Journal of Hazardous Materials B137 (2006) 820–826

Journal of Hazardous Materials

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In situ oil/water separation using hydrophobic–oleophilic fibrous wall: A lab-scale feasibility study for groundwater cleanup

Teik-Thye Lim *, Xiaofeng Huang

Environmental Engineering Research Centre, School of Civil & Environmental Engineering, Nanyang Technological University, 50 Nanyang Avenue, Singapore 639798, Republic of Singapore

Received 24 November 2005; received in revised form 1 March 2006; accepted 1 March 2006
Available online 18 April 2006

Abstract

Kapok, a natural plant fiber, possesses excellent hydrophobic–oleophilic characteristics. Its innovative use as hydrophobic–oleophilic wall that allows permeation of oil but not water into an oil recovery well is proposed. Its performance was investigated through laboratory experiments, in which diesel was used as the experimental oil. A two-dimensional hydraulic flume was setup to physically model the oil/water separation by the kapok wall. The influences of packing density, kapok wall thickness and oil thickness on the oil recovery rate were examined. The oil permeability of the packed kapok decreased from 0.0165 cm^2 at 34 g/L packing density to 0.0038 cm^2 at 70 g/L packing density. The kapok wall exhibited complete rejection of water while allowed oil to permeate through. The excellent oil/water separation by the kapok wall was due to surface interaction between the kapok fibers and the oil, which resulted in spontaneous penetration and permeation of the oil through the kapok wall. The oil recovery rate increased with thickness of the oil layer in the feed stream. When the oil thickness exceeded 60 mm, a constant flux of 3.8–5.0, 3.2–3.3 and 2.5–2.7 L/(m^2 min) could be achieved by the kapok wall of 55-, 75- and 95-mm thick, respectively, under the natural pressure gradient. The kapok wall could be reused for several wetting/drying cycles, and only lost 27% of its initial oil permeability.

Keywords: Oil/water separation; Immiscible liquids; Kapok; Groundwater; Hydrophobic; Oleophilic; Selective filtration

1. Introduction

Inland oil spills have been deteriorating land and groundwater quality. Their occurrences can be caused by leakages of oil conduits and storage facilities, human mistakes, illegal dumpings, natural disasters or vandalisms. The spilled oils may percolate into the subsurface and eventually form free product within the capillary fringe or mount above the water table [1]. They are immiscible with water and commonly referred to as light non-aqueous phase liquids (LNAPLs). They are sources of many groundwater contaminant plumes and thus need to be removed or contained prior to groundwater cleanup.

Cleanups of spilled oils from the subsurface are much more complicated and costly than from the surface waters. Excavation of soil to expose the spilled oil for skimming off is a common practice for the sites with high groundwater table, but it disturbs site operations. Other conventional technologies such as air sparging, pump-and-treat and soil flushing with co-solvent or surfactant either are ineffective, producing large volume of liquid to be treated above ground, or causing dissolution and dispersion of the oil. In principle, removal of oils from the subsurface saturated zone can best be carried out using a free product recovery system that only causes minimum site disturbance. In this context, a recovery well has to be installed and penetrated into the target treatment zone. A handful of such technologies have been invented [2]. There are two broad categories of the recovery systems. One of which involves a downhole recovery system with either single or dual pumps. In a dual pump system, one pump is used for withdrawing groundwater from a greater depth to create draw-down and the other pump with floatation intake is used for extracting the floating oil [3]. The pumping operation while increases the oil recovery rate, causes smearing of the pollutant in the previously uncontaminated zones [4]. In addition, the system tends to recover both the oil and water from the recovery well for above-ground separation, and therefore entails additional capital and treatment cost. The other recov-

* Corresponding author. Tel.: +65 6790 6933; fax: +65 6791 0676.
E-mail address: cttlim@ntu.edu.sg (T.-T. Lim).

0304-3894/$ – see front matter
doi:10.1016/j.jhazmat.2006.03.008

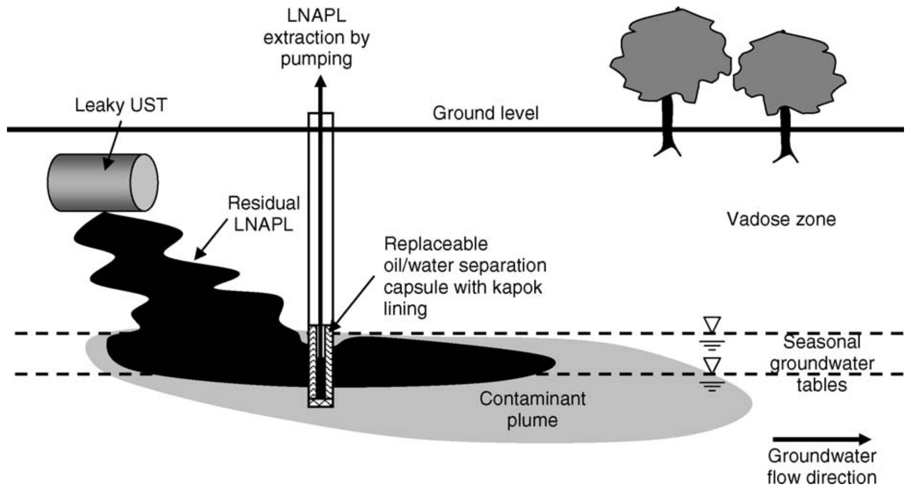

Fig. 1. Schematic illustration of proposed in situ oil/water separation with downhole kapok filter capsule for oil recovery.

ery system involves skimming devices that only remove the oil from the recovery well. The oil recovery rate is low with such system because it relies on the usually small natural hydraulic gradient and radius of influence around the well. Another drawback is that the skimmers may recover oil as well as water if the oil slick is very thin. In recent years, hydrophobic membrane screen has been developed and used to recover oil from the oil recovery well [5]. The drawback with this membrane is the irrecoverable membrane fouling and membrane crimping [6].

We propose a downhole, modular oil/water separation unit consisting of a perforated capsule (casing) with kapok lining on its inner face and bottom end. It is essentially like a household water filtration unit, except that the casing is perforated. The concept of this remediation system is to install a recovery well into the subsurface such that its well screen is embedded within the NAPL layer. This scheme is as illustrated in Fig. 1. The oil/water separation unit is made hydrophobic–oleophilic so that it only allows oil to permeate through but not water. The module can be attached permanently to the recovery well screen or made retrievable for replacement. It does not require a skimming device. The permeant (primarily NAPL) can be delivered to the ground surface by pumping mechanism.

Conventionally, synthetic fibers such as polypropylene, polyester and polyamide (nylon) are used as the filter media for oil. They are non-biodegradable and face end-of-use disposal problem. Natural vegetable products that show hydrophobic–oleophilic characteristics have been found suitable for oil/water separation. Varghese and Cleveland [7] reported that the filter medium made of kenaf could remove 70–95% of oil from surfactant-stabilized oil-in-water emulsions. Deschamps et al. [8] used cotton as a filter bed material to recover oil from oily water, and the effluent was almost free of oil. Pasila [9] studied reed canary grass, flax and hemp fiber and found that the fibrous materials were able to separate oil from water during filtration of oily water. Ribeiro et al. [10] reported that large surface area, hydrophobicity, expandability and hair-like surface of Salvinia, a hydrophobic aquatic plant, were the contributing factors to its excellent oil/water separation.

Kapok fiber is also an agricultural product produced by the plant species *ceiba pentandra*. It has high oil absorbency characteristic and hydrophobicity because of the waxy cutin on its fiber surface. The earliest published work on its use for oil absorption was perhaps carried out by Kobayashi et al. [11], followed by Choi and co-workers [12–14]. Besides excellent oil absorbency, kapok has been found able to remove more than 80% of oil mass present in a simulated runoff containing engine oil [15]. A recent study reported that when used as a deep bed filter, it could remove more than 99% of low- to high-viscosity oils from oily water [16].

The rate of NAPL flow through the oil/water separation unit depends on physical characteristics of the filter medium (e.g., pore diameter, pore geometry and filter thickness), characteristics of the immiscible fluids (e.g., surface tension and viscosity) and operating conditions (e.g., trans-filter pressure). The design considerations of the proposed module, besides diameter and length, are thickness and packing density of the kapok medium. This study examined the influences of physical characteristics of the kapok packing and oil thickness on performance of the kapok in in situ oil/water separation as schematized in Fig. 1. The study was primarily pursued through a laboratory modeling with a two-dimensional flume, in which the kapok was packed into a vertical wall that served as oil/water separation unit. Specifically, oil permeation rate and water repellency by the kapok wall were assessed.

2. Materials and methods

2.1. Kapok

The kapok used in this study was a product of Thailand. As shown by its SEM image (Fig. 2), the fiber had a hollow tubular structure (or lumen) with average external diameter of $16.5 \pm 2.4\ \mu m$, internal diameter of $14.5 \pm 2.4\ \mu m$ and length of 25 ± 5 mm. This indicated that 77% of the fiber volume was contributed by its lumen. The specific gravity of the kapok fiber material was $1.31\ g/cm^3$, as determined with Ultrapycnometer 1000. The raw kapok fiber did not need

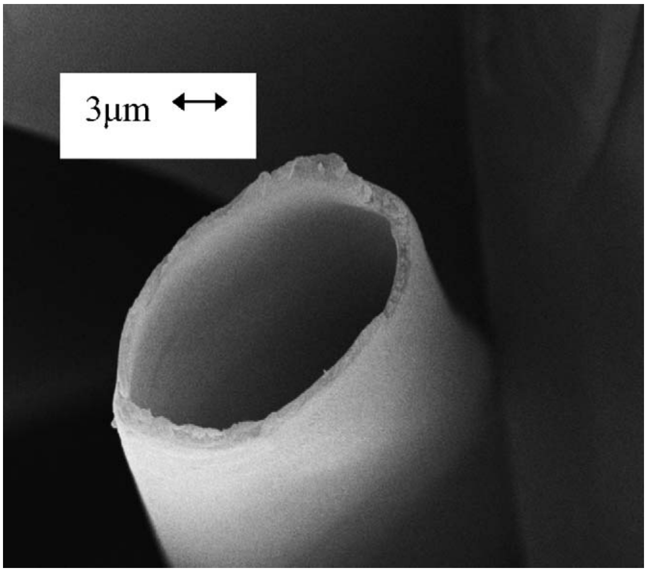

Fig. 2. SEM image of the cross-section of a kapok fiber.

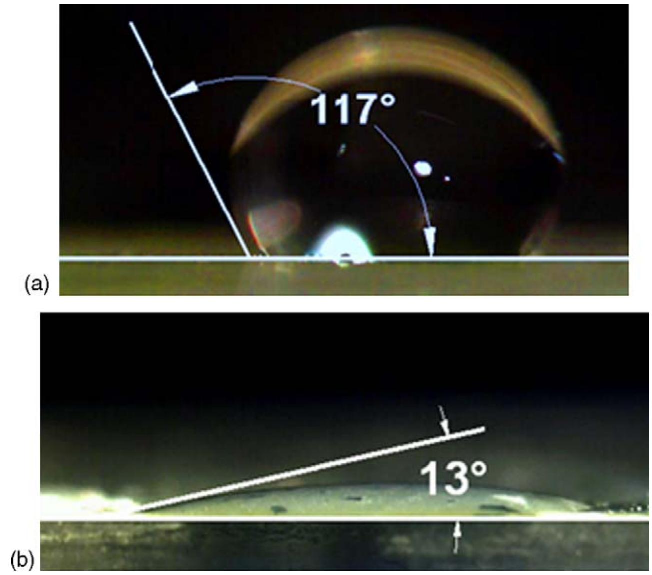

Fig. 3. Contact angles between kapok surface extract and experimental liquids: (a) water and (b) diesel.

pretreatment as it was an excellent oil sorbent in its native state.

For measurement of contact angles between the kapok fiber and the experimental liquids (diesel and water), the chemical extract of the kapok fiber surface was obtained with chloroform extraction for 15 min. The extract was then evenly deposited on a microscope glass slide, after evaporation of the chloroform. Drops of experimental liquids were then applied on the glass slide. Adhesions of the liquid droplets on the slide were observed through a microscope equipped with a CCD camera. Fig. 3a and b show a water droplet and a diesel droplet, respectively, on the glass slide. The contact angles of >90° for the water droplet and 13° for the diesel droplet attest to the hydrophobic–oleophilic surface characteristics of the kapok fiber.

2.2. Diesel

Diesel used was purchased from a gasoline station in Singapore. The diesel represents low-viscosity oils such as light crude oil, kerosene and gasoline. The diesel was investigated in favor of gasoline or other light-weight hydrocarbon oils because of its relatively non-volatility, which would minimize transient change in its chemical and physical characteristics during experiment. Density of the diesel was measured using gravimetric method. Its viscosity was measured using a Viscolab Viscometer (VL-4100) of Cambridge Applied Systems Inc. A dynamic contact angle measuring instrument and tensiometer (DCAT 11) from DataPhysics were used to measure surface tensions of the experimental liquids. Table 1 summarizes the properties of the diesel as compared to those of water.

2.3. Investigation of permeability of kapok packing

Permeability is one of the important parameters for assessing ability of a fibrous medium in oil/water filtration and separation. The experiment was carried out using a constant-head permeameter, according to recommendation by American Petroleum Institute [17]. The column, packed evenly with the kapok, was 204 mm in height which was sufficiently long to close up any short-circuit flow path through it. The permeabilities of the kapok columns with various packing densities were assessed. Darcy's Law expresses intrinsic permeability, k (in $[L^2]$), as follows:

$$k = \frac{\mu Q L}{\rho g h A} \quad (1)$$

where μ is the viscosity of the fluid, Q the flow rate, L the flow path length, ρ the density of the fluid, g the acceleration of gravity, h the differential pressure head and A is the average cross-sectional area perpendicular to the flow direction.

2.4. Experimental setup

A two-dimensional simulation of the diesel recovery from groundwater was conducted in a 2000 mm (L) × 130 mm (W) × 500 mm (H) flume, as shown in Fig. 4. A model recov-

Table 1
Properties of liquids used in experiments and their interactions with kapok

Liquids	Density at 21 °C (g/cm^3)	Viscosity at 21 °C (cp)	Surface tension (against air) at 21 °C (MN/m)	Contact angle with kapok (degree)
Water	1.00 ± 0.01	1.0 ± 0.1	72.00 ± 0.01	117
Diesel	0.83 ± 0.01	5.0 ± 0.1	26.29 ± 0.01	13

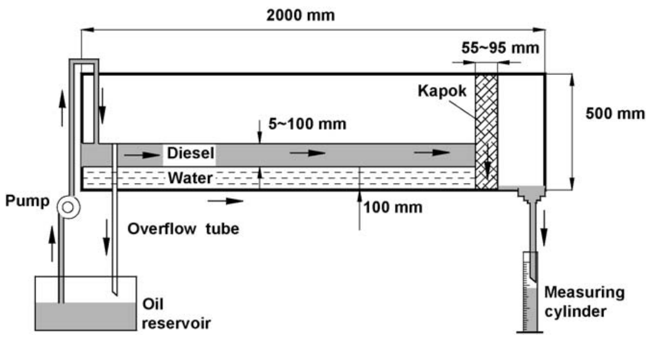

Fig. 4. Experimental setup for diesel permeation through kapok wall into model recovery well.

ery well was placed at one end of the flume. An oil inlet and an overflow tube were installed at the other end of the flume to control the oil level.

The flume was not filled with granular material (or its porosity equaled to unity). This allowed complete free flow of the feed stream, and unambiguous interpretation of the observed diesel flow through the kapok wall. The model well comprised a pair of acrylic plates, each with 500 mm (*H*) × 200 mm (*L*) × 10 mm (*T*) dimension, to be placed against the flume wall. There were several sliding guides (20 mm spaced *c*/*c*) pre-carved onto the inner face of the acrylic plates to guide the position of two steel perforated plates that served as support for the kapok wall. By placing the steel perforated plates into different sliding guides, different thicknesses of the kapok wall could be constructed. The kapok would be packed between the two steel plates, to form the full-height kapok wall across the flume. The packing was done lift by lift, with each lift compacted such that it produced good uniformity in packing density throughout the full height of the kapok wall. Gaps found between the acrylic plates and the flume wall was sealed with silicon gel while the interface between the kapok wall and the interior surface of the acrylic plates was sealed with molten wax, both were to prevent leaking of experimental liquids due to short circuiting through interfaces.

2.5. Operation procedure

The experimental variables were thickness of the kapok wall, kapok packing density and thickness of the diesel layer in the feed stream. All experiments were carried out with a constant water height of 100 mm but varying diesel thicknesses of 5, 10, 20, 40, 60, 80 and 100 mm. The kapok wall thicknesses investigated were 35, 55, 75 and 95 mm. In a preliminary study, the kapok walls of different kapok packing densities ranging from 40 to 70 g/L were tested for their water proofing capability and resistance to deformation under the natural hydraulic pressure. Deformation of the kapok wall was undesirable as it could produce preferential flow path, leading to leakage. It was found that the kapok walls with at least a packing density of 70 g/L must be used to ensure good water proofing and resistance to deformation. Therefore, this packing density was used subsequently in all the simulation experiments.

The simulation began with filling of the flume with tap water to 200 mm level to verify water proofing of the kapok wall. The 200 mm water head was tested because it exceeded the maximum pressure head that would be exerted by the feed stream in actual experiment. After full water proofing of the wall was confirmed, the water level was lowered to 100 mm, and the diesel (dyed orange) was then pumped into the flume. The diesel level was maintained by the adjustable overflow tube, as shown in Fig. 4. The diesel permeated through the kapok wall into the model recovery well was collected in a measuring cylinder and measured at 10 min interval until steady permeation rate was achieved. The permeants were analyzed for the diesel and water contents. After that, the oil overflow tube was manually lifted to a new elevation to increase the diesel thickness, and new observation was made. After one complete series of experiment with various diesel thicknesses, both the diesel and water were pumped out from the flume and the kapok wall was left in place for 12 h (overnight drying stage) before it was analyzed for its liquid retention along its full height. For this analysis, the kapok wall was divided into 8–12 sections along its wetted height, and the liquid absorbed in each segment was extracted by 1400 × *g* centrifugation for 30 min. The extracted liquid was allowed to completely separate in a measuring cylinder where the water volume was determined. The mass of the diesel extracted with centrifugation was corrected with its amount remained in the kapok lumen as determined using gravimetric method.

To investigate the durability of the kapok wall for reuse, the experiment with the 55-mm thick kapok wall and 100-mm thick diesel layer was repeated for a total of four drying/wetting cycles. For each cycle of experiment, the steady diesel permeation rate was recorded.

3. Results and discussion

3.1. Permeability

Fig. 5 shows that the intrinsic permeabilities of the kapok columns decreased drastically with increasing packing density for the diesel flow. Also indicated in the figure are the corresponding porosities of the kapok columns over the range of the packing densities investigated. The permeability decreased from 0.0165 cm^2 at 34 g/L packing density to 0.0038 cm^2 at 70 g/L packing density, almost linearly.

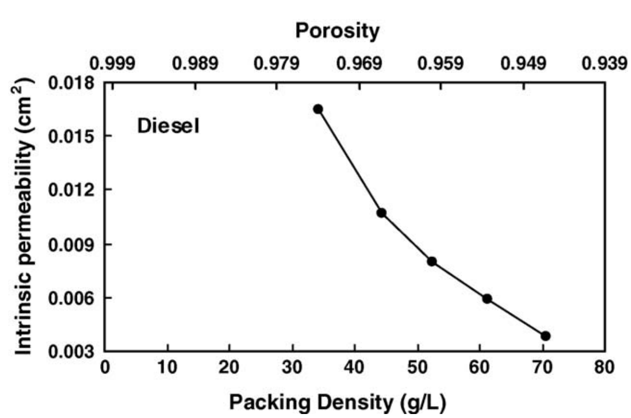

Fig. 5. Diesel permeability of packed kapok as a function of packing density.

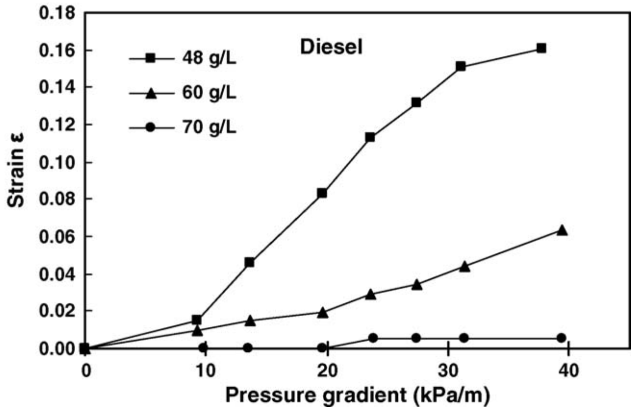

Fig. 6. Deformation of kapok columns with various packing densities under different applied pressures.

Although the results suggested that it would be better to use a low packing density for achieving maximum diesel permeation rate, significant deformation of the loosely packed kapok columns was observed, as depicted in Fig. 6, under the applied hydraulic gradient of up to 40 kPa/m during the permeability test. The deformation was undesirable as it resulted in uneven flow path (pattern) through the kapok columns. When packed at a high packing density, e.g., 70 g/L, the kapok columns only deformed marginally (<0.5%) under 40 kPa/m of applied pressure.

3.2. Performance of kapok wall in selective oil/water filtration

When the diesel was pumped into the flume, it penetrated the kapok wall, and the wetting front migrated from the feed stream towards the recovery well slowly. It took around 30 min for the diesel to break through the 55-mm thick kapok wall. The permeant apparently did not contain water. Within the kapok wall, vertical dispersion of the diesel wetted zone due to the capillary action was observed.

Fig. 7 shows the permeation rates of diesel through the kapok wall with thicknesses of 55, 75 and 95 mm. Result of experiment

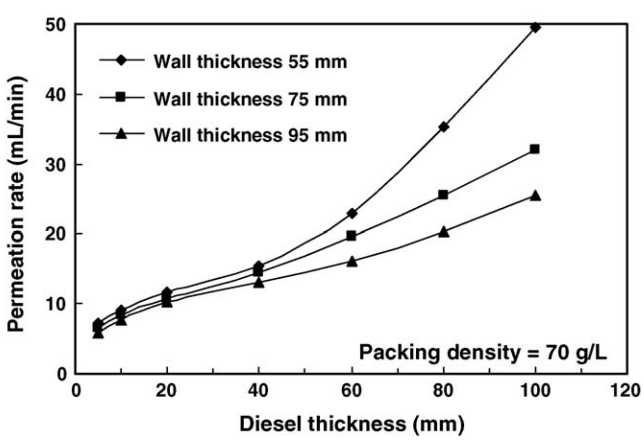

Fig. 7. Rate of diesel permeation through kapok walls of various thicknesses as a function of diesel thickness in feed stream.

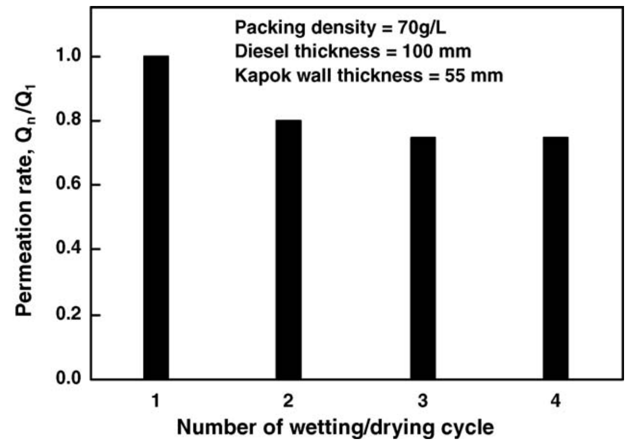

Fig. 8. Changes in diesel permeation rate through the 55-mm thick kapok wall as a function of number of wetting/drying cycle.

on the 35-mm thick kapok wall is not shown as the wall failed in water proofing test. In general, the permeation rates increased almost linearly with the thickness of the diesel layer, or increased with decreasing thickness of the kapok wall. By normalizing the permeation rate with the cross-sectional area of the wetted zone in contact with the feed stream, it can be derived that at diesel thicknesses of greater than 60 mm, constant diesel fluxes of 3.8–5.0, 3.2–3.3 and 2.5–2.7 L/(m^2 min) could be achieved with the kapok walls of 55-, 75- and 95-mm thick, respectively, under the natural pressure gradients. The thinner wall allowed higher permeation rate because of existence of greater hydraulic gradient, which agreed with Darcy's Law. Therefore, the optimum wall thickness should be such that it is as thin as possible to allow maximum oil permeation, but remains thick enough to stop infiltrating water. In this study, the optimum wall thickness was 55 mm.

The performance of the kapok wall in separating the oil/water flow for several cycles of wetting/drying is illustrated in Fig. 8. Apparently, the kapok wall could maintain a substantial portion of its initial oil recovery rate after several cycles of reuse. The permeation rates in the 3rd and 4th cycles were 73% of that achieved in the 1st cycle. A prolonged experiment showed that the diesel permeation rate remained unchanged at the end of 1 week (results not shown). It is worth noting that the decrease in the permeation rate in the 2nd cycle was partly attributed to contractions of some flow channels by action of liquid bridges [18] formed between fibers after wetting. Overall, the results show a high reusability of the kapok oil/water separation wall.

3.3. Liquid distribution in kapok wall

Fig. 9 shows the amount of oil and water retained (expressed in g liquid/g kapok) in the kapok wall (70 g/L packing) along its height. Also indicated are the water level (100 mm) and the ultimate diesel level (200 mm) in the feed stream. In general, the kapok wall retained far less water than diesel. It is noted that the water retained above 100-mm height corresponded to the water that penetrated into the kapok wall during water proofing stage (in which water level was raised to 200 mm). Above 200 mm,

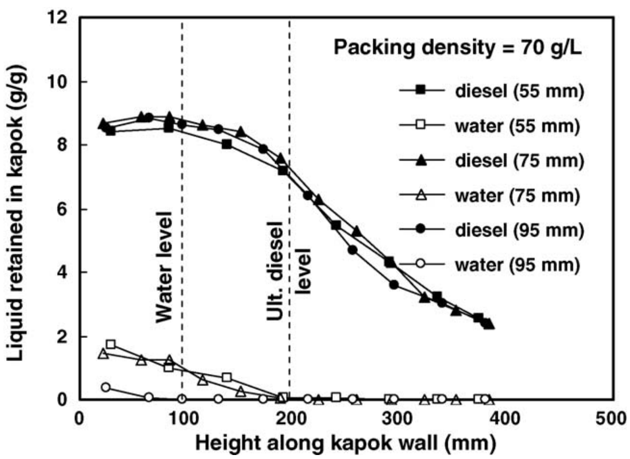

Fig. 9. Diesel and water retentions at various heights of kapok walls of 55-, 75- and 95-mm thicknesses.

no water was found retained in the kapok wall, indicating no capillary migration (rise) of water along the wall. For the kapok walls of 55- and 75-mm thick, the water retained was found to increase linearly with the hydraulic pressure. For example, at 30-mm height (or liquid head of 170 mm) of the 55-mm kapok wall, the water retention was 1.75 g/g. This decreased to 1.0 g/g at 85-mm height (or liquid head of 115 mm), and to a negligible amount at 200-mm height. Because water had never emerged through the wall, with thicker wall (95 mm), lower water retention in the kapok wall was found because a substantial portion of the wall was not wetted by water.

In contrast to water, the diesel could migrate up along the kapok wall to above 200-mm high due to capillary action. It also migrated down into the water regime (height < 100 mm) through combined actions of gravity and capillary. If considering the porosity of the kapok wall of 70 g/L packing, which was 0.95, the wall was only partially saturated with the diesel. It is therefore postulated that more diesel would penetrate through the wall if pressure was applied at the feed stream or vacuum was applied at the model recovery well.

3.4. Discussion

Water repellence and oil permeability were the two important characteristics of the proposed in situ oil/water separation system for selective liquid filtration of the oil-contaminated groundwater. The highly selective filtration of oil/water demonstrated by the hydrophobic–oleophilic kapok wall can be explained with interfacial phenomena that govern the interaction of oil/water and the kapok fiber.

In an assumed circular tube of a filter medium containing a liquid phase and a gas phase (air), the shape of the curvature formed at the interface between the two fluids (gas and liquid) determines the direction at which the interfacial force acts, which in turn determines absorption or repellence of the liquid by the filter medium. One main factor that determines the shape of the curvature is the difference in adhesive tendency of the two fluids with the solid surface they come into contact. The curvature is shaped such that the fluid with stronger affinity for the solid surface is on the concave side of the interface while the fluid with weaker affinity for the solid surface is on the convex side. The resultant interfacial force (or capillary force) acts to draw the fluid on the concave side of the interface to displace the fluid on the convex side. For the case of the hydrophobic–oleophilic kapok wall whose pore space was initially filled with air, when exposed to oil, the oil was on the concave side at oil–air interface. It is because the contact angle between the oil and the kapok surface material is small, as evidenced by Fig. 3b. The oil is therefore termed as the wetting liquid for the oleophilic kapok, and it would penetrate spontaneously into the kapok wall under the action of positive capillary entry pressure [19]. In the contrary, when the kapok was exposed to water, water would be on the convex side at the water–air interface, as evidenced by the contact angle of greater than 90° (as shown in Fig. 3a). As a result of water being a non-wetting fluid for the hydrophobic kapok, negative capillary entry pressure would exert on water, preventing it from penetrating into the kapok wall.

Capillary entry pressure (p_c) is related to gravity, degree of saturation, pore-size, pore-shape and interfacial forces [20]. The capillary entry pressure into a circular channel can be predicted using the well-known Laplace–Young equation:

$$p_c = \left(\frac{2\sigma}{r}\right)\cos\theta \qquad (2)$$

where σ is the interfacial tension, θ the contact angle formed between the air–liquid interface and the solid surface at the point of contact, and r is the radius of the flow channel. The contact angle depends on temperature and physico-chemical properties of the two fluids and the solid surface [20]. Under static conditions, the contact angle formed between the air–liquid interface and the solid boundary is dependent on the three interfacial forces acting at air–liquid, air–solid and liquid–solid interfaces. Eq. (2) also suggests that the capillary entry pressure at the air–liquid interface is inversely proportional to the size of the capillary tube. This implies that when packing density of the filter medium increases, the penetration rate of wetting liquid will increase.

The term spontaneous imbibition has been also used to describe the process by which a wetting fluid is drawn into a porous medium by action of the capillary force [21]. Absorption of oil by the kapok would occur spontaneously and the spontaneous imbibition would continue as long as the capillary entry pressure was greater than the pressure that acted against the oil motion (such as gravity). For water, entry of water into the kapok wall would begin only when the external pressure (such as hydraulic pressure) overcame the negative capillary entry pressure exerted by the wall.

4. Conclusions

The kapok is a natural hydrophobic–oleophilic fiber that can separate oil from oil/water immiscible flow. Its innovative use in oil recovery well for recovering oil without water is proposed. The kapok wall constructed with packing density of 70 g/L and thicknesses of 55 mm or greater exhibited complete rejection of water while allowed adequate flux of the diesel oil into the model oil recovery well. When oil thickness exceeded 60 mm, a constant flux of 3.8–5.0, 3.2–3.3 and 2.5–2.7 L/(m^2 min) could be achieved by the kapok wall of 55-, 75- and 95-mm thick, respectively, under the natural pressure gradient. The ability to selectively filter the oil/water immiscible flow is crucial for practical application as it reduces the volume of liquid to be handled on site. The good reusability of the kapok wall promises its potential application for in situ NAPL removal from groundwater.

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