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## Separation Science and Technology

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713708471>

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**To cite this Article** Varghese, Bose K. and Cleveland, Theodore G.(1998) 'Kenaf as a Deep-Bed Filter Medium to Remove Oil from Oil-in-Water Emulsions', *Separation Science and Technology*, 33: 14, 2197 – 2220

**To link to this Article:** DOI: 10.1080/01496399808545723

URL: <http://dx.doi.org/10.1080/01496399808545723>

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## **Kenaf as a Deep-Bed Filter Medium to Remove Oil from Oil-in-Water Emulsions**

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### **ABSTRACT**

This study investigated the feasibility of deep-bed filtration using kenaf (agricultural fiber) media for the removal of oil from oil-in-water emulsions. Continuous flow, constant pressure filtrations were conducted using surfactant stabilized emulsions. Removal of oil and grease varied from 70 to 95% for 500 mg/L oil-in-water emulsion stabilized by surfactants. Oil removal was better for larger oil drops, finer media particles, higher filtration pressure, lower pH, cationic surfactant, and deeper media. Moisture contents and heating values of the spent media were determined. Moisture content decreased with increasing filtration pressure and decreasing particle size of the media. Heating values of the spent media increased with the volume of emulsion filtered. Heating values were high enough to produce surplus energy after accounting for the energy required for driving out the moisture. The results indicated that it may be possible to dispose of the spent medium by combustion without further drying and extract net energy in the process.

### **INTRODUCTION**

Organic contamination of water is a universal problem. Often the contaminants are dissolved in water, but there are also situations when the organics are in the dispersed form and exist as small droplets varying in size from submicrons to hundreds of microns. These low concentration organic dispersions in water, generally referred to as oil-in-water emulsions, are formed

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when there is an immiscible organic phase in contact with water and there is some form of mixing energy involved.

Unwanted emulsions are formed in many industrial operations where they create problems of separation and/or disposal. Produced water, surfactant-enhanced soil wash fluid, aircraft and automobile wash, tanks and drums wash, cooling and lubricating emulsions, industrial laundry, wastewater from ship engine rooms, and bilge water from tankers contain considerable amounts of emulsified oil and grease. Oil concentration in these industrial wastes could vary from a few to thousands of parts per million. These wastes generally form part of the wastewater and require the removal of the dispersed phase for either discharge or reuse.

Oil drilling and production operations produce huge quantities of water that has some emulsified crude oil stabilized by naturally occurring agents like organophilic clay particles or chemicals added to the injection water (1). Although the concentration of oil in the produced water discharged is regulated, an increase in the number of wells and increasing concern about the environment have encouraged stricter discharge limits for produced water. The USEPA has promulgated a zero discharge regulation for coastal wells effective as of January 15, 1997. In locations where discharge is allowed, discharge limits of 29 mg/L monthly average and 42 mg/L daily maximum exist for oil and grease (2). These limits essentially require either treating the produced water to remove oil or reinjecting the produced water. Rejection further requires the removal of solid and oil particles to a level that will allow continuous injection without formation damage (3).

Surfactant-enhanced soil washing to remove residually trapped NAPL (nonaqueous phase liquid) from soil produces a combination of microemulsions (drop size  $< 1 \mu\text{m}$ ) and macroemulsions (drop size  $> 1 \mu\text{m}$ ) stabilized by the surfactants (4). A major hurdle in the implementation of this process is the removal of the organic phase from the solution so that the surfactant solution can be recycled (5).

This research evaluates the effectiveness of kenaf (see Materials Section) as a deep-bed filter medium in removing oil from surfactant-stabilized emulsions and the possibility of disposing the spent medium by combustion.

## BACKGROUND

### **Stabilized Emulsions and Oil Removal**

Stability of an emulsion is a measure of its resistance to coalescence. Although oil–water interfaces are inherently negatively charged (6), oil droplets coalesce in the absence of a stabilizing agent due to the thermodynamic instability arising from the finely dispersed state (7). Chemicals like polymers and surfactants, and particles like clay, when adsorbed on the surface of the drop-

lets give all the droplets the same charge, preventing coalesce. Sometimes too much of a substance is adsorbed, making it physically impossible for two particles to coalesce. The first process is called electrostatic stabilization and the second process is called steric stabilization. Regardless of the mechanism, the practical implication is that stabilized emulsions are hard to break.

Various methods used for the removal of dispersed oil include gravity separation, centrifugation, hydrocyclones, air floatation, membrane separation, demulsification (chemical, heat, and electric), coalescence filtration, biological treatment, and deep-bed filtration.

### **Deep-Bed Filtration: Principles and Applications**

Deep-bed filtration is a common technique used in drinking water/waste-water treatment to remove very fine particles at a very low loading. In deep-bed filtration, particle removal occurs within the body of the filter medium as opposed to filtration on the surface. Very fine particles are caught in much bigger pores in the medium through the mechanisms of impaction, interception, and diffusion.

Small liquid droplets in dispersion behave like solid particles. Large drops can behave differently because of the possibility of drop deformation. A filtration study by Mackie and Bai (8) showed that there is a critical particle size of about 1  $\mu\text{m}$  for which the removal efficiency is a minimum. For particles larger than 1  $\mu\text{m}$  and up to about 100  $\mu\text{m}$ , interception and gravity sedimentation predominates. For particles larger than 100  $\mu\text{m}$ , removal takes place by physical entrapment (9).

Viraraghavan and Mathavan (10) report 34 to 99% removal of oil from different oil-in-water emulsions by deep-bed filtration using horticultural peat as the filter medium. The first cellulose-based deep-bed filter to remove dispersed oil was designed by Wemco, California. The filter uses pecan/walnut shell as the filter medium, and it removes particles of 2  $\mu\text{m}$  and above at 98% efficiency (11).

### **Disposal of Spent Media**

In traditional multimedia filtration, the spent bed is backwashed to remove the retained particles and the same media is kept in service for a long time. A cellulose-based medium has to be replaced before microbial degradation of the medium sets in. Although the disposal of spent medium appears to be a problem, it could be straightforward and even profitable. All cellulose materials have an inherent heating value, and cellulose-based waste materials like sugarcane bagasse have proven to be economic sources of energy (12). The oil caught up in the bed during filtration enhances the heating value of the spent bed. The spent bed can be disposed of by burning, and the net energy

that can be produced depends on the level of oil saturation and the heating value of the oil.

## MATERIALS AND METHODS

### Filtration Setup

A filtration unit in which the kenaf is held tightly as a bed was designed and fabricated for the study (see Fig. 1). The unit consists of a 5-cm long Plexiglas pipe of ID 4.6 cm with a wire mesh spacer and a perforated disk at both ends. In this downflow arrangement the top wire mesh and perforated disk distribute the flow uniformly over the medium, and the bottom set supports the medium and prevents the loss of grains with the filtrate. A positive displacement pump supplies the emulsion to the unit, and filtrate is collected from the outlet at the bottom. A constant pressure of 45 or 60 psi is maintained

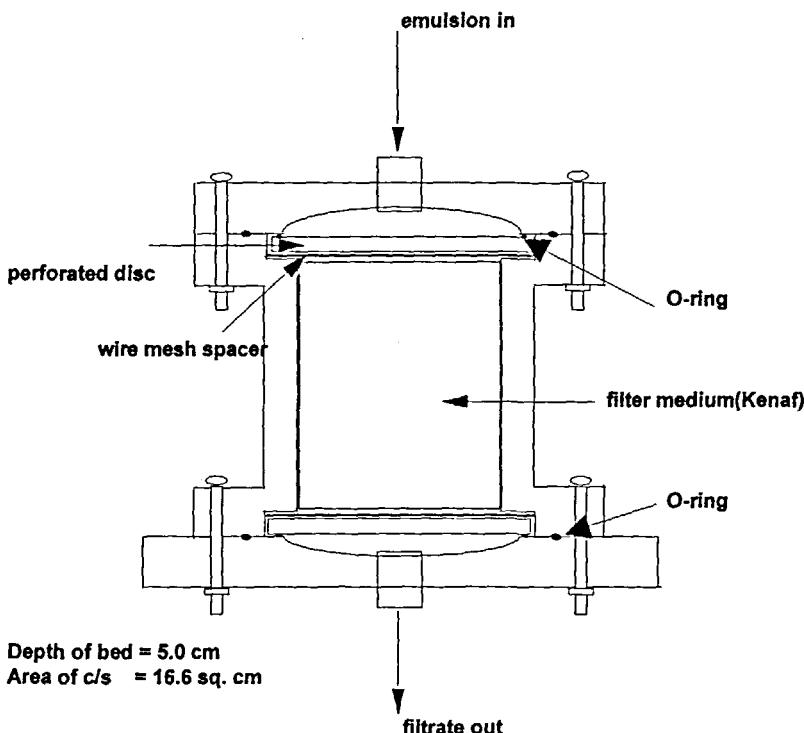


FIG. 1 The filtration unit.

TABLE 1  
Properties and Amounts of Different Surfactants Used

Surfactant	CMC (g/L)	Purity (%)	Quantity used <sup>a</sup> (g)
Triton X100	0.14	100	0.66
SDS	2.38	99	9.8
CPC	0.306	95.2	1.39

<sup>a</sup> CMC + 5 wt% of the oil used, calculated for 4-L samples.

with the help of a valve that controls the flow and a pressure gauge that reads the pressure in the feed line.

### Production and Characterization of Oil-in-Water Emulsion

#### Materials

**Oil.** Light paraffin oil with a density of 0.845 to 0.86 g/cm<sup>3</sup> and a Saybolt viscosity of 80–90 at 25°C was used for the preparation of emulsions. The paraffin oil used in the study had a 3.1 wt% loss of volatiles at 85°C.

**Surfactants.** Triton X100, a nonionic surfactant, sodium dodecyl sulfate (SDS), an anionic surfactant, and cetyl pyridinium chloride (CPC), a cationic surfactant, were used in the study. Triton X100 was used for the experiments to determine the relationship of removal with other parameters. SDS and CPC were used to determine qualitatively the effect of the nature of the surfactant charge on oil removal.

#### Preparation of Emulsion

The emulsions were prepared in deionized (DI) water in 4-L batches. The oil and the surfactant were weighed in an aluminum cup and put in a large beaker with 4 L of DI water. The contents were stirred vigorously using a magnetic stirrer (Fisher Scientific) with simultaneous application of sonic disruption at a power output of 50 W (Tekmar, 250 W input power), and the sample was then transferred into a 10-gal bucket. Table 1 gives the critical micelle concentration (CMC) values, the purity, and the actual amounts of different surfactants used.

To determine the effect of drop size on removal, two drop size distributions as distinct as possible were prepared. After 8 minutes of mixing, the sample essentially had a single peak with a  $D_{50}$  of 3  $\mu\text{m}$  and a range of 0.5–100  $\mu\text{m}$ , and was selected for the smaller sized drops. For the larger drop size, the sample after 2 minutes of mixing that had a bimodal distribution with a

significant peak around 35  $\mu\text{m}$ , a  $D_{50}$  of 14  $\mu\text{m}$ , and a range of 0.5–150  $\mu\text{m}$  was selected.

The stabilities of the emulsions was measured by measuring the change in particle size distribution over time. A sample that had the same particle size distribution over a period of 1 day was said to be stable over a period of 1 day. All the samples used in the study were stable for at least 1 day. The criterion of no floating oil was used in the preparation of all emulsion samples.

### Filter Medium

Kenaf (*Hibiscus cannabinus*), an annual plant cultivated in Mississippi, Texas, Arizona, and Louisiana, and widely cultivated in Africa, was used in this study. Kenaf stalk has a bast (bark) of long fibers and a core. Kenaf core has commercial applications as a potting mix, absorbent, drilling mud additive, and raw material for paper. The bast has different applications, principally as filler for plastics in the automotive industry.

Kenaf is preferentially wetted by organic solvents like acetone and alcohol, and by oils. Kenaf can absorb and hold four to eight times its weight of oil, depending on the kenaf fiber size and the nature of the oil. Kenaf, being a natural fiber (mostly cellulose), is biodegradable. Its excellent capacity to absorb and retain organics along with the presence of native bacteria makes it a good soil remediation agent. Borazjani and Diehl (13) demonstrated the use of kenaf-enhanced bioremediation of oil-contaminated soils. Kenaf is combustible with a calorific value of about 7000 Btu/lb and an ash content of about 1.6%.

Whole kenaf (Kenaf International, Louisiana) was further ground to smaller particles using a laboratory mill (hammer/cutting) from Glen Mills, Inc. The different size ranges used in the experiments were particles that exit the mill through a 200- $\mu\text{m}$  sieve (Kenaf 200), a 500- $\mu\text{m}$  sieve (Kenaf 500), and a 1000- $\mu\text{m}$  sieve (Kenaf 1000).

### Characterization of Ground Kenaf

Kenaf media were characterized by measuring the particle size distribution, bulk density, porosity, and oil absorption capacity. Table 2 lists the properties of different kenaf samples used in the study as measured in the laboratory. The oil absorption capacity of different kenaf media depends on the porosity. The oil gets caught in the bulk pores, resulting in a better absorption capacity for a larger medium. The values shown are for kenaf at an ambient moisture content of 8%.

### Filtration Experimental Procedure

The filter medium was placed in the filtration unit manually, and filtration was started by starting the positive displacement pump. The filtration pressure

TABLE 2  
Properties of Kenaf Samples Used in the Study

Sample	Bulk density (g/cm <sup>3</sup> )	Porosity	Oil absorption, <sup>a</sup> (g/g)
Kenaf 200	0.133	0.9	3.93
Kenaf 500	0.125	0.906	4.9
Kenaf 1000	0.1	0.92	5.29

<sup>a</sup> For paraffin oil at room temperature, g oil/g kenaf.

was slowly increased and held at 45 or 60 psi using the discharge valve to control the flow. Filtrate was collected in clean beakers in fixed volumes, and one sample from each was analyzed for oil and grease. The time required to filter each fixed volume batch of filtrate was noted.

### ***Analytical Procedures***

The oil in the feed and filtrate were measured using Method 1664 of USEPA (Hexane Extractable Materials—HEM). The method, initially designed for a 1000-mL sample (14), was modified for a 100-mL sample.

The calorific values of the pure kenaf and spent media was measured using a Parr Oxygen Bomb Calorimeter. The calorimeter was calibrated using benzoic acid pellets.

The moisture contents of the spent media and percent volatiles in the paraffin oil were measured at 85°C using a Sartorius moisture analyzer.

All particle size analyses were done using a Malvern Mastersizer with a size range of 0.5 to 900 µm. Microscopic analysis was used as a confirmatory test.

## **RESULTS AND DISCUSSION**

A preliminary filter run with DI water showed that the initial filtrate was highly colored, but most of the color was leached out with the first 200 mL of the filtrate. To avoid the interference of color bodies in the oil analysis, the first 200 mL of the filtrate was discarded in all the filtration experiments. The first 200 mL of the filtrate is expected to have a higher oil concentration because the medium will be loose. In industrial filtration, it is a common practice to recycle the initial filtrate to meet the filtrate quality. Discarding the initial filtrate is an approximation of this practice that also improves the resolution of the oil analysis method used in this research.

TABLE 3  
Depths and Bed Volumes of the Compressed Media

Medium	45 psi		60 psi	
	Bed depth (cm)	Bed volume (cm <sup>3</sup> )	Bed depth (cm)	Bed volume (cm <sup>3</sup> )
Kenaf 200	3.4	59	3.3	57.3
Kenaf 500	4	68.4	3.9	67.7
Kenaf 1000	4.3	74.6	4.2	72.9

The kenaf media are highly compressible. Table 3 gives the depths of different media at 45 and 60 psi for the initial depth of 5 cm. The bed volumes were calculated based on the depth of the compressed media. These bed volume values were used in calculating the number of bed volumes filtered for all the experiments. The 7.5-cm deep bed used in one of the experiments had a bed volume of 105.8 cm<sup>3</sup> at a filtration pressure of 60 psi.

### Oil Removal

#### **Effect of Oil Concentration on Removal**

The emulsions with 500 mg/L oil (165 mg/L Triton) and 5000 mg/L oil (250 mg/L Triton) were filtered through a kenaf 500 bed at 60 psi pressure. The plot in Fig. 2 shows that removal was better for the 5000-mg/L emulsion. In Fig. 2 a 50-mg/L oil in the filtrate corresponds to 99 and 90% removals for the 5000 and 500-mg/L emulsions, respectively. Because the 5000-mg/L emulsion has a lower relative surfactant concentration and possibly larger droplets, we observed improved percent removal as compared to the lower concentration emulsion. This comparative study was done to demonstrate that the kenaf medium can remove oil from emulsions of very high oil concentration, but cannot achieve higher bed volumes.

#### **Effect of Filtration Pressure on Removal**

For an incompressible medium, the effect of pressure will be reflected by the change in the flow rate. For a compressible medium like kenaf, increasing pressure results in bed compaction and decreased permeability. Consequently, an increase in pressure may not increase the flow rate proportionally.

Figures 3 and 4 show the result of filtering the 2-minute sample through kenaf 500 and kenaf 200 beds, respectively. Removal at 60 psi is better than that at 45 psi in both cases. In these cases the increase in the flow rate due to the increase in pressure was offset by the increased compaction of the

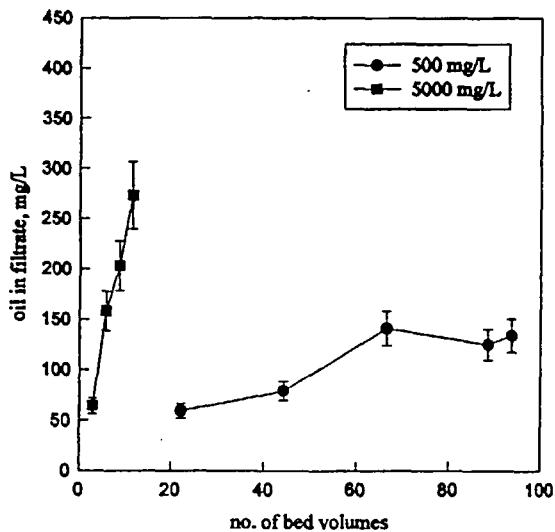


FIG. 2 Effect of oil concentration in the emulsion on oil removal for kenaf 500 medium at 60 psi. Flow rates: 500 mg/L emulsion, 34.7 mL/min; and 5000 mg/L emulsion, 63.8 mL/min.

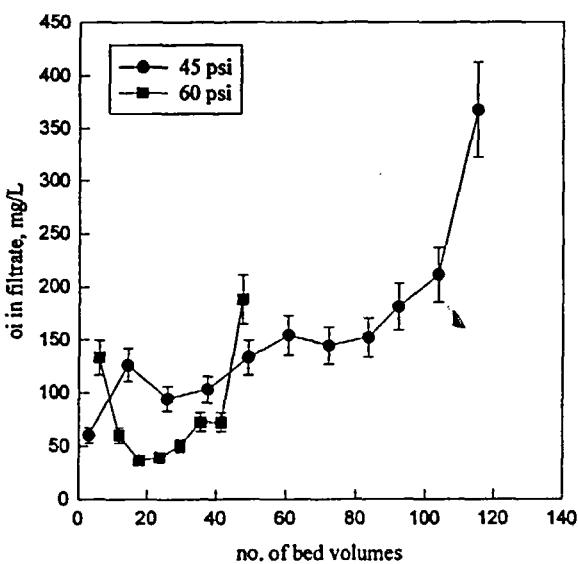


FIG. 3 Effect of pressure on oil removal for kenaf 500 bed and a 2-minute sample of 500 mg/L emulsion. Flow rates: 45 psi, 36.1 mL/min; and 60 psi, 44.3 mL/min.

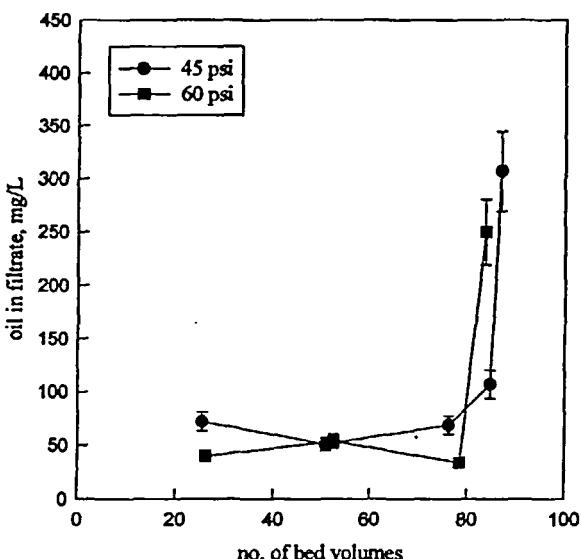


FIG. 4 Effect of pressure on oil removal for kenaf 200 bed and a 2-minute sample of 500 mg/L emulsion. Flow rates: 45 psi, 54.6 mL/min; and 60 psi, 29.4 mL/min.

medium. The compacted medium had smaller pores and smaller pore volume. Smaller pores retained the droplets better than bigger pores.

#### ***Effect of Drop Size on Removal***

The effect of drop size on oil removal was studied by filtering 500-mg/L oil emulsions with two different drop size distributions. The 2-minute emulsion had larger drops than the 8-minute emulsion. Filtrations were carried out for a kenaf 500 bed at pressures of 45 and 60 psi. Figures 5 and 6 show that removals were not much different between the two samples at both pressures, although Fig. 5 shows slightly better removal for the sample with a larger volume fraction of larger droplets.

Figures 7 and 8 show the filtrate drop size distributions for the two different emulsions. Figures 9 and 10 show the emulsion drop sizes presented to the filter. Droplets larger than 10  $\mu\text{m}$  are removed. Most of the remaining droplets are in the range of 0.2 to 5  $\mu\text{m}$ , with the mean particle size near 2  $\mu\text{m}$ . Therefore, the sample with a larger volume fraction (and larger mass fraction) of drops should have better removal. The lower bound on droplet size removal observed is consistent with the theory of deep-bed filtration that predicts the least removal for droplets of the 1–2  $\mu\text{m}$  size range.

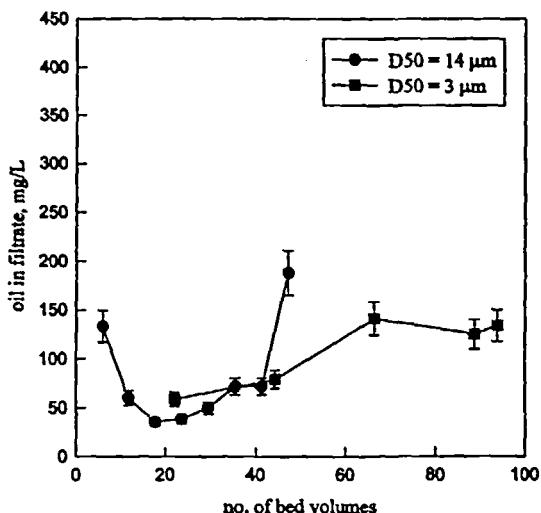


FIG. 5 Effect of drop size on oil removal for the kenaf 500 bed at 60 psi with the 2-minute ( $D_{50} = 14 \mu\text{m}$ ) and the 8-minute ( $D_{50} = 3 \mu\text{m}$ ) samples of the 500-mg/L emulsion. Flow rates: 2 minutes, 44.3 mL/min; and 8 minutes, 34.7 mL/min.

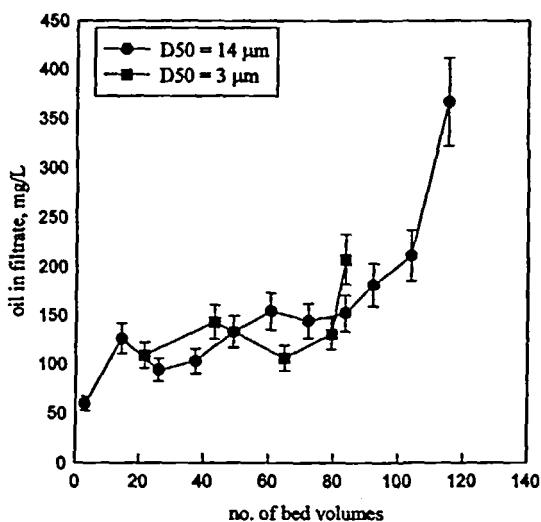


FIG. 6 Effect of drop size on oil removal for the kenaf 500 bed at 45 psi with the 2-minute ( $D_{50} = 14 \mu\text{m}$ ) and the 8-minute ( $D_{50} = 3 \mu\text{m}$ ) samples of the 500-mg/L emulsion. Flow rates: 2 minutes, 36.1 mL/min; and 8 minutes, 27 mL/min.

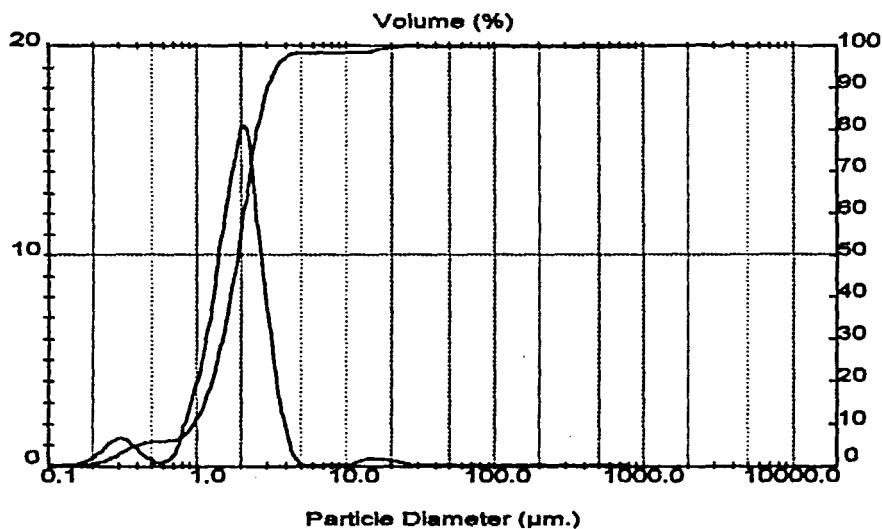


FIG. 7 Particle size distribution of the filtrate at breakthrough for the filtration of the 500 mg/L emulsion (2 minutes) through kenaf 500 bed at 45 psi.

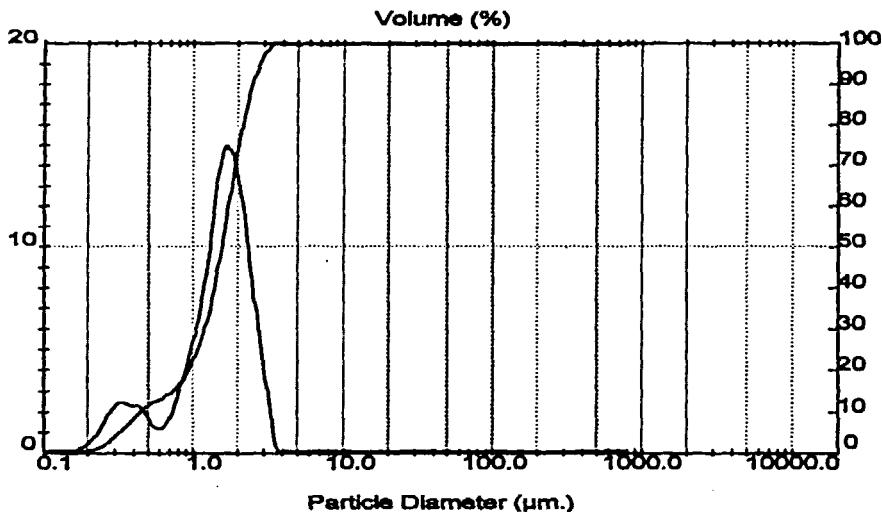


FIG. 8 Particle size distribution of the filtrate at breakthrough for the filtration of the 500-mg/L emulsion (8 minutes) through kenaf 500 bed at 45 psi.

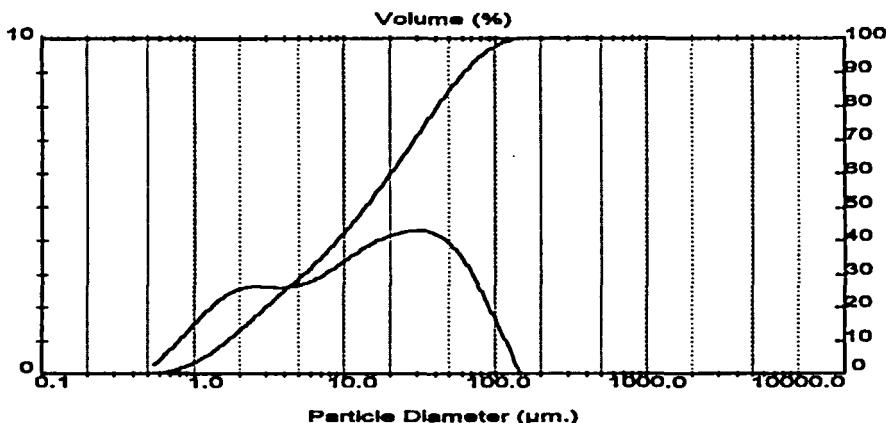


FIG. 9 Drop size distribution at 2 minutes with 500 mg/L oil and 165 mg/L Triton.

#### Effect of Media Particle Size on Removal

Kenaf 200, kenaf 500, and kenaf 1000 were used in the study. Two sets of filtrations were carried out with all the three media: 1) a 2-minute sample of the 500-mg/L emulsion with Triton at 45 psi, and 2) an 8-minute sample of the 500-mg/L emulsion with Triton at 60 psi. Figure 11 shows the results of the first set of filtrations and Fig. 12 shows the results of the second set of filtrations. The results show that removal is best for the kenaf 200 medium

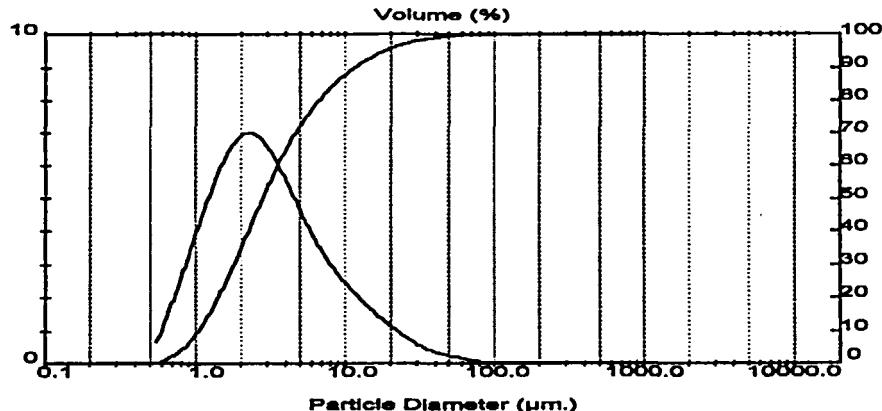


FIG. 10 Drop size distribution at 8 minutes with 500 mg/L oil and 165 mg/L Triton.

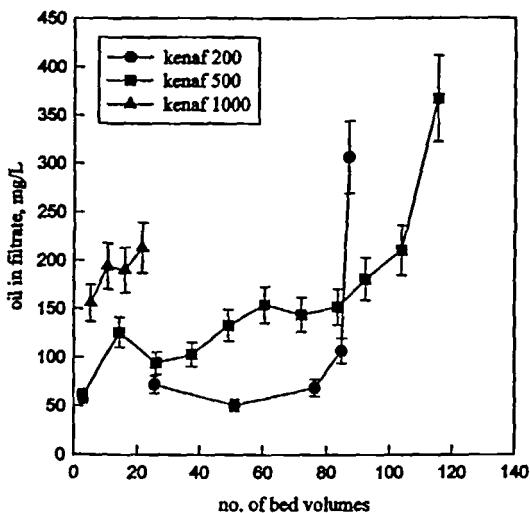


FIG. 11 Effect of particle size of the media on oil removal for the 2-minute sample of the 500-mg/L emulsion at 45 psi. Flow rates: kenaf 200, 54.6 mL/min; kenaf 500, 36.1 mL/min; and kenaf 1000, 178.4 mL/min.

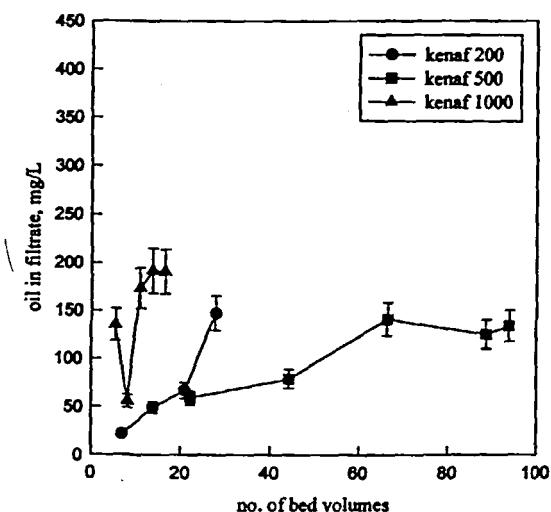


FIG. 12 Effect of particle size of the media on oil removal for the 8-minute sample of the 500-mg/L emulsion at 60 psi. Flow rates: kenaf 200, 29.5 mL/min; kenaf 500, 34.7 mL/min; kenaf 1000, 140 mL/min.

while removal for the kenaf 1000 was the worst and removal for the kenaf 500 fell in between. The increased surface area and the reduced pore sizes of the finer media can explain these results. A larger surface area means more locations for attachment of the droplets. A reduced pore size implies a shorter distance for the droplets to transport from the bulk liquid to the surface of the medium. Both conditions increase the removal.

### **Effect of pH on Removal**

A change in the emulsion pH can cause a change in oil removal and the behavior of the medium. For an emulsion stabilized by a nonionic surfactant, a change in pH cannot change the ionic balance because of the nonionic nature of the surfactant in solution. Therefore, a nonionic surfactant was used in this experiment to stabilize the emulsion. Emulsions were prepared with Triton X100 at three different pHs: a pH 4.5 sample prepared by adding HCl, a pH 9.5 sample prepared by adding NaOH, and an emulsion in DI water, like all other untreated emulsions, that measured pH 6.7. Figure 13 shows that at removal was much better pH 4.5 compared to removal at the other pH values. Removal increased considerably with a reduction in pH. The improved

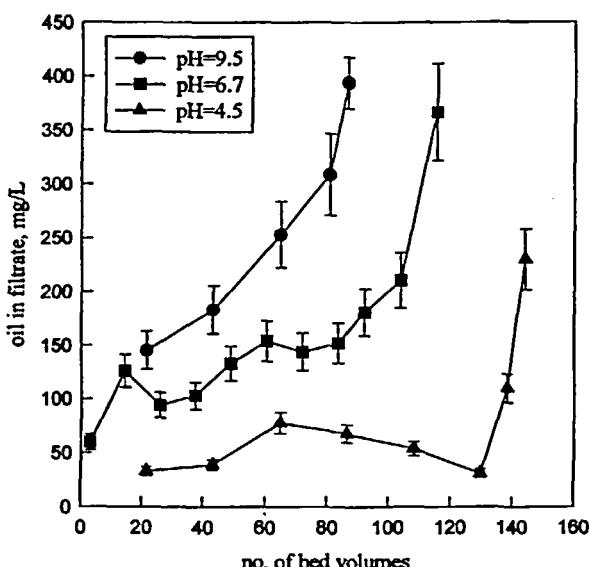


FIG. 13 Effect of pH on oil removal for the kenaf 500 bed and the 2-minute sample of the 500-mg/L emulsion at 45 psi. Flow rates: pH 4.5, 47.1 mL/min; pH 6.7, 36.1 mL/min, and pH 9.5, 37 mL/min.

removal at lower pH can be attributed to protonation of the droplets, or to the change in the ionic strength, or to both. No attempt was made to optimize the pH because: 1) a change in pH will change the ionic equilibrium of an ionic system, which could be good *or* bad for the expected result, and 2) adjusting the pH and neutralizing it later are expensive if the process is scaled up.

### ***Effect of the Depth of the Medium on Removal***

A 7.5-cm long unit was fabricated and used in this experiment. The filtration rate was very low with this unit at 45 psi. A 500-mg/L emulsion with Triton was filtered through a kenaf 500 bed at 60 psi. The plots in Fig. 14 show that the 7.5-cm deep medium had a better removal than the 5-cm medium. Increased medium depth increased the detention time and, in turn, improved the removal.

### ***Effect of Surfactant Type on Removal***

Different types of surfactants were used to obtain insight into the nature of the surface charge of the kenaf media. Triton X100, SDS, and CPC were

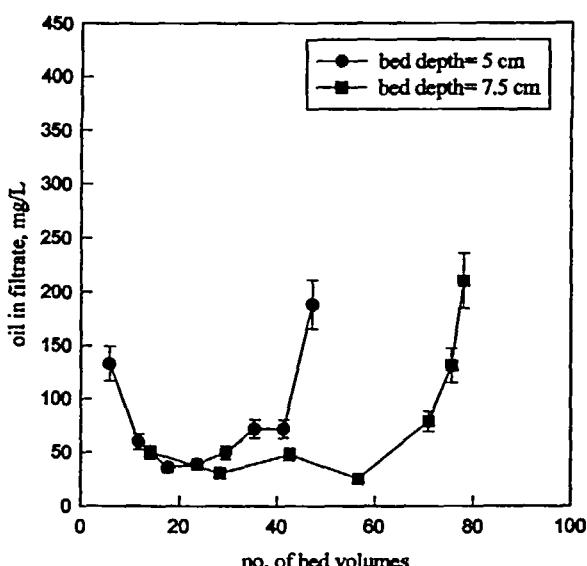


FIG. 14 Effect of media depth on oil removal for the kenaf 500 bed with the 2-minute sample of the 500-mg/L emulsion at 60 psi. Flow rates: 5 cm bed, 44.3 mL/min; and 7.5 cm bed, 31.4 mL/min.

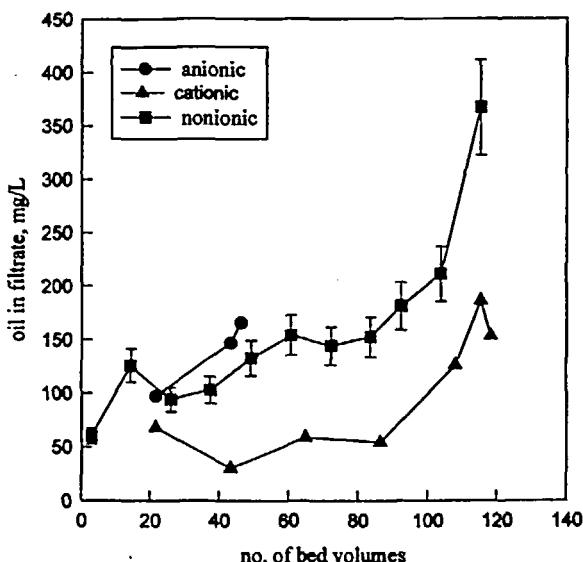


FIG. 15 Effect of surfactant type on oil removal for the kenaf 500 bed with the 500-mg/L emulsion at 45 psi. Flow rates: anionic, 46.7 mL/min; nonionic, 36.1 mL/min; and cationic, 72.7 mL/min.

used in the study to prepare emulsions. Two-minute samples were prepared for Triton and SDS. For CPC, after 2 minutes of mixing, there was a floating oil sheen. The sample had to be mixed for 4 minutes to completely emulsify the oil. All three emulsions were filtered through a kenaf 500 bed at 45 psi until breakthrough. No attempt was made to optimize the surfactant dosage because the optimum or the best result should be obtained with very little or no surfactant, resulting in an unstable dispersion.

Results (Fig. 15) show that removal was best for CPC, the cationic surfactant. Oil removals for SDS, the anionic surfactant, and for Triton, the nonionic surfactant, were not very much different, although removal for the anionic surfactant was less. This observation corroborates the presumption that the kenaf surface is negatively charged. Oil drops covered by the positively charged surfactant were easily removed by the kenaf bed due to the electrostatic force of attraction between the drops and the kenaf grains. The uncharged drops in the Triton-stabilized emulsion had better removal than the negatively charged drops in the emulsion stabilized by SDS. The mutual repulsion between the negatively charged drops and the negatively charged grains caused a very poor removal for SDS.

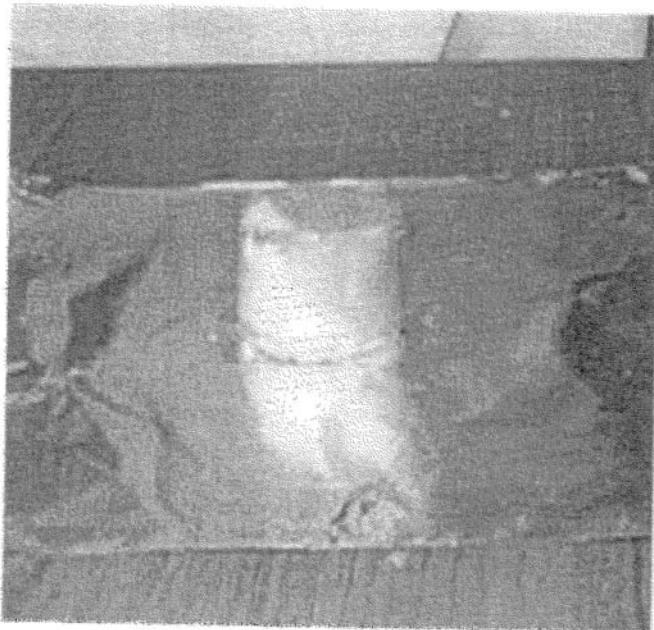


FIG. 16 A typical spent medium showing channeling of flow (note the diagonal line from top left to bottom right; the bed to the left of the line was bypassed due to channeling).

### ***Effect of Channeling Flow on Removal***

All the filtration runs described above were carried out until emulsion breakthrough in the media. The number of bed volumes that can be filtered before bed exhaustion is very important for any media; the more the number of bed volumes processed, the better. The bed volumes filtered in the runs described above were small in comparison to what was expected. The small numbers were a direct result of channeling flow in the kenaf bed. Channeling left 50 to 90% of the beds unused during filtration (Fig. 16) and resulted in early breakthroughs. Channeling might also have caused increased oil content in the filtrate because the flow did not use the full depth of the medium. If fully used, the media should filter three to four times the number of bed volumes achieved in the study.

Uneven packing of the media caused the channeling. It is difficult to achieve even packing with uneven particles, unlike the case of a resin bed. It might be possible to create an array of small diameter tubes individually packed with the medium to give a uniform pressure drop throughout a chamber. This solution would involve a complicated design and time-consuming media preparation, and these were not viable in the time frame of the project.

Although channeling occurred in all the runs, the early portion of the runs was normal filtration, uniform across the medium cross-section, typically for the first 1–2 cm of bed depth. Once channeling began, the medium broke through within a 500-mL filtrate interval. This effect is evident in the plots where the last analytical value is very high compared to the rest. The conclusions reported in this study are based on the analytical results before channeling took place.

### Spent Media

#### *Backwashing the Spent Media*

Backwashing of the exhausted media in order to remove the oil retained in the media was attempted. Backwashing attempts were not successful because the media were well compacted as a cylindrical bloc, and the backwash flow pushed the whole media upward until they hit the top plate. The media were never expanded in the process, and no backwashing was achieved.

#### *Moisture Content of the Spent Media*

The moisture contents in the spent media were analyzed using the Sartorius moisture analyzer at 85°C. The values in Table 4 show that the moisture content decreased with increasing filtration pressure and decreasing particle size of the media. Increasing the pressure compacted the media and reduced the pore volume, consequently reducing the moisture retention. Reducing the particle size of the media reduced the pore volume due to more compact packing of particles in the bed.

#### *Heating Values of the Spent Media*

The calorific values of the spent media were determined using a Parr Oxygen Bomb Calorimeter. The individual Btu values measured were used to

TABLE 4  
Average Moisture Contents of Various Media  
at Various Pressures

Medium	Pressure (psi)	Moisture (%)
Kenaf 200	45	74
Kenaf 200	60	72
Kenaf 500	45	77.5
Kenaf 500	60	75.8
Kenaf 1000	45	80.9
Kenaf 1000	60	79.5

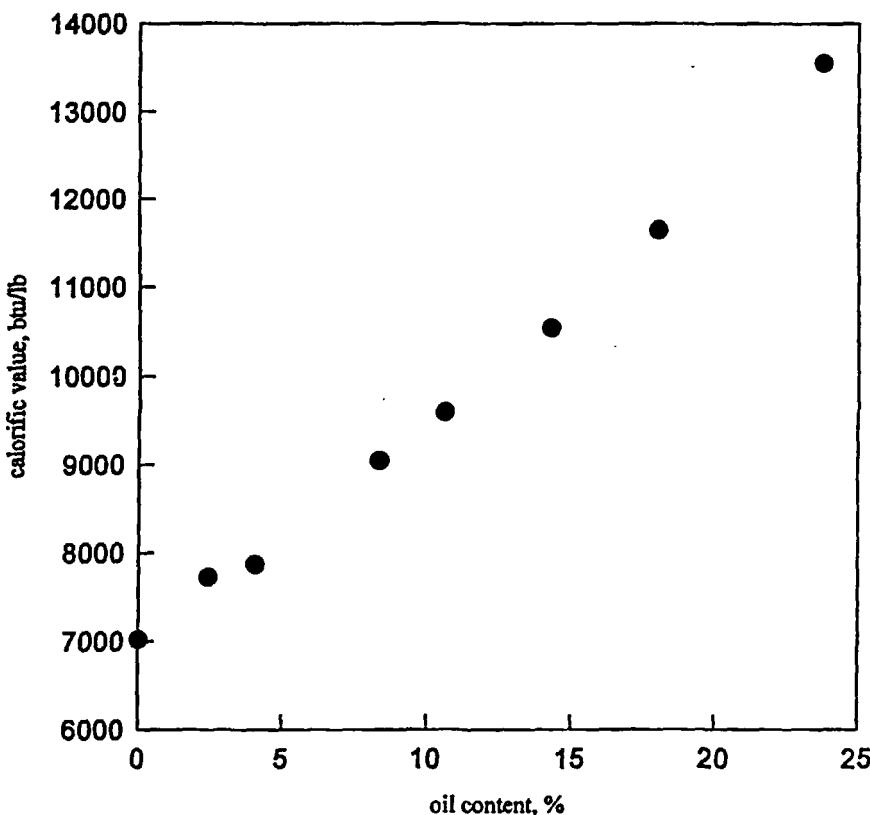


FIG. 17 Calibration curve of calorific value vs oil content in the medium.

estimate the percent oil in the spent media. A calibration curve of calorific value vs percent oil was used for converting the calorific values to percent oil content. The calibration curve was obtained by measuring the Btu values of kenaf samples spiked with different amounts of oil and plotting the values against the oil content (Fig. 17).

Using the calibration curve and the calorific values measured, the percent oil contents in spent media were calculated on a dry basis (Table 5). It is evident from the values in the table that the calorific value of the spent medium depended on the volume of emulsion filtered. The more emulsion filtered, the higher the calorific value because of the increased amount of oil caught in the media.

TABLE 5  
Calorific Values and Oil Contents of Various Spent Media

Medium	Pressure (psi)	Emulsion drop size	Surfactant	Volume filtered (L)	Heating value (btu/lb)	Oil content <sup>a</sup> (%)
Kenaf 200	45	Big <sup>b</sup>	Triton	5.13	9,854	11.05
Kenaf 200	60	Big	Triton	4.8	9,482	9.67
Kenaf 500	45	Small <sup>b</sup>	Triton	5.8	10,089	11.93
Kenaf 500	60	Small	Triton	6.35	10,632	13.94
Kenaf 500	45	Big	Triton	8	11,364	16.66
Kenaf 500	60	Big	Triton	3.2	8,792	7.11
Kenaf 500	45	Big	SDS	3.2	9,101	8.26
Kenaf 500	45	Big	CPC	8.2	12,370	20.39
Kenaf 1000	45	Big	Triton	1.6	7,639	2.83
Kenaf 1000	60	Small	Triton	1.2	7,451	2.13

<sup>a</sup> The values are for the top part of the filter media. The bottom part of the media had calorific values in the 7000–8000 Btu/lb range with a corresponding oil content of 0.5 to 4%.

<sup>b</sup> Emulsion drop sizes "Big" and "Small" refer to the 2- and 8-minute samples.

## SUMMARY

Removal of oil from low concentration, surfactant-stabilized emulsions was investigated by carrying out constant pressure, continuous flow filtrations using kenaf media. Oil removal varied from 70 to 95%. The relationships of oil removal with the concentration of emulsion, the drop size of the emulsion, the particle size of the media, the filtration pressure, the pH of the emulsion, the depth of the media, and the type of surfactant were established.

Regeneration of the spent media was attempted by backwashing the media with DI water. The moisture content of the spent media was measured and related to filtration pressure and particle size of the media. Heating values of the spent media were measured and converted to percent oil content of the media.

Table 6 summarizes the percent removals for the different cases studied. Percent removals were calculated at 40 bed volumes for the cases that filtered 3.2 L or more of emulsion; calculation was done for all other cases at 10 bed volumes. For emulsions stabilized by Triton, a total oil and grease value of 660 mg/L was used, and for those stabilized by SDS and CPC, a value of 500 mg/L was used.

TABLE 6  
Percent Removal for the Various Cases

Medium	Pressure (psi)	Emulsion (drop size)	Surfactant	pH	Volume filtered (L)	Removal (%)
Kenaf 200	45	Big	Triton		5.13	84
Kenaf 200	60	Big	Triton		3.2	95
Kenaf 200	60	Small	Triton		1.6	90
Kenaf 500	45	Small	Triton		5.8	80
Kenaf 500	60	Small	Triton		6.35	80
Kenaf 500 <sup>a</sup>	60	Big	Triton		3.2	89
Kenaf 500	45	Big	Triton	4.5	10	95
Kenaf 500	45	Big	Triton	6.7	8	77
Kenaf 500	45	Big	Triton	9.5	6	53
Kenaf 500/7.5 cm	60	Big	Triton		8.25	88
Kenaf 1000	45	Big	Triton		1.6	68
Kenaf 1000	60	Small	Triton		1.2	71
Kenaf 500 <sup>b</sup>	60	Unknown	Triton		0.8	95
Kenaf 500	45	Big	SDS		3.2	67
Kenaf 500	45	Big	CPC		8.2	91
Kenaf 1000	45	Product water	None	6	1.8	98
Kenaf 500	45	Product water	None	6	1.2	94

<sup>a</sup> Used for comparing the oil removal with the 7.5-cm deep medium.

<sup>b</sup> Oil concentration for this one is 5000 mg/L; all the other emulsions are at 500 mg/L.

## CONCLUSIONS

Kenaf media were effective in removing oil from surfactant-stabilized emulsions. Removal ranged from 70 to 95%. Oil removal was better for larger droplets, higher pressure, finer media, lower pH, cationic surfactant, and deeper medium. This study found that it is not possible to backwash the spent media because of the compact nature of the media. Moisture content of the spent media decreased with increasing pressure and decreasing particle size. Oil content of the spent media increased with the volume of emulsion filtered. Filtration of about 9 L of 500 mg/L emulsion produced a spent medium with a heating value (on a dry basis) comparable to the best quality coal. The bed volumes filtered in the study were low because of the early breakthrough of emulsion that resulted from channeling. If fully used, the kenaf media should be able to filter 300 to 500 bed volumes of an emulsion having a concentration of 500 mg/L and produce a tenfold reduction in oil content.

## RECOMMENDATIONS

Based on the results of the study and other observations, the following areas are suggested for continuation of the study: Use of produced fluid from surfactant-enhanced soil-washing process to determine removal of oil and recovery of surfactant, treatment of kenaf to either give the surface a positive charge or to increase the negative charge depending on the charge of the droplets to be removed, treatment of emulsions to improve removal, and a bench scale combustion study of the spent media to determine combustibility with and without ambient drying, energy recovery, and the production of any harmful gaseous pollutants.

## ACKNOWLEDGMENTS

This research was supported by a grant from the Texas Higher Education Coordinating Board-Advanced Technology Program, and the United States Department of Agriculture. The views expressed are those of the authors and do not imply endorsement by either of the sponsors.

## REFERENCES

1. J. D. Sherwood, "A Model for Static Filtration of Emulsions and Foams," *Chem. Eng. Sci.*, 48(19), 3355-3361 (1993).
2. USEPA, *Final Effluent Limitations Guidelines and Standards for the Coastal Sub-category of the Oil and Gas Extraction Point of Source Category*, 1996.
3. H. A. Ohen, L. Nnabuihe, B. J. Felber, D. Ososanwo, and C. M. Holmgren, "A Systematic Laboratory Core and Fluid Analysis Program for the Design of a Cost Effective Treatment and Cleanup Guidelines for a Produced Water Disposal Scheme," in *SPE/DOE Tenth Symposium on Improved Oil Recovery*, 1996, pp. 245-254.
4. I. Okuda, J. F. McBride, S. N. Gleyzer, and C. T. Miller, "Physicochemical Transport Processes Affecting the Removal of Residual DNAPL by Nonionic Surfactant Solutions," *Environ. Sci. Technol.*, 30, 1852-1860 (1996).
5. Advanced Applied Technology Demonstration Facility, *Technology Practices Manual for Surfactants and Cosolvents*, Rice University, 1997.
6. K. G. Marinova, "Charging of Oil-Water Interfaces Due to Spontaneous Adsorption of Hydroxyl Ions," *Langmuir*, 12, 2045-2051 (1996).
7. E. E. Isaacs and R. S. Chow, "Practical Aspects of Emulsion Stability," in *Emulsion Fundamentals and Applications in the Petroleum Industry* (Advances in Chemistry Series 231), American Chemical Society, Washington, DC, 1992, pp. 51-77.
8. R. I. Mackie and R. Bai, "Suspended Particle Size Distribution and the Performance of Deep Bed Filters," *Water Res.*, 26(12), 1571-1575 (1992).
9. A. C. Payatakes, *Deep Bed Filtration: Theory and Practice*, Course Notes for the ASEE Summer School for Chemical Engineering Faculty, Snowmass, Colorado, 1977.
10. T. Viraraghavan and G. N. Mathavan, "Treatment of Oily Waters Using Peat," *Water Pollut. Res. J. Can.*, 25(1), 73-90 (1990).

11. J. C. Schulz, "Nutshell Filter Technology," *Fluid/Part. Sep. J.*, 9(1), 14-20 (1996).
12. W. A. C. da Silva, B. S. Valdir, and N. L. A. Horta, *Thermodynamic and Economic Simulations of Steam Turbine Co-generation Systems Fueled by Sugar-Cane Bagasse*, Paper Presented at the ECOS '92 International Symposium on Efficiency, Cost, Optimization and Simulation.
13. A. Borazjani and S. V. Diehl, "Kenaf Core as an Enhancer of Bioremediation, A Summary of Kenaf Production and Product Development Research 1989-1993," *Miss. Agric. For. Exp. Stn. Bull.*, 1011, 26-27 (1994).
14. USEPA, *Method 1664* (EPA-821-B-94-004b), Office of Water, Engineering and Analysis Division, Washington, DC 20460, 1995.

*Received by editor December 12, 1997*

*Revision received March 1998*