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Effect of hydrochloric acid treated neetle fibre on oil absorbency

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Hydrochloric acid (HCl) treatment on nettle fibres has been performed to improve the oil absorbency. Box-Behnken experimental design is used to study the influence of parameters, such as treatment time, treatment temperature and concentration on oil absorbency. It is observed that the maximum oil absorbency of 15.39 g/g of nettle fibres is achieved at 2 % of HCl concentration, 75°C of treatment temperature and 60 min of treatment time. Scanning electron microscopic study reveals that the raw nettle fibre surface exhibits waxy and protruding parts, and on HCl treatment the surface becomes rougher. The influence of other parameters such as stirring speed, environmental temperature and reusability on oil absorbency is also studied. Oil sorbed nettle fibres are also subjected to soil burial tests and burning tests. In conclusion, the raw nettle fibres only show an oil absorbency of 9.25 (g/g), whereas HCl treated nettle fibres show a maximum oil absorbency of 15.39 g/g.

Keywords: Burning test, Nettle fibre, Oil absorbency, Soil burial test, Vegetable oil sorption

1 Introduction

Engineering technical textile products with the use of natural cellulose-based fibres are increasingly gaining attention due to their unique features, such as easiness of recycling, less tool wear along with the low processing cost and environmentally friendliness. There are more than 1000 varieties of plant that bears utilizable fibres[1]. Nettle is a cellulose based plant fibre with relatively large lumen available abundantly in tropical wasteland region around the world. *Urticaceace* nettle family includes more than 500 species. Of them, *Girardinia diversifolia* generate nettle fibre[2].

Development of natural sustainable woven fabrics has been made from 100 % nettle, nettle–cotton, nettle–wool, nettle–flax and nettle–ramie blends[3]. Weaving of union fabrics has been performed with nettle–cotton, nettle–viscose, nettle–flax and nettle–wool. Tight-woven structure for winter garment and open-woven structure for summer garment are broad use of these products. Nettle is also used to develop products such as hand-bags, wall-hangings, door-chain, flower vase, etc[2]. The use of nettle fibre in towel production and investigation of the performance properties was carried out by Sabir and Unal[4]. There are huge potentials to develop various textile products for different technical applications out of nettle fibres.

Needle punched nonwoven fabrics were developed from 100 % nettle fibres and its suitability in oil spill cleanups applications have been investigated in a recent study[5]. In another study, investigations on oil sorption capacity of 100% nettle and nettle/kapok needle-punched nonwovens were carried out[6]. Nettle fibres have also been blended with polypropylene fibre and the characteristic features of nettle /polypropylene blended nonwovens were studied for oil spill cleanups[7]. Oil sorption behavior of acetylated nettle fibres has been explored in recent studies[8]. Navdeep Kumar and Das[9,10] studied the physical, chemical, mechanical and structural characteristics of alkali-treated nettle fibres. Wang *et al.*[11] studied the effect of kapok fibre treated with various solvents on oil absorbency. They reported that oil absorbency of solvent treated kapok was noticeably higher than the raw kapok fibres. Previous studies in the literature showed that the investigations related to the acid treatment of nettle fibre and its influence on oil absorbency characteristics is not well documented.

In this study, hydrochloric acid (HCl) treatment on nettle fibres has been performed to improve the oil absorbency. Box-Behnken experimental design is used to study the influence of parameters such as treatment time, treatment temperature and concentration on oil absorbency.

2 Materials and Methods

2.1 Materials

The Himalayan nettle (*Girardinia diversifolia*) filaments were used as the raw materials for the

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current study. Hydrochloric acid and all other chemicals were supplied by Precision Scientific Company, Tamilnadu, India. Viscosity and density of vegetable oil used for the testing purpose were 252 cP and 0.853 g/cm^3 respectively.

2.2 Methods

2.2.1 Acid Treatment on Nettle Fibres

The nettle filaments were cut into staple fibres of 60 mm length, followed by mechanical cleaning and physical sorting. Cleaned nettle fibres were subsequently treated with hydrochloric acid solution in a batch process. In one batch, 10 g of cleaned nettle fibres were immersed into 500 mL of hydrochloric acid solution. The HCl treated nettle fibres were taken-out from the solution, squeezed and washed with distilled water. This was followed by drying. The dried nettle fibres were placed in standard testing atmospheric conditions as prescribed in ASTM D1776-04 standard. This method was followed to treat the nettle fibres with HCl solution at different time, temperature and concentration.

2.2.2 Experimental Design

To understand the individual and interactive effects of time, temperature and concentration on vegetable oil sorption, nettle fibres were treated as per Box and Behnken factorial design for three variables. Based on the preliminary investigations, the independent variables, namely time, temperature and concentration, were selected (Table 1).

2.2.3 Measurement of Oil Sorption Capacity

The oil absorbency of the nettle fibres was measured as per ASTM F 716-09 standard. To test the oil absorbency, nettle fibres were dipped into vegetable oil for 15 min and then wet nettle fibres were hung in free air for one minute to drip out the excess oil absorbed by the fibres. Subsequently, the weight (W_w) of wet nettle fibres was measured. The oil absorbency was calculated using the following relationship:

$$\text{Oil absorbency (\%)} = [W_w\text{-}W_d/W_d] \times 100$$

where W_w and W_d are the weights of wet nettle fibres and dry nettle fibres respectively.

Table 1 — Particulars of process parameters

Parameters	Levels		
	(-1)	(0)	(1)
Time, min	30	60	90
Temperature, ˚C	50	75	100
Concentration, %	1	2	3

2.2.4 Measurement of Oil Absorbency at Various Temperature Levels

A known weight of HCl treated nettle sample was placed in 150 mL of oil in a beaker for 15min at different temperature levels (10, 20, 30 and 40°C). After 15 min, the nettle fibres were taken out and kept for one minute, in order to drain out loosely held oil on the fibre surface and its mass was measured.

2.2.5 Measurement of Oil Sorption at Different Stirring Speed

In order to evaluate the effect of stirring speed on oil absorbency, known weight of HCl treated nettle sample was dipped in the oil for 15 min. The oil absorbency were obtained at different stirring speed (0, 50, 100, 150 rpm).

2.2.6 Reusability Test

The nettle fibres with oil was weighted and subsequently squeezed between two rollers at a pressure of 10 kgf/cm. The nettle fibres were then reweighed to determine the amount of recovered oil[12,13]. The squeezed nettle fibres were again used in the sorption process as before. Nettle fibres reusability was determined by oil absorbency of nettle fibres after repeated sorption and desorption cycles.

2.2.7 Scanning Electron Microscopy (SEM)

Morphology analysis of the raw and treated nettle fibre samples was carried out using field emission scanning electron microscope from PSGTECHS COE INDUTECH, Coimbatore, India. The nettle fibres were sputter coated with gold layers and figures were recorded using scanning electron microscopy.

3 Results and Discussion

3.1 Effect of Treatment Parameters on Oil Absorbency

The independent variables such as treatment time, temperature and concentration of HCl have been considered at three levels each. Based on Box-Behnken experimental design, fifteen different combinations to carry out the experiment are obtained. The influence of variables on oil absorbency is studied and presented in Table 2.

The ANOVA results of individual and interaction effects of different independent variables namely treatment time, temperature and concentration are given in Table 3. The “p” values and “f” values for various response variables are also given in Table 3. It is noted that individual effects of parameters, such as time and temperature on vegetable oil absorbency are significant, whereas the interaction effects of time *vs* temperature and temperature *vs* concentration are significant.

The coefficient of determination (R^2) of the vegetable oil absorbency (g/g) is 0.9908. From the value of R^2, it can be understood that more than 99 % of variability can be explained by the chosen variables for the variation in vegetable oil absorbency.

Table 2 — Experimental conditions and measured values

Expt. No.	Time, min (A)	Temp, ˚C (B)	Concentration % (C)	Oil absorbency g/g
1	30	100	2	10.22
2	30	50	2	13.19
3	60	100	3	10.12
4	60	50	1	12.10
5	30	75	3	10.89
6	60	100	1	12.29
7	60	75	2	15.39
8	90	50	2	13.24
9	90	100	2	11.89
10	60	75	2	15.21
11	90	75	3	11.82
12	60	50	3	14.12
13	30	75	1	12.19
14	90	75	1	11.93
15	60	75	2	15.35

Table 3 — F-values and p-values for various response variables

Source	Oil absorbency, g/g	
	F-value	p-value
Model	59.77	0.0001*
Time (A)	9.23	0.0288*
Temperature (B)	106.84	0.0001*
Concentration (C)	3.93	0.1041
AB	8.48	0.0333*
AC	4.58	0.0854
BC	56.76	0.0007*

*Indicates significant values.

Adequate precision is the measurement of signal to noise ratio, and the values greater than 4 is desirable to select the model. In this study, for all the three factors, the adequate precision is 23.862. The regression equation in terms of actual factors is shown below:

$$Y = 15.32 + 0.2987 \times A - 1.02 \times B - 0.1950 \times C - 1.82 \times A^2 - 1.37 \times B^2 - 1.79\,C^2 + 0.4050 \times A \times B - 1.05 \times B \times C + 0.2973 A \times C$$

The surface responses curves (Fig. 1) are drawn to understand the individual and interaction effects of reaction time, temperature and concentration on oil absorbency of nettle fibre using Design expert statistical software. Figure 1(a) shows the effect of HCl concentration and treatment time on oil absorbency %. With the increase in concentration of HCl and treatment time, the oil absorbency of nettle fibre increases up to a certain point and thereafter decreases. Similar type of trend also appears in case of the interactive effect between time and concentration [Fig. 1(b)] and temperature and concentration [Fig.1(c)].

External layer of natural cellulose fibres is usually covered by wax, natural oils and pectin. HCl treatment can remove these substances and thus fibrils will be exposed and generation of rough surface can be obtained[11]. Our scanning electron microscopic photographs of raw and acid treated nettle fibres are shown in Fig. 2. In Fig. 2(a) it is observed that the untreated fibre surface exhibits waxy and protruding parts. On acid treatment [Fig. 2(b)], waxes as well as cuticle on the surface are removed by the interaction with acid and surface becomes rougher. In addition,

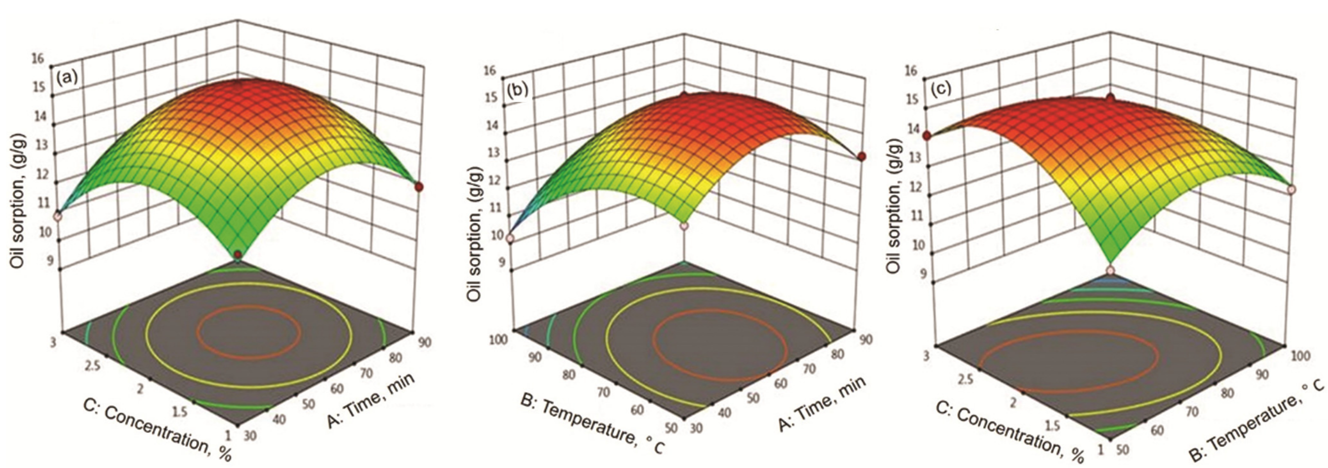

Fig. 1 — (a) Effect of time and concentration, (b) effect of time and temperature, and (c) effect of temperature and concentration on oil absorbency %

HCl treatment improves the crystalline packing order in cellulosic fibre, providing more access to diffusion of chemicals into fibre structure [14]. Hence, the oil absorbency of nettle fibres increases with treatment parameters.

However, beyond certain point, oil absorbency of nettle fibre decreases with treatment time, temperature and HCl concentration. With HCl treatment, some hydrogen bonds are destroyed due to hydrolysis, and configuration of the fibre is damaged. Also, under high temperature, HCl will remove partial hemicellulose and cellulose, leading to a shorter fibre length in morphology[15]. Thus, oil might be more difficult to be entrapped within the hollow lumen. From the responses, optimum concentration, time and temperature for maximum oil absorbency (15.39 g/g) of nettle fibres are obtained. As observed from the results, the optimum conditions are 2 % of HCl concentration, 75°C of treatment temperature and 60 min of treatment time. In case of raw nettle fibres, the oil absorbency is 9.25 (g/g) only, which is considerably lower than the HCL treated nettle fibres.

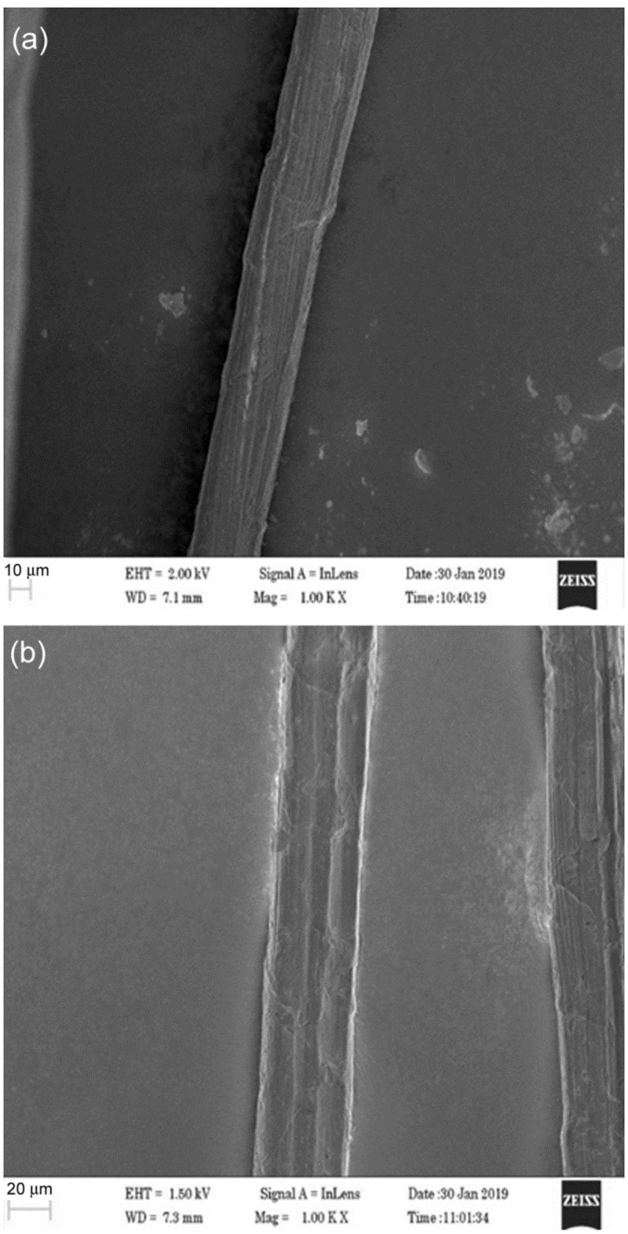

Fig. 2 — SEM Micrographs of (a) raw and (b) HCl treated nettle fibres

3.2 Effect of Other Parameters on Oil Absorbency

3.2.1 Temperature

Figure 3(a) shows the effect of the environment temperature on the oil absorbency of HCl treated nettle fibres. With the increase in temperature from 10°C to 40°C, the oil absorbency gradually decreases. This shows that higher temperature is not favorable for oil absorbency. At higher temperature, the movement of oil molecules can accelerate and can also lead to a decrease in oil viscosity[16].

3.2.2 Stirring Speed

Stirring speed is one of the major factors for oil removal from water. As the stormy waves are not similar in different places, different stirring speeds are used in this study. The oil absorbency of HCl treated nettle fibres at different stirring speed values are shown in Fig 3(b). The oil absorbency increases with the increase in stirring speed increasing from 0 rpm to 100 rpm, but significantly decreases at 150 rpm. The maximum oil absorbency of HCl treated nettle fibres is achieved at 100 rpm. At low stirring speed, oil molecules cannot be fully contacted with fibrous material and hence the oil absorbency is low. At higher stirring speed, more oil molecules can be attached to the fibrous material due to Brownian motion and oil absorbency increases[16]. However, beyond certain speed level, the oil absorbency decreases.

3.2.3 Reusability

Reusability of the fibrous material is normally governed by predicting the number of cycles that the fibrous material can tolerate before becoming unusable due to crushing, tearing or other common deterioration. Figure 3(c) shows the oil absorbency of nettle fibre for three cycles of sorption/ desorption. It is noted that after each sorption/desorption cycles, the oil absorbency of nettle fibre decreases. The contraction of fibre interspace, irreversible deformation of partial hollow lumens and the presence of residual oil in fibre bundles are related to the reduction of oil absorbency[17]. It is also noted that after three sorption/desorption cycles, the oil absorbency of nettle fibres is 8.95 g/g.

3.3 Soil Burial Test

Biodegradation is the process by which organic substances are broken down by the enzymes produced by living organisms[18]. This terminology is often used in relation to environmental remediation, ecology and waste management. Recently, lot of research is going on to develop biodegradable sorbents, which can be vanished from the surface of the earth after they are of no use. The aim of this work is to study the biodegradation behavior of nettle fibre when buried in soil, so that they can be disposed of easily with no harmful effects on the environment. Plot of biodegradation of raw nettle fibre after burial in soil is shown in the Fig. 4. It is noted that the biodegradation of raw nettle fibre is quite quick and after fifteen days it is extremely difficult to separate the fibre sample from the soil. Samples of vegetable oil covered nettle fibre are the one who are not degraded by microorganisms (Fig. 5). This may be due to the inherent characteristics of the oil. In general, biodegradation capacity of vegetable oils is considerably higher than mineral oil under aerobic and anaerobic conditions[18]. It has also been reported that vegetable oils undergo biodegradation after 28 days[18]. Hence, these materials are also subjected to burning tests.

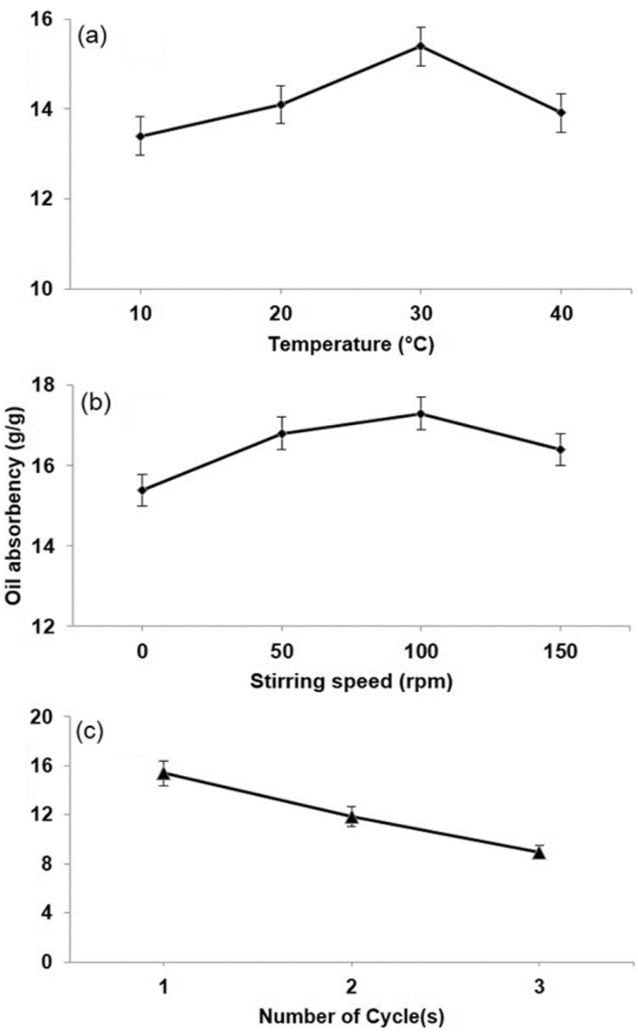

Fig. 3 — Effect of (a) temperature (b) stirring speed (c) no. of cycles (reusability) on oil absorbency

3.4 Burning Test

Pasila[19] recommended that after removing the oil from water surface, the fibrous material can be taken to bio-power plant and burned for energy. Therefore, after reusability test, the nettle fibres sample (1g) is subjected to burning test and it is observed that only small residue of ashes remains. From this study, it can be stated that the developed material will not load the land after use and disposed of easily with no harmful effects on the environment.

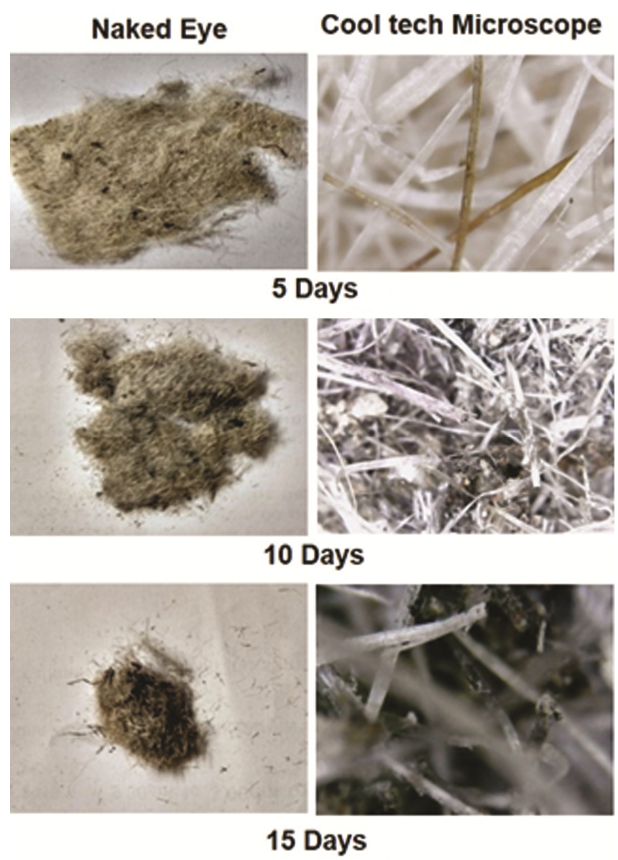

Fig. 4 — Biodegradation of raw nettle fibre after burial in soil

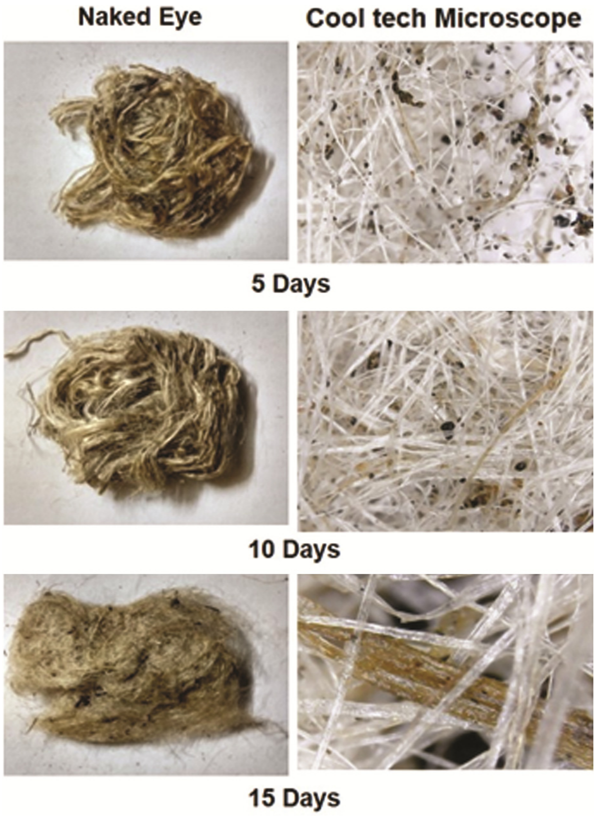

Fig. 5 — Biodegradation of vegetable oil covered nettle fibre after burial in soil

4 Conclusion

4.1 From the responses, it is noted that the maximum oil absorbency of 15.39 g/g of nettle fibres is achieved at 2 % of HCl concentration, 75°C of treatment temperature and 60 min of treatment time.

4.2 Scanning electron microscopy reveals that the raw nettle fibre surface exhibits waxy and protruding parts and on HCl treatment surface becomes rougher.

4.3 The influence of other parameters such as stirring speed, environmental temperature and reusability on oil absorbency was studied. From the reusability test, it is noted that after each sorption/desorption cycles, the sorption capacity of nettle fibre decreases

4.4 From soil burial test, it is noted that the biodegradation of raw nettle fibre is quite quick and after fifteen days it is extremely difficult to separate the fibre sample from the soil. Samples of vegetable oil covered nettle fibre are the one who are not degraded by microorganisms. From burning test results, it can be stated that the developed material will not load the land after use and disposed of easily with no harmful effects on the environment.

4.5 In conclusion, the raw nettle fibres show an oil absorbency of 9.25 (g/g), whereas HCl treated nettle fibres show a maximum oil absorbency of 15.39 g/g.

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References

1 Leonard Y, Mwaikambo & Martin P Ansell, *J Appl Polym Sci,* 84(2002)2222.
2 Debnath S, *Handbook of Sustainable Luxury Textiles & Fashion* (Springer), Vol. 1, 2016, 43.
3 Harwood J, Horne M R L & Waldron D, *Asp Appl Bio*, 101(2010)133.
4 Emel Ceyhun Sabir & Belkıs Zervent Ünal, *J Nat Fibres* 14(2017)1.
5 Viju S & Thilagavathi G, *J Inst Eng (India): Series E*, 2019. DOI: org/10.1007/s40034-018-0131-6.
6 Thilagavathi G & Praba Karan C, *J Ind Text,* 2018. DOI:org/10.1177/1528083718787532.
7 Brindha R, Thilagavathi G & Viju S, J *Nat Fibres, 2019.* DOI: 10.1080/15440478.2019.1578717.
8 Viju S, Thilagavathi G, Vignesh B & Brindha R, *J Text Inst*, 2019. DOI: 10.1080/00405000.2019.1603184.
9 Navdeep Kumar & Dipayan Das, *J Text Inst,* 108(2017a)1.
10 Navdeep kumar & Dipayan Das, *J Text Inst,* 108 (2017b)1.
11 Jinato Wang, Yian Zheng & Aiqin Wang, *Ind Crops Prod,* 40(2012)178.
12 Teli M D, Valia S P & Mifta J, *J Text Inst ,* 108(2016)1106.
13 Teli M D & Valia S P, *Carbohydrate Polym,* 92(2013)328.
14 Mwaikambo L Y & Ansell M P, *Die Angewan Makrom Chemie,* 272(1999)108.
15 Huang X F & Lim T T, *Desal,* 190(2006)295.
16 Chai W, Liu X, Zhang X, Li B, Yin T & Zou J, *Desal Wat Treat,* 57(2015) 18560.
17 Deschamps G, Caruel H, Borredon M E, Bonnin C & Vignoles C, *Environ Sci Technol,* 37(2003)1013.
18 Emmanuel Aluyor O, Kessington Obahiagbon O & Mudiakeoghene Ori-jesu, *Sci Res Essay*, 4 (2009), 543.
19 Pasila A, *Marine Pollution Bull*, 49 (2004) 1006.