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Comparative analysis of *Calotropis procera* and *Ceiba pentandra* fibre-based filters used to separate oil from emulsified effluent

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This research compared the filters made of kapok and milkweed fibres, which separated 5 μm and 2 μm droplets from oily wastewater with 5% oil concentration. Kapok and milkweed fibre coalescence filters were constructed with varying porosity and bed heights, specifically 10 mm, 20 mm, and 30 mm. The emulsion was pumped at a rate of 2 mL min^{-1} through the filter column by a peristaltic pump. Methods employed in calculating oil concentration, oil droplet size, oil saturation, experimental calculations, and other tests were conducted. This research contributes to the development of efficient filtration materials by comparing kapok and milkweed fibres for separating oily wastewater droplets. A key quantitative finding is that the milkweed fibre filter achieved a higher separation efficiency of 93%, compared to 89.6% for the kapok fibre filter, at a bed height of 30 mm and 0.98 porosity. According to the study, milkweed fibre beds had a lower oil saturation than kapok fibre filters. As the number of filter cycles increases, the oil saturation in the bed decreases significantly.

Sustainability spotlight

Industries release emulsified effluent as industrial waste, which is a major environmental hazard that has a detrimental effect on communities and the ecosystem. The goal of this effort is to explore environmentally friendly, cost-effective, and waste-to-wealth strategies that foster sustainable communities. In order to find a feasible answer, this study compares two distinct fiber-based filters that are made from natural waste fibers. Notably, kapok and milkweed fibres are biodegradable and easy to dispose of, offering a more sustainable alternative. This research emphasizes the relevance of the following UN sustainable development goals: affordable and clean energy (SDG 7), industry, innovation, and infrastructure (SDG 9), climate action (SDG 13), and life below water (SDG 14).

1. Introduction

Emulsified oily wastewater is produced in various industries, including oil and gas exploration, oil refining, and pharmaceuticals. The direct discharge of oil/water emulsions into the environment causes a severe oil pollution issue.[1,2] Oil pollution in water bodies is a major environmental issue with significant consequences for ecosystems, human health, and the economy. It results from the release of petroleum-based products, such as crude or refined oil, into aquatic environments. These oils contain harmful substances, including polycyclic aromatic hydrocarbons (PAHs) and heavy metals, which can severely impact aquatic life. These toxins disrupt the reproductive, respiratory, and digestive systems of fish, birds, and other wildlife. Moreover, oil pollution can lead to seafood contamination, posing a health risk to humans when consumed.[1] Therefore, it is essential to efficiently separate emulsified oil before its disposal into water bodies.

The two types of membranes used to separate oil-in-water emulsions are size-sieving membranes and demulsification membranes, based on their differing modes of operation. The size-sieving membrane allows the water phase to freely pass through it when the oil-in-water emulsion is in contact with it, but the membrane surface intercepts the emulsion oil droplets. The emulsion oil droplets are caught by the membrane media and progressively assemble into larger oil droplets (hundreds of microns) when the oil-in-water emulsion passes through the demulsification membrane.[3,4] Emulsions in oily wastewater are typically formed through the dispersion of oil droplets in water, stabilized by natural emulsifiers such as asphaltenes, resins, and naphthenic acids, as well as by mechanical agitation during oil extraction and processing. These emulsifiers create a protective layer around the oil droplets, preventing them from coalescing and separating from the water phase. The stability of these emulsions is influenced by factors such as the size of the oil droplets, the interfacial tension between oil and water, and the presence of surfactants or other stabilizing agents.[2] High

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shear forces during pumping and transportation can further enhance emulsion stability by reducing the droplet size and increasing the surface area for emulsifier adsorption. As a result, breaking these emulsions to separate oil from water requires specialized treatment methods, such as chemical demulsification, thermal treatment, or advanced filtration techniques.[5]

Natural fibres have attracted great interest in oil–water separation due to particular characteristics such as easy handling, flexibility, biodegradability, environmental friendliness, low cost, and high efficiency.[6] *Calotropis procera* is a plant species that belongs to the family Apocynaceae. The fibre obtained from the stem of *Calotropis procera* is a natural and renewable material with the large lumen and hydrophobic–oleophilic characteristics, providing it with a good oil absorption capacity.[7] The fibre is hydrophobic, oleophobic, and selective for oil, which makes it an excellent material for oil sorption. The fibre has been evaluated for crude oil removal on the water surface, and the results showed oil sorption capacities higher than untreated fibres.[7] Treated *Calotropis procera* fibres can be considered an alternative for the removal of oil from leaks and spills due to their high availability and excellent absorption properties for various oils.[7,8] *Ceiba pentandra*, commonly known as kapok, is a tropical tree that produces a soft, fluffy fibre used in various applications, including insulation, life jackets, and oil sorption.[7] The kapok fibre is hydrophobic and oleophilic, making it an excellent oil sorption material.[7] *Calotropis gigantea* fibre was evaluated as an oil-absorbing material for the removal of oil from water, and preliminary experiments showed that water absorption was low while oil absorption was high.[9] *Calotropis procera* fibre was evaluated for the removal of crude oil on the water surface, and the results showed oil sorption capacities higher than untreated fibres.[7,8] *Ceiba pentandra* fibres have been used in various applications, including oil sorption. The performance of kapok fibres in oily water filtration was assessed through column breakthrough time, filtration rate, filtrate quality, and the amount of oil retained by the fibres.[7] A depth filtrating system with rotatable and taper-shaped filter columns made of structured kapok filters was reported to remove and recover oil from wastewater.[8] Wang, Kui *et al.*[1] developed ultralong ceramic/polymeric fibre asymmetric membranes with good mechanical stability, fouling resistance, and high oil/water selectivity to meet the strict requirements for practical oil/water separation. Due to its integrated and interconnected structure, ceramic nanofibres/polymeric microfibres' ultra-long dimensions give this new membrane mechanical flexibility and robustness. The nanoporous selective layer of ceramic nanofibres in this membrane separates oil/water emulsions with 99.9% efficiency. Underwater superoleophobicity and ultra-low oil adherence of the ceramic-based selective layer give this membrane outstanding antifouling capabilities. High water penetration flow (6.8×10^4 L m^{-2} h^{-1} bar^{-1}) at low pressures is due to the 3D linked fibre-based structure of this membrane. This membrane's easy manufacture and low cost indicate industrial uses.[10] Saththasivam, Jayaprakash *et al.*[11] presented an eco-friendly method for removing emulsified oil from water by utilizing chitosan and beach sand. Chitosan serves as a biodegradable flocculant, while beach sand enhances floc formation and accelerates settling velocity. The method achieved 94% oil removal efficiency and reduced settling time from 90 minutes to 15 minutes with 100 mg per L^{-1} chitosan and 500 mg per L^{-1} beach sand, possessing a particle size distribution of 50–100 μm. This reduction in settling time results in lower capital expenditure compared to traditional methods. Additionally, the use of natural materials like chitosan and beach sand minimizes toxic sludge generation, making this approach a promising eco-friendly alternative for treating oily wastewater.

Saththasivam, Jayaprakash *et al.*[12] reported that carbon nanotubes (CNTs) are robust and have demonstrated promise as effective materials for oil/water separation membranes. However, according to classic fluid dynamics theory, achieving high permeation flux without sacrificing other membrane properties is challenging due to the trade-off between key membrane parameters. To overcome this challenge, they introduced a novel approach to design CNT membranes with a three-dimensional (3D) architecture. These advanced membranes not only achieve impressive oil separation efficiency (over 99.9%) but also show a substantial increase in water flux (5500 L m^{-2} h^{-1} bar), which is ten times higher than that of conventional CNT membranes. Most importantly, significant improvements are made without nanoscale membrane thickness reduction. This discovery expands oil/water separation applications for CNT-based membranes. High oil loads can rapidly saturate filters, limiting performance and requiring frequent maintenance or replacement. Elevated salinity levels can alter the surface tension of water, affecting the coalescence efficiency of fibres and potentially lowering oil capture rates. Additionally, extreme pH conditions may degrade natural fibres, weakening their structural integrity and diminishing filtration performance over time.[2]

This research contributes to the development of wastewater treatment technologies by evaluating the performance of kapok and milkweed fibres as coalescence filters for separating oily droplets. The study demonstrates how factors like porosity and bed height affect separation efficiency, with milkweed fibre filters outperforming kapok fibres. The findings offer valuable insights into enhancing oil–water separation in environmental applications. It was observed that finer droplets exhibit lower separation efficiency as they pass through the filter's inter-fibre pores more easily.

2. Materials and methods

2.1. Materials

Kapok fibres were procured from kapok trees situated at the Indian Institute of Technology in Delhi, whereas milkweed fibres were gathered from an agricultural land in Kagarol village, Agra, India. Merck Specialties Pvt. Ltd (India) supplied *n*-hexane, while Thermo Fisher Scientific Pvt. Ltd (India) provided the surfactant (Tween 80). The studies used deionized water and high-density engine oil (0.9 g cm^{-13}) sourced from a local supplier in New Delhi, India.

2.2. Preparation of oil–water emulsion

2.2.1 Emulsion droplet size 5 μm. A mixture comprising 5 mL of high-density oil and 1% of non-ionic Tween 80, employed as an emulsion stabilizer, was subjected to mechanical stirring at a speed of 1500 rpm for 30 min at room temperature in 95 mL of deionized water until no visible foam was observed. The oil–water emulsion stayed stable for three days without experiencing any phase separation when the conditions were quiescent.

2.2.2 Emulsion droplet size 2 μm. The emulsion preparation follows the same procedure as outlined in the preceding section, with one notable distinction. In this case, the mechanical stirrer operates at a higher speed of 1700 rpm for 30 min at room temperature, and the addition of oil is carried out gradually, drop by drop, utilizing a medical syringe, as opposed to the previous description.

2.3. Coalescence filter bed and filtration experiment

The selected porosities of the kapok and milkweed filter beds are 0.92, 0.95, and 0.98, corresponding to the bed's bulk densities. The fibre that had been oil pre-wetted was stuffed into the filter column. The emulsion was pumped at a rate of 2 mL min^{-1} through the filter column by a peristaltic pump. Oil concentration in the filtrate was measured using UV-vis spectroscopy. During the experiment, meticulous monitoring, and documentation of both the inlet and outlet pressures were conducted. Notably, even after the completion of the coalescence filtration process, a certain amount of oil residue persisted on the filter bed.

2.4. Characterization and measurements

The chemical structure and functional groups of kapok and milkweed fibres were determined using FTIR Spectroscopy (Thermo Scientific, Nicolet iS50, USA). The morphologies of kapok and milkweed fibres were examined using a scanning electron microscope (Zeiss EVO 50, Germany) at 20 kV. The oil concentrations in the influent and effluent were measured using a UV-vis spectrophotometer (UV-2450 Shimadzu, Japan). The maximum absorption peak was identified for each concentration required for calibration curves and the oil separation efficiency (η) was computed as follows (Singh *et al.* 2021).[13,14] The contact angle between an aligned bundle of fibres (kapok and milkweed) and water was measured using a Kruss DSA 100 (Kruss Company, Ltd., Germany) equipment at room temperature. A Mastersizer 2000 (Malvern Instruments Ltd., UK, Hydro 2000MU) was used to determine the distribution of oil droplets in the influent and effluent utilizing a laser light scattering approach. Three samples were used for each measurement, and we present the average results.

2.5 Oil saturation and oil film thickness in the filter

After the experiment, the percentage of oil saturation in the filter bed was calculated as given in eqn (1).

$$\text{Saturation (\%)} = \frac{\text{Oil volume retained by the filter bed}}{\text{Total pore volume}} \times 100 \tag{1}$$

The entire length of fibres in the filter bed and the total fibre surface area were estimated (eqn (2)), to determine the thickness of the oil layer on the outer surface of the fibres after saturation.

$$l = \frac{4M_\mathrm{f}}{\pi\left(d_o^2 - d_i^2\right) \times \rho_\mathrm{f}} \tag{2}$$

2.6. Reusability of the filter

Following the test, the oil was removed from the filter bed by 4000 rpm centrifugation. The second cycle of filtration utilized the centrifuged filter bed once again to examine the effectiveness of the oil separation process. The filter bed was centrifuged at the end of each experiment cycle. For the purpose of determining whether or not the filter could be reused, the filter bed was performed up to five times.

3. Results and discussion

3.1 Morphological structure

SEM images of kapok fibre cross-sections are shown in Fig. 1(a). SEM shows that the kapok fibre has a cylindrical shape, smooth surface, thick wax layer, wide lumen, thin wall, and oval to circular cross-section. The kapok fibre has a fine surface, hollow lumen, 20.5 ± 2 μm exterior diameter, 18.5 ± 2 μm interior diameter, and 28 ± 5 mm length. Thus, the lumen is 81% of fibre volume. Fig. 1(b) displays milkweed fibre cross-sectional SEM images. Circular or non-circular milkweed fibres varied in thickness. The average dimensions of milkweed fibre are 27 ± 5 mm for length, 24 ± 2 μm for external diameter, and 22 ± 2 μm for interior diameter. The hollow lumens of milkweed (84%), compared to kapok (81%), comprise the fibre volume. Oil could potentially be absorbed by the lumen. The oil sorption properties may benefit from such a large lumen.

3.2 Chemical composition

According to the research,[15,16] milkweed contains 48% to 55% cellulose, while kapok contains 35% to 64%. There is 18–24% milkweed hemicellulose in kapok, along with 23% pentosan or 22% xylan. Comparatively, the lignin content of kapok and milkweed is between 13% and 23%, and 13% and 21.5%, respectively. Plant cell walls have 2–4% acetyl groups, whereas kapok contains 13%. There is no proof that milkweed has more acetyl groups. *N*-Alkane, alcohol, fatty acids, aldehyde, ketone, and *n*-alkyl ester waxes are present in both fibres. However, as shown in Fig. 1(c), kapok peaks are at 2916.99 cm^{-1} and 1739.03 cm^{-1} (C=O groups in aldehydes, esters, and ketones), while Fig. 1(d) shows that the milkweed peaks are at 3000–3600 cm^{-1} (O–H stretching from alcohols, carboxylic acids, and phenols) and 2920 cm^{-1} (aliphatic CH_2 and CH_3 stretching of aliphatic

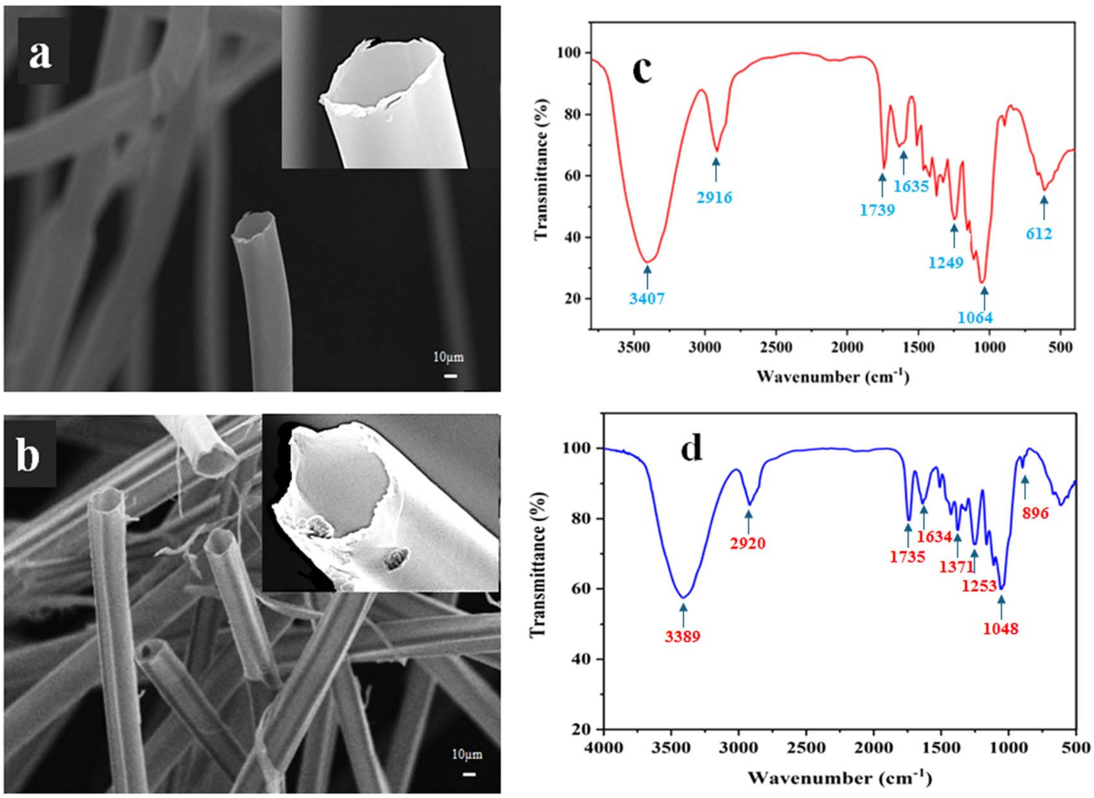

Fig. 1 SEM images of the (a) kapok fibre and (b) milkweed fibre, and the FTIR spectra of the (c) kapok fibre and (d) milkweed fibre.

compounds). The ester and carbonyl (C=O) groups, observed around 1730–1750 cm^{-1}, are associated with natural waxy substances in kapok and milkweed fibres that contribute to their hydrophobicity and enhanced oil absorption. The wax coating gives fibres their hydrophobic and oleophilic properties. Compared to milkweed, kapok's cellulose range is wider and contains more acetyl groups. Because of its higher syringyl/guaiacyl ratios than milkweed, kapok has unique lignin properties. According to FTIR peaks, different functional groups dominate the wax composition of each fibre.

3.3. Effect of porosity and filter beds on oil separation efficiency

The porosity of porous media affects the pore diameters. Since the fibrous network (filter) has a range of non-circular pore sizes, it is appropriate to use the average hydraulic pore diameter to characterize the filter bed's pore sizes.[17] At 0.98, 0.95, and 0.92 porosities, the theoretical average hydraulic inter-fibre pore diameters of the kapok filter bed were determined to be 169.74, 55.59, and 27.06 μm, respectively. The theoretical average hydraulic inter-fibre pore diameters of the milkweed filter bed were 167.66 μm at 98.0% porosity, 52.66 μm at 95.0% porosity, and 23.91 μm at 92.0% porosity. The average hydraulic inter-fibre pore diameters for both the fibre-filters are very similar. The average diameters of the lumens of kapok and milkweed fibres are 18.5 ± 2.4 μm and 22 ± 2 μm, respectively. Because of the pore size distribution, a few inter-fibre pores might be narrower than the lumen diameter, especially with filters having low porosity, 0.92. As a result, filter beds characterized by lower porosity (0.92) are expected to contain a higher proportion of smaller inner fibre pores. The separation efficiency is shown in Fig. 2(a and b) as a function of bed porosity and height with ratio of the median value of droplet size at 50% in the cumulative distribution in the influent ($D50_{in}$) 5 μm and $D50_{in}$ 2 μm.

Table 1 shows a higher separation efficiency (99.80%) observed in the milkweed fibre-based filter. The lowest separation efficiencies are observed for the highly porous filter beds (0.98 porosity), having the shortest bed height (10 mm). This is due to the least amount of fibres in the bed. This contributes to the lowest fibre surface area available for coalescence. By reducing the porosity to 0.95 (in other words, increasing the fibre surface area in the filter bed) while keeping the bed depth constant at 10 mm, a slight improvement in filtration efficiency was observed for both types of fibres.

3.4. Effect of $D50_{in}$ and pressure drop on filter beds

The separation efficiency, droplet ratio, pressure drop and saturation using kapok and milkweed filters are summarised in Table 1 for $D50_{in}$ 5. The trends are similar as observed earlier with larger influent droplet size. A shorter bed might result in less interaction between fibres and oil droplets and insufficient drop coalescence. The milkweed filter beds have higher separation efficiency (Fig. 2(a) and Table 1) than kapok filter beds with all porosity and bed heights. But the value of the separation efficiency for the kapok fibre is larger than that of the milkweed in the case of 0.92 porosity with 10 mm and 20 mm,

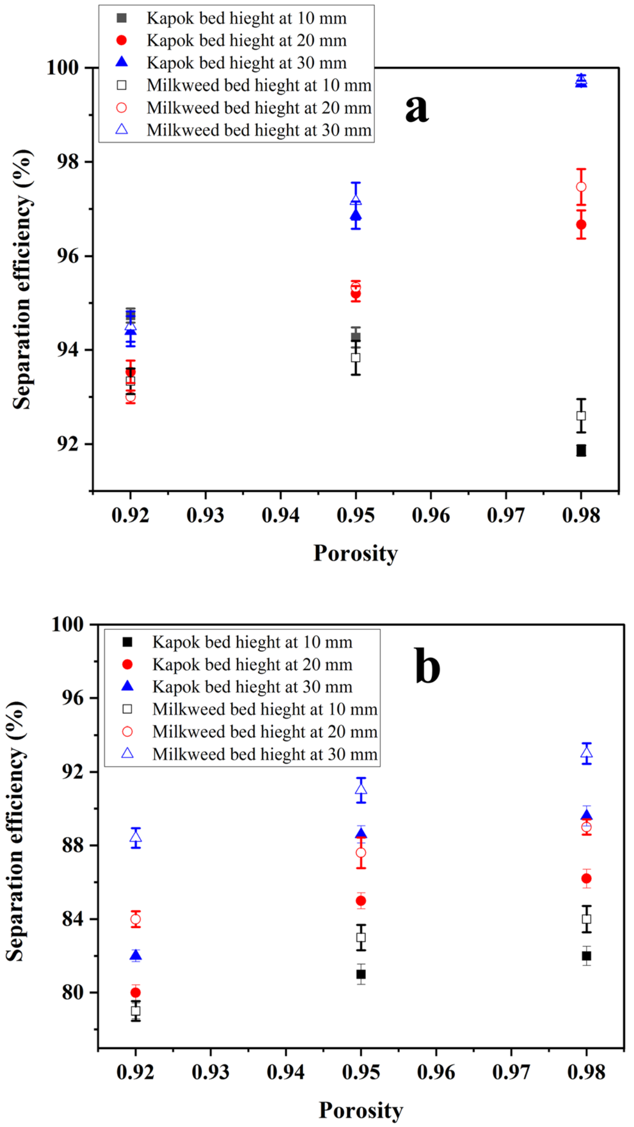

Fig. 2 Kapok and milkweed filter bed height, bed porosity on oil separation efficiency for (a) $D50_{in}$ 5 μm and (b) $D50_{in}$ 2 μm.

however there is experimental error. Increasing the bed height at medium and high porosities (0.95 and 0.98) increases proportionally the available surface area of the fibres for the coalescence. Previous studies have indicated that increasing the bed height enhances oil coalescence, as documented by Sareen *et al.* [13,18,19] As shown in Table 1, in both the kapok and milkweed filters, increasing the bed height has a greater effect on reducing the droplet ratio in the effluent at higher porosity (0.98) than at lower porosity (0.92 and 0.95). The pressure drop across the filter bed increases sharply while increasing the bed depth at lower porosities, this ejects the droplets before being coalesced into larger droplets by the dominant hydrodynamic forces on the droplets. Similar observations were noticed for $D50_{in}$ (2 μm) as shown in Table 2.

At higher packing densities of the filter bed (porosities of 0.92 and 0.95), the hydraulic pore sizes between the fibres tend to be smaller. This results in reduced permeability, as evidenced by the higher-pressure drops observed across the filter. The milkweed filter exhibits slightly lower pressure drops than the kapok filter for all the combination of filter depth and porosities. In the case of a porosity of 98%, extending the bed height from 10 mm to 30 mm leads to a decrease in the D50 droplet ratio. Specifically, for the kapok filter bed, the D50 droplet ratio is reduced from 0.28 to 0.22, while for the milkweed filter bed, it decreases from 0.26 to 0.21 as expressed in Fig. 3(a and b). This significant reduction in droplet size in the effluent indicates that the probability of smaller droplets being caught on the fibre surfaces and coalesced increased with a deeper bed, thereby enhancing the efficiency of oil separation. Due to the increased surface area of the fibres and an extended duration for droplet release, droplets have more time to coalesce and adhere together. As a result, droplets experience a prolonged coalescence process, allowing them to grow before being released.

As shown in Table 2, there is a possibility of exposed lumens that were not filled with oil, deviating from the assumed condition. In the case of filter beds with smaller porosities, this is especially true. Since the filter bed has 0.92 porosity, it is appropriate to say that the estimated oil layer thickness on the outer surface of the fibres is underestimated. The data show

Table 1 Separation efficiency, droplet size ratio, steady state pressure drop, and oil saturation of kapok (K) and milkweed (M) filters (influent oil concentration 5% and $D50_{in}$ 5 μm)

Porosity	Bed height (mm)	Separation efficiency (%)		Droplet ratio ($D50_{eff}/D50_{in}$)		Steady state pressure drop (kPa)		Saturation of oil in the filter (%)	
		K	M	K	M	K	M	K	M
0.98	10	91.87	93.33	0.28	0.26	9.6	9.4	63.78	49.2
	20	96.67	98.20	0.29	0.25	11.0	10.7	35.53	31.2
	30	99.67	99.80	0.22	0.21	11.7	11.0	24.95	21.4
0.95	10	94.27	94.33	0.27	0.28	11.9	11.5	86.89	60.1
	20	95.20	95.53	0.23	0.25	18.6	17.1	44.69	35.8
	30	96.87	97.20	0.22	0.21	22.1	21.4	30.39	25.0
0.92	10	94.73	93.67	0.28	0.29	20.5	19.0	90.31	66.8
	20	93.53	93.00	0.23	0.27	20.8	20.5	46.30	45.7
	30	94.40	95.33	0.21	0.23	23.2	23.0	31.68	31.0

Table 2 Separation efficiency, droplet size ratio, steady state pressure drop for kapok (K) and milkweed (M) filter saturation (influent oil concentration 5% and $D50_{in}$ 2 μm)

Porosity	Bed height (mm)	Separation efficiency (%)		Droplet ratio ($D50_{eff}/D50_{in}$)		Steady state pressure drop (kPa)		Saturation of oil in filter (%)	
		K	M	K	M	K	M	K	M
0.98	10	82.0	84.0	0.54	0.53	9.5	10.0	44.8	46.8
	20	86.2	89.0	0.52	0.50	9.8	10.5	24.6	26.5
	30	89.6	93.0	0.43	0.40	10.7	10.7	17.4	19.4
0.95	10	81.6	83.0	0.44	0.48	11.1	11.3	52.5	51.5
	20	85.0	87.6	0.36	0.38	14.5	16.0	32.2	31.6
	30	88.6	91.0	0.30	0.33	18.6	19.5	24.3	24.0
0.92	10	79.0	79.0	0.41	0.41	16.1	16.5	58.7	56.5
	20	80.0	84.0	0.34	0.35	18.2	19.0	41.5	40.9
	30	82.0	88.4	0.26	0.28	18.9	19.9	29.5	28.9

that the porosity of the filter bed correlates with an increase in oil film thickness. As the bed height increases, the oil film thickness decreases.

3.5. Effect of bed height on oil saturation in the filter bed

The oil saturation decreases as the bed length and porosity increase, as seen in Fig. 4(a and b). In the case of longer bed

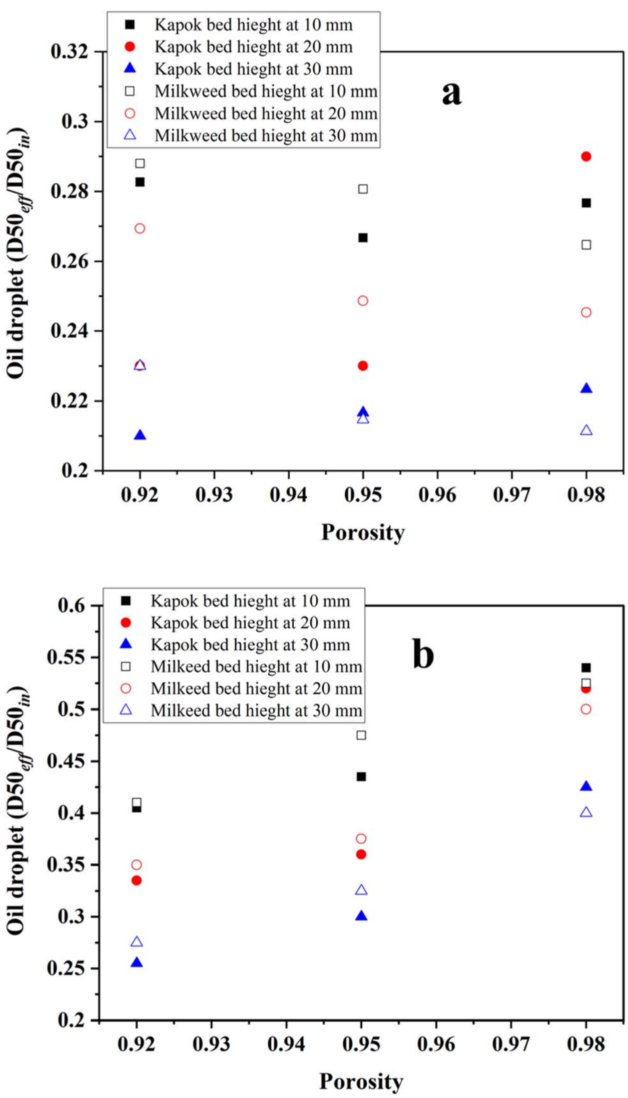

Fig. 3 Dispersed oil droplet size ratio and bed porosity interaction with (a) $D50_{in}$ 5 μm and (b) $D50_{in}$ 2 μm.

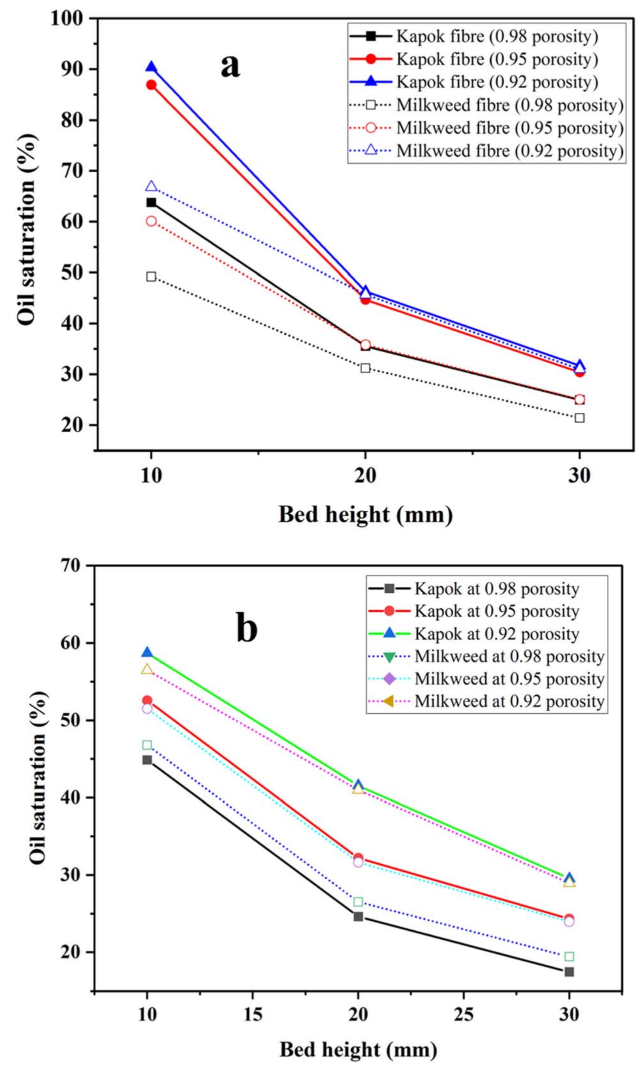

Fig. 4 Oil saturation *vs.* bed height and bed porosity for (a) $D50_{in}$ 5 μm and (b) $D50_{in}$ 2 μm.

Table 3 Oil extracted from the kapok (K) and milkweed (M) filter bed by centrifuge

Porosity	Bed height (mm)	Intra-fibre pore volume (cm^3)		Inter-fibre pore volume (cm^3)		Total pore volume (cm^3)		Oil extracted from the bed by centrifuge (ml)	
		K	M	K	M	K	M	K	M
0.98	10	0.32	0.51	4.48	4.29	4.80	4.80	3.07	2.4
	20	0.66	1.03	8.96	8.59	9.62	9.62	3.42	3.0
	30	0.98	1.54	13.44	12.88	14.42	14.42	3.60	3.1
0.95	10	0.82	1.29	3.84	3.37	4.66	4.66	4.05	2.8
	20	1.64	2.57	7.68	6.75	9.32	9.32	4.17	3.3
	30	2.46	3.86	11.52	10.12	13.98	13.98	4.25	4.0
0.92	10	1.31	2.05	3.20	2.46	4.51	4.51	4.08	3.0
	20	2.62	4.12	6.40	4.91	9.02	9.03	4.18	4.1
	30	3.94	6.18	9.60	7.36	13.54	13.54	4.29	4.1

Table 4 Surface area of fibres and oil film thickness in the kapok (K) and milkweed (M) filters after oil saturation

Porosity	Bed height (mm)	Surface area of all fibres (m^2)		Outer surface area of all fibres (m^2)		Oil film thickness after saturation (μm)	
		K	M	K	M	K	M
0.98	10	0.2	0.2	0.10	0.10	25.7	18.1
	20	0.39	0.39	0.20	0.20	12.4	9.6
	30	0.59	0.59	0.31	0.31	7.5	5.0
0.95	10	0.49	0.49	0.26	0.26	11.6	5.9
	20	0.98	0.98	0.51	0.51	3.9	1.5
	30	1.47	1.47	0.77	0.77	1.3	0.2
0.92	10	0.78	0.78	0.41	0.41	5.7	2.3
	20	1.57	1.57	0.83	0.82	0.9	0.0
	30	2.35	2.35	1.24	1.23	−0.7	−1.6

depth, the oil droplets that do not coalesce upstream of the bed can encounter fibres downstream and coalesce into bigger droplets. Further, the residence time for a droplet to grow into a bigger size is longer in the case of a longer bed.

Other authors [20] have shown that the bigger droplets in fibrous beds break off from the surface, move through the continuous phase, and don't stick back to the surface. This decreases the surface's oil and improves the separation efficiency. Oil saturation is higher at lower porosities due to a reduction in inter-fibre pore volume. The oil saturation level in the kapok filter is greater than that in the milkweed filter due to the higher inter-fibre pore volume in the kapok fibre. The lower intra-fibre pore volume produced higher capillary pressure in the kapok fibre than in the milkweed fibre (Table 3). The kapok fibre accumulated higher oil in its structure due to its lower intra-fibre pore volume.

The oil film thickness on the outer surface of kapok fibres (25.7 μm) and milkweed fibres (18.1 μm) is shown in Table 4, respectively. To ascertain the film thickness and gain insight, an assumption was made that all the lumens would be filled with oil following filtration. However, it should be noted that this may not reflect the actual scenario, as certain fibres could have collapsed due to the pressure applied during their insertion into the filter column.

3.6. Oil recovery by filter beds with $D50_{in}$ droplet ratio

Fig. 5 shows the quantity of oil retained in the kapok and milkweed filter beds that were recovered from the effluent following the experiment. Due to the available fibre surface area, the amount of oil in the filter bed directly correlates with bed height. Fig. 5(a) shows that at the same bed height (with 0.98 porosity), the kapok filter retained more oil in its structure than the milkweed filter, while the milkweed filter seeped more oil out of its structure than the kapok. The milkweed fibre is more efficient in coalescing the oil than the kapok fibres as indicated by the data on separation efficiency, D50 droplet ratio and saturation of the filter.

Fig. 5(a) and (b) show oil retention in kapok and milkweed filter beds with droplet ratio $D50_{in}$ and different porosities. The filter bed's oil content is proportionate to its height due to the fibre surface area. Kapok filters held slightly more oil than milkweed filters. High intra-fibre pore space is found in milkweed filter beds. As milkweed and kapok filter beds become more porous, floating oil collection decreases. All porosities see less floating oil from kapok and milkweed as the bed height rises. Thus, the milkweed filter gathered more floating layer oil than the kapok filter. Milkweed is more hydrophobic than the kapok fibre. Fig. 6 shows that the milkweed fibre (147°) water contact angle is higher than that of the kapok fibre (143°). So,

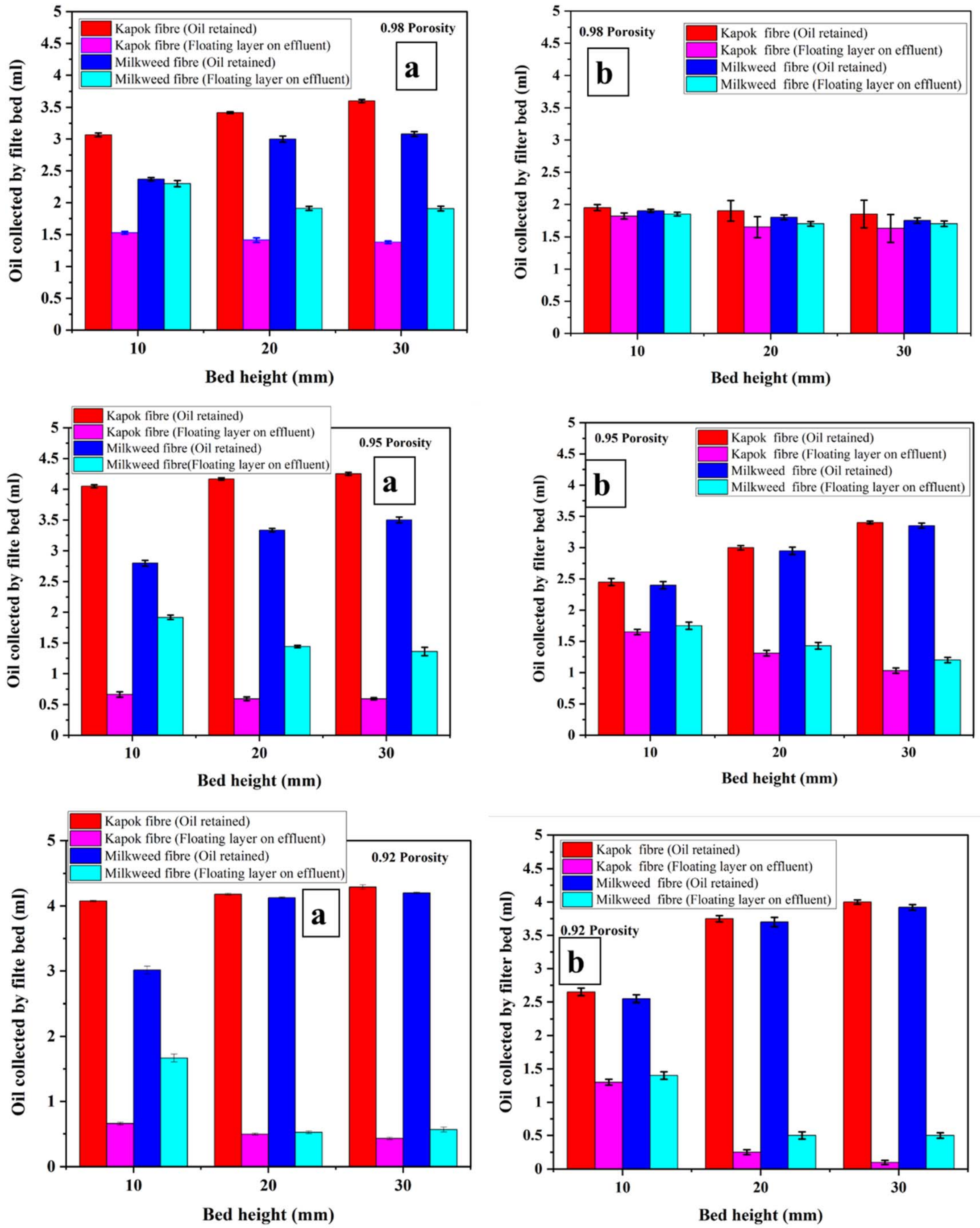

Fig. 5 Oil retained by kapok and milk weed filter beds of different heights, bed porosity with oil droplet size: (a) $D50_{in}$ 5 μm and (b) $D50_{in}$ 2 μm.

the milkweed is more hydrophobic than the kapok fibre and separation efficiency is also slightly higher than that of the kapok fibre.

At an identical porosity of 0.95, milkweed fibres exhibit a lower retention of oil within the structure, primarily due to the occurrence of draining in the effluent as the floating oil. Fig. 5(c) shows the lower porosity of the filter bed (0.92); more oil droplets accumulate inside the kapok and milkweed filters. Table 4 shows that the fibre volume is more in the kapok and milkweed filter beds with 92% porosity; oil is firmly retained by intra and inter-fibre pores but fails to flow through the structure. For all porosities, the amount of floating oil decreases as the bed height increases. The kapok filter bed retained a higher amount of oil that was extracted by centrifugation as compared to the milkweed filter bed (Table 4).

3.7. Effect of influent oil droplet size ($D50_{in}$)

Smaller droplets exhibit strong buoyancy due to their low settling, rising, or terminal velocity, causing them to take longer

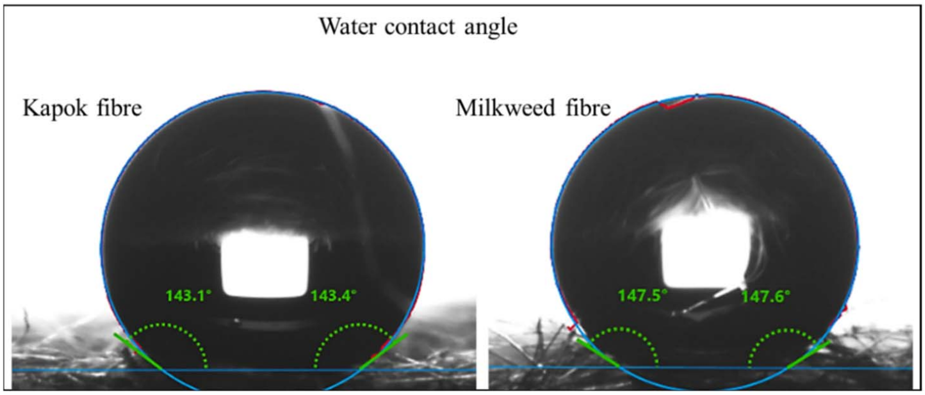

Fig. 6 Water contact angle on kapok and milkweed fibres.

Table 5 Effect of influent oil droplet size on kapok (K) and milkweed (M) filter bed efficiency

Influent droplet size (μm)	Porosity with bed height 30 mm	Oil droplet D50 ratio (μm)		Separation efficiency (%)	
		K	M	K	M
2	0.98	0.43	0.40	89.6	93.0
	0.95	0.30	0.33	88.6	91.0
	0.92	0.26	0.28	82.0	88.4
5	0.98	0.22	0.21	99.67	99.80
	0.95	0.22	0.21	96.87	97.20
	0.92	0.21	0.23	94.40	95.33

to reach the fibre surfaces. Conversely, larger droplets reach the filter surface faster and have longer residence times, favoring higher separation efficiency. As shown in Table 5, across all porosities, 2 μm droplets consistently demonstrate lower separation efficiency compared to 5 μm droplets. Notably, the D50 ratio decreased significantly for the 5 μm influent oil droplets, highlighting the enhanced separation performance for larger droplet sizes.

As the influent droplet size decreases, the separation efficiency of both kapok and milkweed fibres declines. However, the milkweed fibre consistently demonstrates superior efficiency across all droplet size ranges compared to the kapok fibre. At a porosity of 0.98, the D50 droplet ratio for the kapok fibre is higher than that for milkweed, although this is attributed to experimental error. A lower D50 droplet ratio indicates better filter performance, signifying that only smaller droplets escape the filter. A low droplet ratio in the effluent indicates higher phase separation efficiency and better filtration performance. In contrast, a higher droplet ratio reflects poorer filtration performance and reduced separation efficiency.

However, at the lowest porosity (0.92), increasing the bed height, the pressure drop increases, which in turn ejects smaller droplets (both uncoalesced and partially coalesced) into the downstream, which might reduce the separation efficiency. On the other hand, the proportional increase in the fibre surface area with the bed depth brings the advantage of larger fibre surface area for the droplets to be caught and coalesced. The conflicting outcome could not increase the oil separation efficiency. The longer fluid residence time with increasing bed depth at high bulk densities of the filter bed (low porosity) does not effectively contribute to the coalescence process. The oil separation efficiency ranges from 91.87% to 99.67% for the kapok bed and 93.33% to 99.80%. For the milkweed filter bed, the latter scores slightly better than the former.

3.8. Reusability of the filter bed

To assess the filter bed's ability to be reused, a kapok and milkweed filter bed with a height of 30 mm and a porosity of 0.98 was used for $D50_{in}$ 5 μm. A series of five filtering tests were conducted on the filter bed. Prior to each examination, centrifugation was employed to remove the oil from the filter, ensuring its clearance and preparing it for the subsequent test. As indicated in Table 6 and Fig. 7, the oil separation efficiency decreases after the first usage and decreases after each reuse cycle.

Long-term degradation of filter performance can occur due to material wear during repeated filtration cycles. Mechanical stress, fibre deformation, and surface fouling may reduce the structural integrity and coalescence efficiency of fibres over time. Factors such as pore collapse, reduced porosity, and fibre breakage can hinder oil capture and separation efficiency. Additionally, oil residue buildup may cause irreversible saturation, reducing the filter's ability to function optimally. Periodic cleaning, regeneration, or replacement strategies could be explored to maintain long-term filtration efficiency.

The results show that the oil separation slightly decreased as the number of reuses increased. The D50 droplet ratio in the

Table 6 Reusability of kapok (K) and milkweed (M) filter beds on filtration performance ($D50_{in}$ 5 μm and 5% influent oil content)

No. of reusability cycles	Separation efficiency (%)		Droplet ratio ($D50_{eff}/D50_{in}$)		Steady state pressure drop (kPa)		Saturation of oil in filter (%)	
	K	M	K	M	K	M	K	M
1	99.07	99.40	0.23	0.27	10.7	10.2	25.19	21.03
2	97.87	98.27	0.25	0.31	10.6	9.3	24.77	19.99
3	96.73	96.67	0.26	0.36	10.4	9.1	24.31	19.87
4	94.67	95.20	0.26	0.42	9.4	8.8	24.15	19.30
5	91.00	91.67	0.27	0.42	8.6	8.3	23.46	19.06

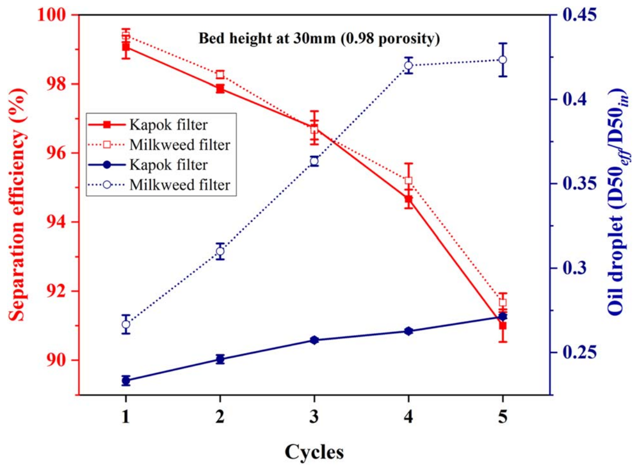

Fig. 7 Efficacy of filter beds on repeated use: filter beds with 30 mm depth and 0.98 porosity for $D50_{in}$ 5 μm; kapok filter bed and milkweed filter bed.

effluent has risen significantly, and the pressure drop across the bed has reduced from the first cycle to the fifth cycle, suggesting that the bed's physical structure is deteriorating. The non-uniformity in the structure of the filter bed after repeated use contributed to some larger inter-fibre pores that allowed a few larger oil droplets to escape through the filter and to reduced pressure drop. The saturation of oil in the bed is slightly reduced with increasing cycles of the use of the filter. The study shows that milkweed fibre beds have a greater separation efficiency than kapok fibre filters but a lower oil saturation. Kapok and milkweed fibre-based filters offer significant environmental advantages due to their renewable sourcing, low production energy requirements, and biodegradability. However, their practical application faces challenges related to durability and scalability compared to synthetic alternatives like polypropylene (PP). As PP is non-biodegradable, it contributes to landfill accumulation and environmental pollution unless properly recycled. Conducting a lifecycle analysis (LCA) can provide valuable insights for selecting sustainable filter materials.

4. Conclusions

This study examined the coalescence filtration performance of kapok and milkweed fibre beds in separating oil from emulsions under varying porosities and bed heights. The findings reveal that the oil separation capability of the filter beds improves with higher porosity and bed height. Notably, filter beds with lower porosity demonstrate a greater saturation of oil within the bed. However, increasing the bed height at lower porosity (92%) does not significantly enhance the oil separation efficiency. This is attributed to the high-pressure drop, which expels larger droplets into the effluent, coupled with a limited availability of fibre surfaces for effective coalescence to occur. The potential for using kapok and milkweed filters to coalesce fine oil droplets present in stable oily emulsions is significant. The separation efficiency for kapok and milkweed filter beds was up to 89.6% and 93% at 0.98 porosity with 30 mm bed height. The separation efficiency is found to be poor with finer droplets as they can escape easily through the inter-fibre pores of the filter. The milkweed filter beds perform slightly better than the kapok filters. The filter beds after repeated uses exhibit lower oil separation.

Data availability

All data included in this study are available upon request by contact with the corresponding author.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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