2.2 Sample preparation

All needlepunched nonwoven samples were prepared using a DILO (Germany) needlepunching machine using a punch density of 50 punches/cm², a needle depth penetration of 8 mm, and a mass per unit area of 200 g/m². Parallel- and cross-laid nonwovens were prepared for 100% cotton fibre only. A cross lapper was used for the preparation of cross-laid nonwovens. The compositions of all prepared nonwoven samples are shown in Table 3.

Table 3: Composition of needlepunched nonwoven fabrics

No.	Sample code	Sample description
1	S1CP	Nonwoven made of 100% cotton, parallel-laid
2	S2CC	Nonwoven made of 100% cotton, cross-laid
3	S3FW	Nonwoven made of 100% cotton flat waste
4	S4C/K	Nonwoven made of 30% cotton and 70 % kapok fibre
5	S5C/K	Nonwoven made of 50% cotton and 50 % kapok fibre
6	S6C/K	Nonwoven made of 70% cotton and 30 % kapok fibre
7	S7PP	Nonwoven made of 100% polypropylene fibre
8	S8NF	Nonwoven made of 100% nettle fibre

2.3 Methods

2.3.1 Measurement of nonwoven properties

The mass per unit area of the nonwoven samples was determined according to the ASTM D6242-98 standard. The nonwoven fabric thickness was determined according to the ASTM D5729-97 standard at a pressure of 4.14 kPa. The bulk density (kg/m³) of nonwoven samples was calculated using equation 1.

$$\text{Bulk density} = \frac{W \times 10^3}{t} \quad (1),$$

where W is the mass per unit area of the sample (g/m²) and t is the thickness of the sample (m).

The porosity and pore size distribution of the nonwoven fabrics were measured using a capillary flow porometer (CFP-1100-AEHXL, PMI Inc.). The measurements were carried out in a dry-up/wet-up test mode using a Galwick solution (surface tension 15.9 mN/m) to saturate the samples after the dry test. The minimum, maximum, average pore diameters and pore size distribution of all samples were measured.

The tensile strength and breaking elongation in machine direction and in a cross direction of nonwoven fabrics was measured using a universal testing machine (Zwick) according to the ASTM D 5035-09 standard and the CRE principle, with a sample size of 20 cm × 10 cm, gauge length of 75 mm and testing speed of 300 mm/min.

2.3.2 Measurement of oil sorption capacity

The ASTM F716-82 (sorber performance of absorbents) and ASTM 726-81 (sorber performance of adsorbents) standards were followed for measurement of oil sorption capacity of the prepared nonwoven samples. The testing procedure of oil sorption capacity was classified in two ways: (a) oil sorption from oil in an artificial seawater bath; and (b) oil sorption from an oil bath. The artificial seawater bath was prepared according to the AATCC 106-8 standard.

a) Measurement of oil sorption from oil bath

To study the oil sorption capacity of oil sorbents without a water medium, a simple procedure was used. 60 g of sample oil was placed in a 1000 ml size glass beaker, and the dry nonwoven specimen was immersed in the oil for 10 minutes. As a result, the nonwoven specimen was soaked with the oil and the excess oil was allowed to drain by free hanging the soaked specimen vertically for 5 minutes. The specimen was then weighed. The oil sorption capacity of sorbents nonwoven was determined using equation 2.

$$\text{Oil sorption capacity} = \frac{W_{SO} - W_S}{W_S} = \frac{W_O}{W_S} \quad (2),$$

where W_S is the mass (g) of a dry and fresh nonwoven, W_{SO} is the mass (g) of the nonwoven saturated with oil and W_O is the mass (g) of oil soaked by the nonwoven.

b) Measurement of oil sorption from artificial sea water bath in static and dynamic conditions

One litre of artificial seawater was prepared by dissolving 30 g of sodium chloride and 5 g of magnesium chloride anhydride in 1000 ml of distilled water. 500 ml of this artificial seawater was poured in a 1000 ml size beaker and 50 g of sample oil was added to it and stirred with a digital magnetic stirrer (Cole Parmer) at 200 rpm, for 5 minutes to prepare an oil in a water emulsion.

A dry nonwoven specimen of known weight (m_o) was immersed in the emulsion beaker for 10 min for

soaking in static condition. In case of dynamic condition of test, after immersing the dry sample into the emulsion beaker stirring was conducted using the same stirrer for 10 minutes at a frequency of 50 cycles/minute to simulate the actual ocean waves. After soaking, the specimen was taken out of the beaker and hanged vertically for 5 min so that excess solution can be drained out of the specimen. After that weight of the soaked specimen (m_f) was taken for analysis.

The amount of sorbed solution was extracted from the soaked specimen by squeezing with a roller squeezer at a roller pressure of 1.5 kg/cm² and collected. The solution contained both oil and water, from which the water (m_w) was separated using a separation funnel. Hence, the oil sorption capacity of sorber material was determined using equation 3.

$$\text{Oil sorption capacity} = \frac{m_f - (m_o + m_w)}{m_o} \quad (3),$$

where m_f is the mass (g) of the soaked specimen after draining, m_o is the initial dry mass (g) of the specimen and m_w is the water content (g) extracted from the specimen.

2.3.3 Theoretically defined oil sorption capacity

The oil sorption capacity of all nonwoven samples was also calculated theoretically and compared with experimental value. The theoretical oil sorption capacity of nonwovens indicates the maximum oil that a nonwoven fabric can adsorb. It is assumed that when all the pores in the nonwovens are filled with oils, the theoretical oil sorption capacity can be calculated using equation 4 [22].

$$\text{Theoretical oil sorption capacity} = \frac{v_p \times \rho_i}{v_f \times \rho_f} \quad (4),$$

where V_p and V_f indicate the volume of pores (equation 6) and fibres (equation 7) in the nonwovens, and ρ_i and ρ_f represent the density of oil and fibre respectively.

The volume of pores (V_p) in a given fabric volume (V_F) can be calculated from the porosity of the fabric (equation 5).

$$P = 1 - \frac{\rho_F}{\rho_f} \quad (5),$$

where ρ_F and ρ_f represent the bulk density of fabric and density of fibre respectively.

$$V_p = P \times V_F \quad (6)$$

$$V_f = (V_F - V_p) \quad (7)$$

2.3.4 Calculation of normalised oil sorption capacity

All nonwovens produced for this study had different levels of porosity with fibres of varying density. It was thus necessary to normalise the oil sorption capacity for comparison purposes. Normalised oil sorption capacities provide information about the effect of fibre characteristics (oleophilicity, contact angle and surface tension). The normalised oil sorption capacity can be expressed using equation 8 [9].

$$\text{Normalised oil sorption capacity} = \frac{\text{Experimental oil sorption capacity} (1 - \phi) \rho_f}{\phi \rho_i} \quad (8),$$

where ϕ denotes the porosity, ρ_i and ρ_f represent the density of oil and fibre respectively.

2.3.5 Measurement of rate of oil release from sorbed nonwovens

Drainage/release of excess oil after soaking from oil bath by nonwoven specimens was measured by hanging the specimens freely in vertical manner so that loose oil can be drained automatically with time. The amount of oil releases from the specimens was calculated by measuring gradual weight loss of the soaked samples after various interval viz. 0, 1, 3, 5, 7, 10, 20 and 30 minutes. The amount of oil retained was determined by taking the difference between the initial weight of the soaked nonwoven specimen and the weight of the specimen after drainage for predetermined time.

2.3.6 Measurement of oil sorption rate

The sorption rate is defined as the amount of oil adsorbed by the nonwovens from oil bath over a period of time. An experimental setup was fabricated to measure the sorption rate of the nonwoven specimens. The experimental setup is shown in Figure 1. A reservoir with oil was placed over an electronic scale that was connected to a computer. The known weight of the specimen was placed over a mesh. The mesh was connected with a vertical rod that hung vertically from the wicking apparatus. The bottom surface of the specimen was then placed in contact with oil in the reservoir, as shown in Figure 1. The oil from the reservoir penetrated into the specimen due to wicking/capillary pressure. The change in weight of the reservoir was recorded over time and thus the sorption rate over time was calculated.

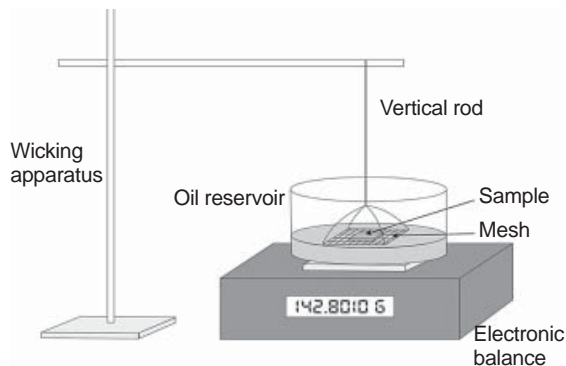


Figure 1: Instrumental setup for measurement of sorption rate

2.3.7 Measurement of recovery of absorbed oil

Oil recovery is defined as the ratio of the amount of oil recovered from a soaked specimen by mechanical squeezing to the total amount of oil soaked by the specimen. Squeezing of soaked specimens was conducted with the help of a squeezing roller keeping a roller pressure of 1.5 kg/cm². This extracted amount of oil is recovered oil (W_r). The amount of oil soaked by the specimen (W_o) was measured by the method discussed in section 2.3.2 a). Then percentage recovery of oil was calculated by equation 9.

$$\text{Oil recovery} = \frac{W_r}{W_o} \times 100 \quad (9)$$

3 Results and discussion

3.1 Nonwovens properties

An engineered fibre structure needlepunched nonwovens are flexible thick sheets which are porous, thick, bulky, and strong. They are developed in such a way that they resemble a spongy low density fabrics, wherein textile fibres are loosely interlocked via fibre entanglement without disturbing the active surface area of fibres and capillary network between the fibres much. No external adhesive was employed for fibre bonding so that surface characteristics of fibres, pore structure and capillary network are not affected and the capillary network is responsible for absorbing oil from seawater. Performance of nonwoven structure for any application depend on its mass per unit area, thickness, tensile properties etc. Average values of mass per unit area, thickness, tensile strength and breaking elongation in machine and cross directions of prepared nonwoven samples are reported in Table 4.

Table 4: Structural properties of nonwovens

Sample No.	Sample code	Sample description	GSM ^{a)} (g/m ²)	t ^{b)} (mm)	Machine direction		Cross direction	
					F _{br} ^{c)} (N/m)	ε _{br} ^{d)} (%)	F _{br} ^{c)} (N/m)	ε _{br} ^{d)} (%)
1	S1CP	Cotton parallel-laid	181.63	4.33	255.00	71.98	121.96	118.58
2	S2CC	Cotton cross-laid	187.35	3.86	117.52	127.74	265.20	69.68
3	S3FW	Cotton flat waste	187.18	3.65	119.48	64.12	55.92	96.06
4	S4C/K	Cotton/kapok (30/70)	181.78	5.33	66.24	50.42	54.28	92.16
5	S5C/K	Cotton/kapok (50/50)	181.60	4.72	141.56	59.46	80.16	95.94
6	S6C/K	Cotton/kapok (70/30)	182.62	4.22	179.32	75.74	98.36	102.14
7	S7PP	Polypropylene	289.49	7.30	9496.00	118.76	4348.00	130.80
8	S8NF	Nettle	287.20	2.43	1206.00	32.48	585.20	75.54

a) mass per unit area, b) thickness, c) tensile strength, d) breaking elongation

The results depict that all nonwoven specimens are sufficient thick and strong for the application of oil spill clean-up from seawater.

3.2 Oil sorption capacity of nonwovens from oil bath

The oil sorption capacities of all nonwovens were determined for engine oil and diesel from oil baths. The results are graphically represented in Figure 2. All the nonwovens displayed higher oil sorption capacity for high viscosity oil (engine oil) than that of low viscosity oil (diesel).

It can be observed from Figure 2 that, among all types of nonwovens, the cotton/kapok blended nonwovens exhibited the highest oil sorption capacity. The oil sorption capacity increased with an increase in kapok content (samples S6–S4). This is due to the lower bulk density of kapok enriched nonwovens and oleophilic nature of the kapok fibres. Porosity and bulk density of all nonwovens are shown in Figure 3 and Figure 4 respectively. Good correlation has been observed between oil sorption capacity of the nonwovens and their porosity. Coefficients of correlation (*r*) between sorption capacity and porosity are found to be 0.92 and 0.97 for engine oil and diesel oil respectively. Kapok fibres are oleophilic in nature and have good affinity to oil. The oleophilicity is related to the surface waxes of fibres and kapok has higher surface waxes than cotton, that makes the kapok more oleophilic [25].

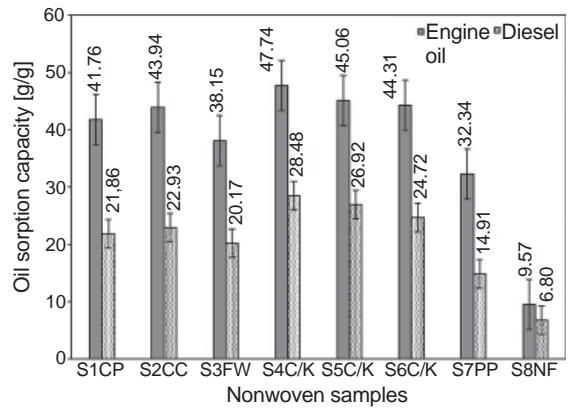


Figure 2: Oil sorption of different nonwovens from the oil bath

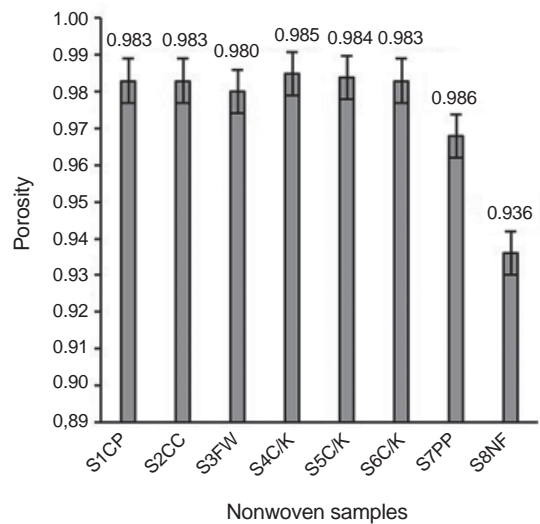


Figure 3: Porosity of nonwoven samples

All of the cotton nonwovens displayed an oil sorption capacity just below kapok blended nonwovens. The cotton flat waste nonwoven (S3FW) displayed significantly lower oil sorption than other cotton nonwovens for both engine and diesel oil because of the lower porosity and higher bulk density of the nonwovens, as shown in Figure 4. Good negative correlation has been observed between oil sorption capacity of the nonwovens and their bulk density. Coefficients of correlation (r) between sorption capacity and bulk density are found to be -0.87 and -0.79 for engine oil and diesel oil respectively. The higher bulk density is attributed to the higher number of short fibres in cotton flat waste. The cotton cross-laid nonwovens (S2CC) displayed significantly higher oil sorption capacity than cotton parallel-laid (S1CP) nonwoven for high viscosity oil (engine oil). This might be due to difference in fibre orientation. For lower viscosity oil (diesel), both nonwovens displayed similar oil sorption capacity.

The polypropylene nonwoven (S7PP) displayed significantly lower oil sorption capacity than cotton and cotton/kapok blend nonwovens. This can be explained as follows. The oil sorption capacity of a nonwoven is generally influenced by the oleophilic nature of the fibre, fibre fineness and the structure of the nonwoven fabric prepared thereof. The oleophilic nature of the fibre was one of the important factors that favourably influenced oil sorption behaviour. A structure that facilitates capillary flow should be able to adsorb more liquids. The capillary flow through a structure depends on the number of pores and their size in the structure. A structure made of finer fibre should yield more pores but with a smaller size. Thus, a structure made of finer fibre is expected to have more oil retention capacity due to higher capillary pressure. If the pore size is higher, then capillary pressure will be lower. In the present experiment, the fineness of polypropylene was 3.33 dtex, which was coarser than all other fibres. Therefore, the nonwoven prepared by the polypropylene fibres had larger pores, which was experimentally verified by the measurement of mean pore diameters, as shown in Figure 5. Hence, oils drained more easily due to a higher gravitational force than capillary pressure on account of a larger pore size. As a result, polypropylene nonwoven fabric displayed a lower oil sorption capacity than that of the cotton and cotton/kapok nonwovens.

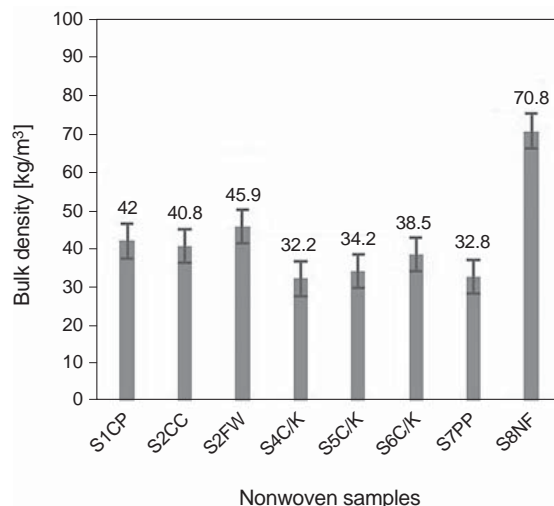


Figure 4: Bulk density of nonwovens

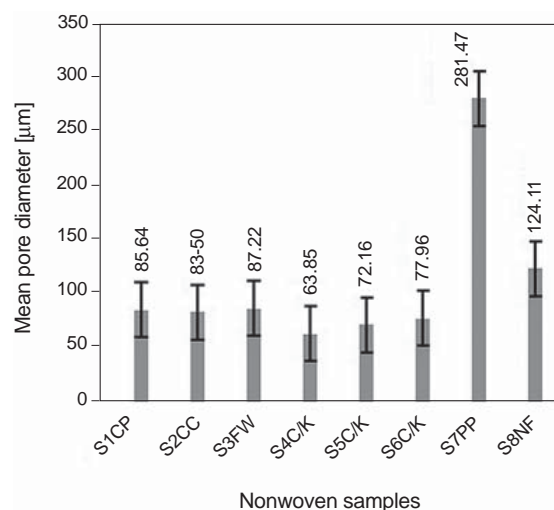


Figure 5: Mean pore diameter of nonwovens

3.3 Oil sorption of nonwovens from artificial sea water bath

Oil spills generally occur on the ocean's surface and in nearby land areas [1]. It was thus necessary to test the oil sorption capacities from oil in a water bath. The dynamic test conditions simulated the actual condition of ocean waves. The oil sorption capacities of all nonwovens from the artificial seawater bath for dynamic condition are shown in Figure 6 and on the Figure 7 the difference between oil sorption capacity from artificial seawater bath in static and dynamic condition is given.

For high viscosity oil (engine oil), all nonwovens displayed higher oil sorption under static conditions than

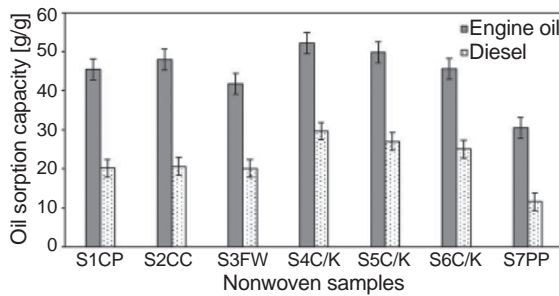


Figure 6: Oil sorption of nonwovens from artificial seawater bath under dynamic conditions

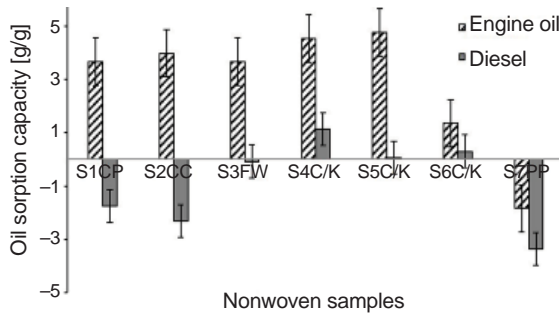


Figure 7: Difference between oil sorption of nonwovens from the static oil bath and dynamic oil in an artificial seawater bath

under dynamic conditions except polypropylene. In dynamic condition the agitation hampered the oil sorption mechanism. Polypropylene showed exceptional behaviour may be due to its hydrophobicity. For low viscosity oil (diesel), the cotton/kapok nonwovens (S4–S6) displayed higher oil sorption under static condition than that of dynamic condition due to the same reason as mentioned above. Exceptional behaviour observed in case of cotton nonwovens because agitation helps better penetration of oil inside these nonwoven structures which are relatively compact due to bulk density in higher side.

3.4 Difference between theoretical and measured oil sorption capacity of nonwovens

The oil sorption capacity of all nonwoven samples was calculated theoretically from equation 4 and then experimentally measured. The results of both theoretical and experimental sorption capacities for both high and low viscosity oil are represented in Figures 8 and 9.

It is evident from these figures that for engine oil, actual oil sorption capacity was higher than theoretical oil

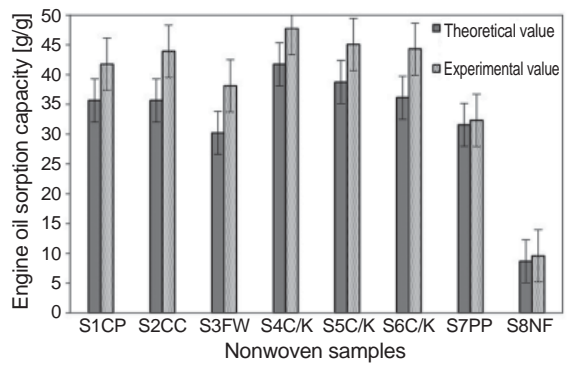


Figure 8: Theoretical and experimental oil sorption capacity of high viscosity liquid (engine oil)

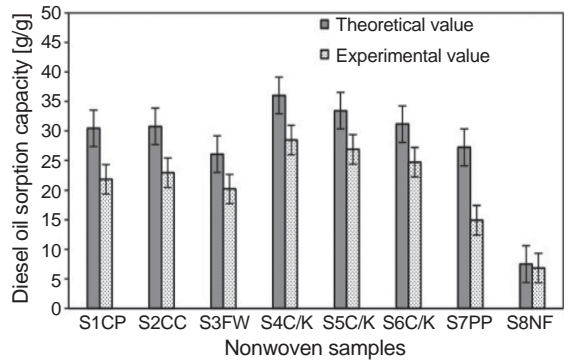


Figure 9: Theoretical and experimental oil sorption capacity of low viscosity liquid (diesel)

sorption capacity for all kinds of nonwovens whereas in case of diesel oil actual oil sorption was lower than that of theoretical. When oil is sorbed by a nonwoven structure the oil molecules are entered and occupied all pores in fibre interstices as well as attached over the surface of the nonwoven structure. As a result actual oil sorption should be higher than the theoretical value that actually happened in case of high viscosity engine oil. In case of low viscosity diesel oil due to poor surface tension there was weak bonding between diesel oil molecules and fibre surface and therefore diesel oil drain out easily from the nonwoven structure during vertical hanging and as a result actual sorption capacity become lower than that of theoretical value.

3.5 Normalised oil sorption capacity

All nonwovens produced for this study had different levels of porosity with fibres of varying density. Sorption capacity also depends on density of oil. For comparison purposes, it was thus necessary to normalise the oil sorption capacity of nonwovens to nullify the effect of density of sorbent fibres, sorbing oil and porosity of the nonwoven structure. The normalised

sorption capacities of all nonwovens are calculated as per equation (8) and shown in Figure 10.

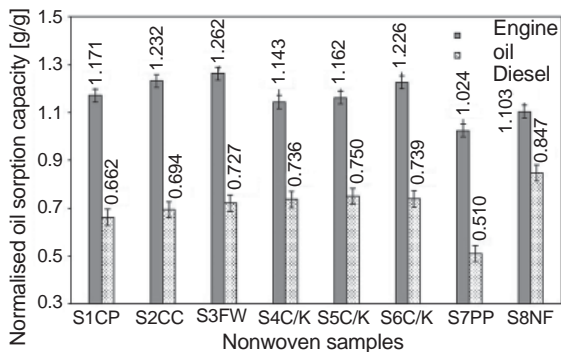


Figure 10: Normalised oil sorption capacity of nonwovens

Among cotton-based nonwovens (S1–S3), cotton flat waste nonwoven fabric (S3FW) showed the highest normalised oil sorption capacity. Though the nonwoven fabric made of cotton flat waste had more immature and shorter fibres resulting in lower porosity, a higher normalised oil sorption capacity was observed due to improved oleophilicity. An immature fibre generally contains higher surface waxes that improve its oleophilicity [23]. The sorption capacity of fibres is influenced by their oleophilic nature. In the case of cotton/kapok nonwovens (S4–S6), the normalised oil sorption capacities were found to be close to that of cotton-based nonwovens (S1–S3). This was due to both the oleophilic nature of kapok fibres and higher fabric porosity. Cotton/kapok nonwovens had a higher porosity because of poor compaction during needling on account of poor cohesiveness between kapok fibres [9, 18].

3.6 Oil sorption rate and rate of release of engine oil from the nonwovens

The oil sorption rate of the nonwovens was measured for engine oil, and the results are shown in Figure 11.

It is evident that all nonwovens adsorb engine oil more rapidly until 1 minute, followed by a slow-down in next two minutes till the nonwovens finally becomes saturated within 5 minutes. The initial steep rise in oil sorption was due to the porous structure of nonwovens that had small pores that exerted high capillary pressure. The next gradual rise in oil sorption might be attributed to larger pores. This can be explained in light of the Young-

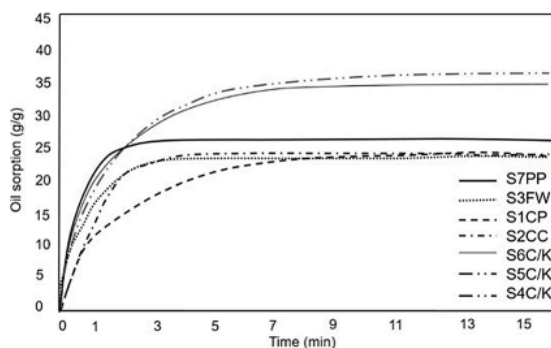


Figure 11: Engine oil sorption of nonwovens with time

Laplace equation of the relationship between capillary pressure and pore radius, as shown in equation 10 [1–2].

$$p = \frac{2 \gamma \cos \theta}{r_c} \quad (10),$$

where p indicates capillary pressure, r_c represents pore radius, θ denotes the oil contact angle and γ indicates surface tension of oil.

Hence, the oil sorption rate depends on the capillary pressure, surface tension and contact angle of liquid, while capillary pressure depends on the size of the capillary. Therefore, the differences in the oil sorption rate among the nonwovens were due to the fibre-oil contact angle and mean pore diameter of the nonwovens. In the case of high viscosity oil, all cotton nonwovens displayed a similar oil sorption rate that was significantly lower than polypropylene nonwovens (S7PP). The cotton parallel-laid (S1CP) and cross-laid (S2CC) fabrics followed an almost similar pattern of oil sorption. It is clear that the difference in fibre orientation did not cause any significant difference in the oil sorption rate in the fabrics.

The release or draining-out of adsorbed oils from the nonwovens due to free vertical hanging is approximately an inverse phenomena of oil sorption. The oil release rate of all nonwovens for high viscosity engine oil is shown in Figures 12. Each oil release curve consists of three distinct phases. First phase is the initial stage of release that occurs within 1 minute. The rate of release is highest during this period. The second or transition phase occurs from 1 to 10 minutes. During this period, the rate of release decreased substantially. The third phase represents the steady-state period. In this period, the

nonwoven sorbent tended to begin a descent towards a steady state. High viscosity engine oil drained very slowly from nonwovens, and thus reached a steady-state after 10 minutes.

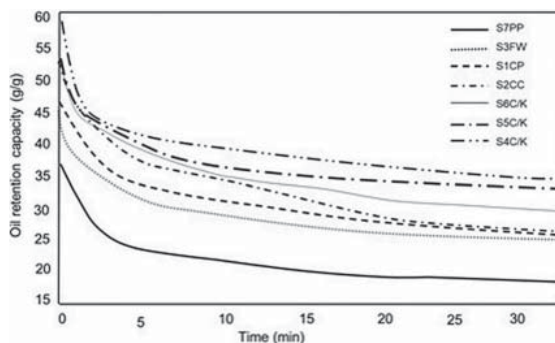


Figure 12: Release of engine oil from nonwovens with time

3.7 Oil sorption rate and retention capacity for diesel oil

The oil sorption rate of the nonwovens was measured for engine oil, and the results are shown in Figure 13. It is evident that nonwovens adsorb diesel oil very fast and reach saturation within 10 seconds. Low viscosity oil (diesel) would enter pores more quickly than high viscosity oil (engine oil), which leads to the quicker absorption of diesel oil. All nonwovens displayed a slightly higher oil sorption rate for lower viscosity oil (diesel) than high viscosity oil (engine oil). High viscosity oils were not able to adsorb upward through larger pores due to insufficient capillary pressure. The heavier oil (engine oil) would require a higher capillary pressure than lighter oil (diesel) to raise the oil to a particular height. The polypropylene nonwoven fabric (S7PP) displayed similar oil sorption rates for both engine and diesel oil, but the time taken to reach the saturation point is higher for high viscosity oil (engine oil). It was thus determined that the fibre type is a critical factor in determining the oil sorption rate.

The rate of release of diesel oil for all nonwovens is shown in a Figure 14. Each of these curves consists of two distinct phases. The first phase is the initial stage of release, which occurs within 1 minute. The rate of release is much high during this period. The second or transition zone lasts from 1 to 10 minutes. During this period, the rate of release was achieved a steady-state. Low viscosity oil (diesel) drained from nonwovens more rapidly and reached a steady-state quickly, i.e. within 1 minute.

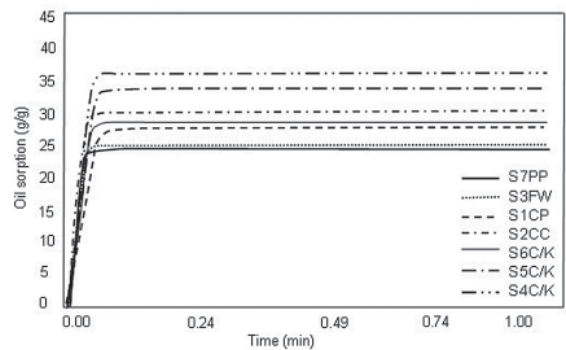


Figure 13: Sorption of diesel oil by the nonwovens with time

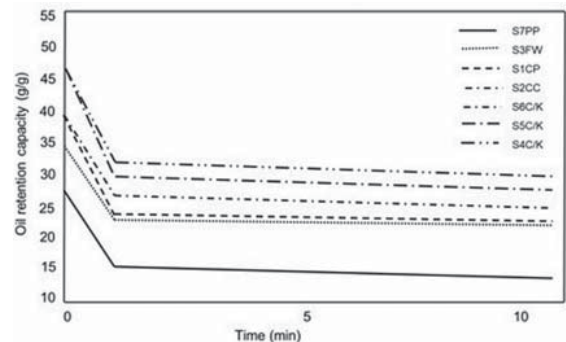


Figure 14: Release of diesel oil from nonwovens with time

The sorption of low viscosity oil (Figure 11) by all the nonwovens from the oil bath was quicker than that of the high viscosity oil (Figure 13). Also, low viscosity oil was found to drain away more rapidly during the draining period (1 minute) (Figure 12), while the draining of high viscosity oil was found to be slow (Figure 14). This is the reason for the ultimately higher oil sorption capacity for high viscosity oil exhibited by all kinds of nonwoven specimens.

3.8 Recovery of oil and reusability

The sorbed oil from nonwovens was recovered by compressing the nonwovens using a roller squeezer with a roller pressure of 1.5 kg/cm². The percentage of recovered diesel oil from different nonwovens for consecutive four sorption cycles is shown in Figure 15. The recovery of diesel for PP nonwoven in the first cycle was observed to be around 94% which was found to be lowest among all nonwovens. The oil recovery showed a higher value in second cycle is attributed to the presence of residual oil inside nonwoven structure even after the squeezing in first cycle. This is the same reason due to which 100% recovery

cannot be achieved. It can be seen from Figure 15 that oil recovery did not deteriorate much after 4th cycle of test.

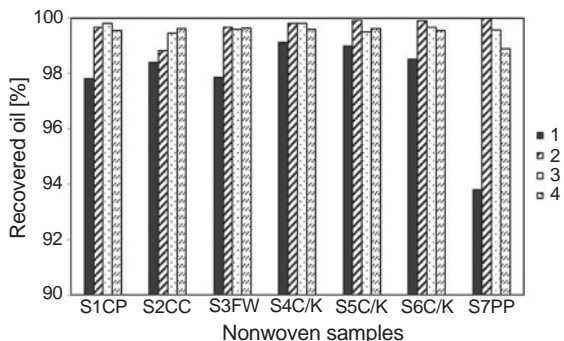


Figure 15: Percentage of diesel oil recovered from nonwovens after different cycles

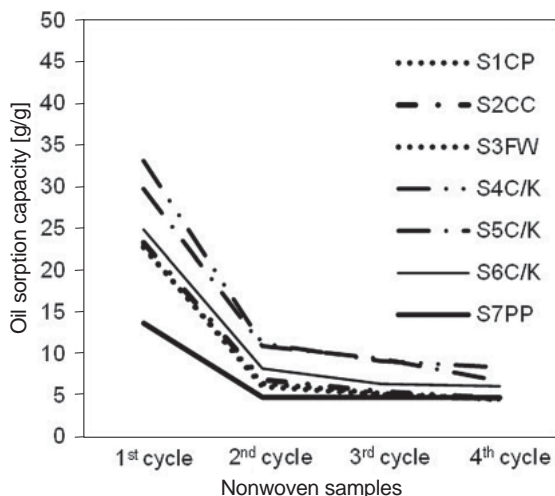


Figure 16: Reusability of nonwovens for diesel oil

An oil sorbent can be considered reusable if it can be easily compressed or squeezed to retain its original size and shape [12, 24]. Figure 16 shows the reusability of nonwovens for diesel oil. All nonwovens displayed a significant reduction in oil sorption capacities of around 50% (10 to 20 g/g) during the second cycle. The oil sorption depends on the porosity of the fabric, while the fabric porosity is in direct correlation with fabric thickness. Fabric thickness reduced after every cycle of padding, leading to a change in porosity and pore size. The flattening of pores was expected, which might result in the inability to hold much liquid. The thickness of the fabric was reduced due to padding after every cycle, which led to a reduction in oil sorption capacity. The percentage of thickness retained by nonwovens

after every cycle is given in Figure 17. The reduction in oil sorption capacities was much higher during the second cycle, while the reduction was not very significant during further successive cycles. During reuse, the reduction in oil sorption for polypropylene nonwoven fabric (S7PP) was found to be lower than in other nonwovens. The oil sorption capacity of polypropylene nonwoven fabric (S7PP), even after four cycles, was found to be lower than the nonwovens from natural fibres.

All nonwovens prepared from natural fibres displayed poor oil sorption capacity during reuse. Sorption is dependent on the availability of pores. Bulkier fabrics with similar mass per unit area should offer more oil retention sites. Nonwovens from natural fibres suffered more loss in thickness. This led

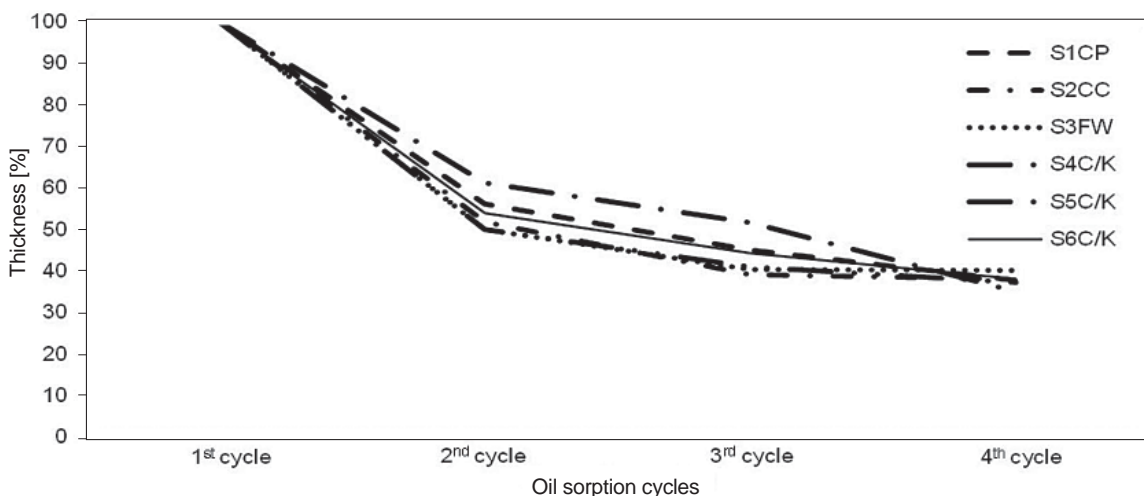


Figure 17: Percentage of thickness retained by nonwovens after every cycle of sorption

to a reduction in both porosity and the number of pores. Hence, the oil sorption capacity of nonwovens from natural fibres was lower during reuse. All nonwovens from natural fibres displayed poor compressional recovery, which needed to be improved.

4 Conclusion

All the studied nonwovens displayed significantly higher oil sorption capacity for high viscosity oil (engine oil) than that of the low viscosity oil (diesel). Nettle fibre nonwoven exhibited lowest oil sorption capacity and poor compressional recovery and therefore considered poor material for this application. Except nettle fibre nonwoven fabric (S8), all other natural fibre nonwovens (S1–S6) displayed higher oil sorption capacity than polypropylene nonwoven fabric (S7). Cotton/kapok blended nonwovens (S4–S6) were the best performer in terms of higher oil sorption and retention capacity, and oil sorption rate. An increase in kapok content in the cotton/kapok nonwoven led to a better oil sorption capacity. Even nonwovens prepared from cotton flat waste fibres exhibited very good normalised oil sorption capacity, which could open up a new door for sustainable usage of cotton waste. All these natural fibre nonwovens achieved a steady-state of sorption quickly, within 1 minute for low viscosity oil (diesel) and within 10 minutes for high viscosity oil (engine oil). In addition, more than 95% of the oils adsorbed by the nonwoven fabrics can be recovered through simple compression. During reuse, the oil sorption capacity of nonwovens gradually fell down due to thickness loss during compression. Thus, cotton/kapok fibres and cotton flat waste may be a sound choice as alternative materials to polypropylene as sea-water oil sorber in terms of low-cost, biodegradable and sustainable material.

References

1. RENGASAMY, R.S., DAS, D., KARAN, C.P. Study of oil sorption behavior of filled and structured fiber assemblies made from polypropylene, kapok and milkweed fibers. *Journal of Hazardous Materials*, 2011, **186**(1), 526–532, doi: 10.1016/j.jhazmat.2010.11.031.
2. RENGASAMY, R.S., DAS, D., RENUKA S. Nonwoven oil sorbing pads from sustainable fibrous materials for marine oil spill removal. In *Proceedings of the 37th AMOP Technical Seminar on Environmental Contamination and Response*. Ottawa : Environment Canada, 2014.
3. SEDDIGHI, M., HEJAZI, S.M. Water-oil separation performance of technical textiles used for marine pollution disasters. *Marine Pollution Bulletin*, 2015, **96**(1–2), 286–293, doi: 10.1016/j.marpolbul.2015.05.011.
4. SINGH, V., JINKA, S., HAKE, K., PARAMESWARAN, S., KENDALL, R.J., RAMKUMAR, S. Novel natural sorbent for oil spill cleanup. *Industrial & Engineering Chemistry Research*, 2014, **53**(30), 11954–11961, doi: 10.1021/ie5019436.
5. CHOI, H.M., CLOUD, R.M. Natural sorbents in oil spill cleanup. *Environmental Science & Technology*, 1992, **26**(4), 772–776, doi: 10.1021/es00028a016.
6. CHOI, H.M., MOREAU, J.P. Oil sorption behavior of various sorbents studied by sorption capacity measurement and environmental scanning electron microscopy. *Microscopy Research and Technique*, 1993, **25**(5–6), 447–455, doi: 10.1002/jemt.1070250516.
7. WEI, Q.F., MATHER, R.R., FOTHERINGHAM, A.F., YANG, R.D. Evaluation of nonwoven polypropylene oil sorbents in marine oil-spill recovery. *Marine Pollution Bulletin*, 2003, **46**(6), 780–783, doi: 10.1016/S0025-326X(03)00042-0.
8. CHU, Z., FENG, Y., SEEGER, S. Oil/water separation with selective superantwetting/superwetting surface materials. *Angewandte Chemie International Edition*, 2015, **54**(8), 2328–2338, doi: 10.1002/anie.201405785.
9. RENUKA, S., RENGASAMY, R.S., DAS, D. Studies on needlepunched natural and polypropylene fiber nonwovens as oil sorbents. *Journal of Industrial Textiles*, 2016, **46**(4), 1121–1143, doi: 10.1177/1528083715613630.
10. RADETIĆ, M.M., JOCIĆ, D.M., JOVANČIĆ, P.M., PETROVIĆ, Z.L., THOMAS, H.F. Recycled wool-based nonwoven material as an oil sorbent. *Environmental Science & Technology*, 2003, **37**(5), 1008–1012, doi: 10.1021/es0201303.
11. GE, J., YE, Y.D., YAO, H.B., ZHU, X., WANG, X., WU, L., WANG, J.L., DING, H., YONG, N., HE, L.H., YU, S.H. Pumping through porous hydrophobic/oleophilic materials: an alternative technology for oil spill remediation. *Angewandte*

- Chemie International Edition*, 2014, **53**(14), 3612–3616, doi: 10.1002/anie.201310151.
12. LI, D., ZHU, F.Z., LI, J.Y., NA, P., WANG, N. Preparation and characterization of cellulose fibers from corn straw as natural oil sorbents. *Industrial & Engineering Chemistry Research*, 2012, **52**(1), 516–524, doi: 10.1021/ie302288k.
 13. HYUNG, Min Choi, RINN M. Cloud. Natural sorbents in oil spill cleanup. *Environmental Science & Technology*, 1992, **26**(4), 772–776, doi: 10.1021/es00028a016.
 14. HYUNG, Min Choi, JERRY, P. Moreau. Oil sorption behavior of various sorbents studied by sorption capacity measurement and environmental scanning electron microscopy. *Microscopy Research and Technique*, 1993, **25**(5–6), 447–455, doi: 10.1002/jemt.1070250516.
 15. HYUNG, Min Choi. Needlepunched cotton nonwovens and other natural fibers as oil cleanup sorbents. *Journal of Environmental Science & Health Part A*, 1996, **31**(6), 1441–1457, doi: 10.1080/10934529609376434.
 16. GE, J., ZHAO, H.Y., ZHU, H.W., HUANG, J., SHI, L.A., YU, S.H. Advanced sorbents for oil-spill cleanup: recent advances and future perspectives. *Advanced Materials*, 2016, **28**(47), 10459–10490, doi: 10.1002/adma.201601812.
 17. CHOI, H.M., KWON, H.J., MOREAU, J.P. Cotton nonwovens as oil spill cleanup sorbents. *Textile Research Journal*, 1993, **63**(4), 211–218, doi: 10.1177/004051759306300404.
 18. SINGH, V., KENDALL, R.J., HAKE, K. and RAMKUMAR, S. Crude oil sorption by raw cotton. *Industrial & Engineering Chemistry Research*, 2013, **52**(18), 6277–6281, doi: 10.1021/ie4005942.
 19. LEE, Y.H., KIM, J.S., KIM, D.H., SHIN, M.S., JUNG, Y.J., LEE, D.J., KIM, H.D. Effect of blend ratio of PP/kapok blend nonwoven fabrics on oil sorption capacity. *Environmental Technology*, 2013, **34**(24), 3169–3175, doi: 10.1080/09593330.2013.808242.
 20. HYUNG Min Choi, HYO-JUNG Kwon, JERRY P. Moreau. Cotton nonwovens as oil spill cleanup sorbents. *Textile Research Journal*, 1993, **63**(4), 211–218, doi: 10.1177/004051759306300404.
 21. ANSARI, Imtiyaz Ahmad, EAST, George C., JOHNSON, D. J. Structure-property relationships in natural cellulosic fibers: part III: flax-an oil sorbent. *Journal of the Textile Institute*, 2003, **94**(1–2), 1–15, doi: 10.1080/00405000308630590.
 22. HSIEH, Y.L. Liquid transport in fabric structures. *Textile Research Journal*, 1995, **65**(5), 299–307, doi: 10.1177/004051759506500508.
 23. PRICE, J. B., CUI, X., CALAMARI, T. A., MEREDITH Jr, W. R. Cotton wax and its relationship with fiber and yarn properties: part II: wax content and yarn properties. *Textile Research Journal*, 2002, **72**(7), 631–637, doi: 10.1177/004051750207200711.
 24. CHOI, H.M. Needlepunched cotton nonwovens and other natural fibers as oil cleanup sorbents. *Journal of Environmental Science and Health . Part A: Environmental Science and Engineering and Toxicology*, 1996, **31**(6), 1441–1457, doi: 10.1080/10934529609376434.
 25. LIM, T.T, HUANG, X. Evaluation of kapok (*Ceiba pentandra* (L.) Gaertn.) as a natural hollow hydrophobic–oleophilic fibrous sorbent for oil spill cleanup. *Chemosphere*, 2007, **66**(5), 955–963, doi: 10.1016/j.chemosphere.2006.05.062.