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Experimental Study on Oil and Solids Removal in Nutshell Filters for Produced Water Treatment

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Abstract

Nutshell filters composed of black walnut or pecan granular media are an established produced water treatment technology for tertiary oil removal. Guidelines for the size and operation of nutshell filters have evolved mainly by trial and error, with limited published operating data. This laboratory research program tested a nutshell filter to determine the operating flux limits (flow rate per unit area) that provide suitable oil removal performance. The separation efficiency target was defined as 5 ppmv oil in the outlet stream. The variables tested included medium type (black walnut shell or pecan shell), medium size, filtration flux, water salinity, and oil concentration. The flux limit for common 12/20 media is 12.0 gpm/ft² for fresh water operation and 13.0 gpm/ft² for saline water. Decreasing the medium size to 20/30 mesh increased the allowable flux limit but at the expense of a substantial increase in the pressure drop. Oil droplet penetration into the filter bed proceeds via a near plug-flow profile, with the top 18 inches of the bed providing 99% of the oil removal. Full breakthrough is a function of inlet oil concentration, with 20-30 hours of operation expected for a 48-inch-deep bed. Testing for removal of fine sand particles showed a average 5 µm separation size for 12/20 media at 13.5 gpm/ft² flux.

Introduction

Deep-bed media filters that use granular nutshells, commonly termed "nutshell filters," are popular for tertiary produced water treatment to remove insoluble hydrocarbons and suspended solids. Inorganic media, such as sand, anthracite, or garnet, are used in deep-bed filters in the municipal water treatment industry for filtering waste water. However, the petroleum industry prefers nutshell filters because of their excellent oil and solids filtration performance at high treatment flow rates, combined with ease of backwashing. Nutshell filters have not necessarily been proven to be the best solution for produced water polishing; however, they are the standard technology, especially for onshore use. The footprint, weight, and motion sensitivity of conventional nutshell filters preclude their use in most offshore facilities.

An example of an onshore produced water treatment process is shown in the schematic in **Fig. 1**. Primary oil and solids removal is accomplished with a skim tank or corrugated plate interceptor (CPI). In this stage, oil at 1000-2000 ppmv is reduced to 300-500 ppmv. This stage also serves to handle upsets or carryover from three-phase separation. Oil droplets in the ≥100 µm range are removed from the water in the primary stage by a combination of coalescence and gravity mechanisms (Chin 2007). Secondary treatment uses induced gas flotation to reduce the oil content to 30-50 ppmv and the droplet size to <20 µm. The target specification for the tertiary, or polishing, stage is <5 ppmv oil. This target level is set for produced water reinjection to protect the disposal or pressure maintenance reservoir from plugging or for water re-use to protect equipment used for the removal of dissolved organics and solids. Validation of the design and the determination of the operating conditions required for a nutshell filter to achieve a 5 ppmv outlet oil content are the subjects of this study.

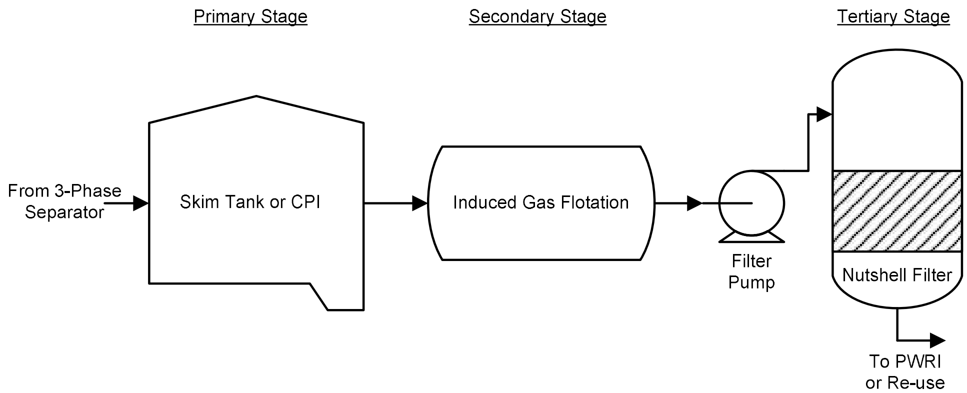

Fig. 1 - Nutshell filter arrangement for an onshore produced water treatment plant.

Basic Nutshell Filter Operation

Nutshell filters operate with two main process cycles: filtration and backwash. Many different nutshell filter models and configurations are commercially available; they generally follow the same principles during the filtration cycle but vary in the backwash method. Design options for backwash scrubbing include an internal impeller, spray jets, an external scrubber pump, and external or internal screens.

An example design with an external pump and internal screen is shown in **Fig. 2**. In filtration mode, valves A and E are open, allowing continuous downflow of the water through the media bed. Oil and solids are captured by the media and accumulate within the bed depth. Contaminant build-up increases resistance to flow, indicated by a corresponding increase in pressure drop. If the pressure drop becomes too high, then fluid channeling or bypass will occur, allowing oil to reach the outlet. A pre-set pressure-drop alarm, e.g., 15 psi, indicates the need to clean the media bed.

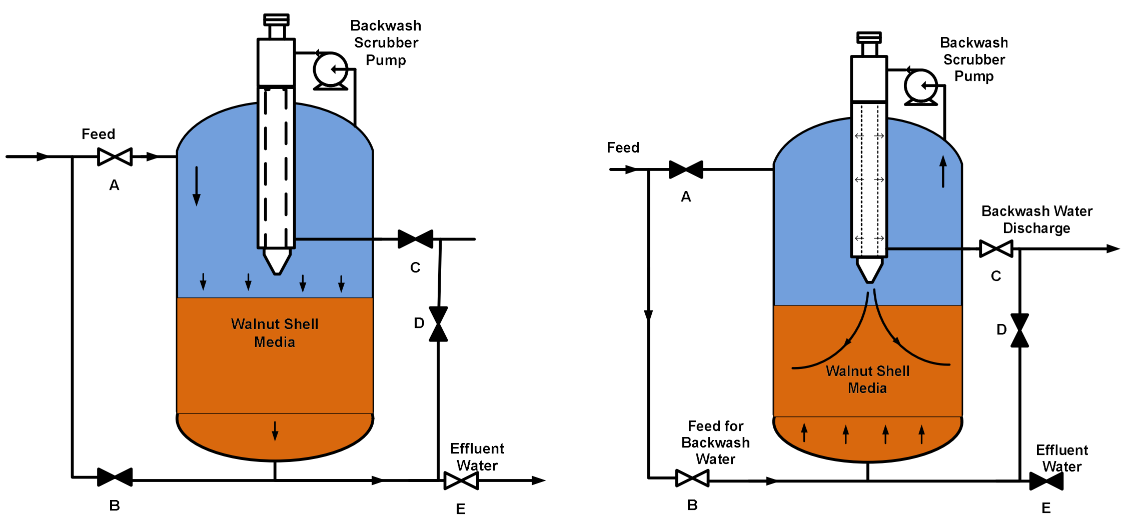

Fig. 2 - Nutshell operation during (left) filtration and (right) backwash cycles.

During backwash, the filter is taken offline (valves A and E are closed) and a standby filter is brought online to allow continuous water treatment in the plant. Several different methods can be used to scrub the media bed (i.e., mixers, impellers, and sprays). In the example shown in **Fig. 2** (right), feed water is first used to fluidize the media bed by opening valve B. The scrubber pump draws in the fluidized dirty media, wherein oil is scrubbed off via shearing as it passes through the pump. The mixture is returned to the filter body by passing through a cylindrical screen enclosed in a pipe. The removed contaminants (oil and small solids) pass through the screen to be collected in the pipe annulus and are flushed from the vessel through a pipe connected to valve C. The screen is sized to retain the filter media, which returns to the vessel. Circulation and backwashing of media lasts 10-20 minutes. Upon completion of the backwash, the media is allowed to settle, and the filter returns to normal filtration mode.

Media Selection for Nutshell Filters

The first nutshell filter, which used walnut shells as the filter medium, was introduced in two patents in 1975 (Hirs 1976a, 1976b). Other minerals and polymers have been evaluated, some of which exhibit better oil affinity (i.e., PVC); however, walnut shells have several unique characteristics. They have a balanced affinity for oil that is sufficiently strong to aid in adsorption filtration but sufficiently weak to release the oil during backwash scrubbing. PVC has a much higher oil affinity, but does not release the oil easily during backwash. Walnut shells have lower density than sand or garnet and therefore require less energy for fluidization and scrubbing; however, they are tougher than anthracite and do not fracture or degrade easily from attrition. Hirs did not state the method of oil capture but claimed that walnut shells coalesce fine suspended oil globules, which are subsequently captured in the filter-bed interstices. Hirs identified black walnut shell (Juglans nigra L.) specifically as the best medium, and this material has become the industry standard. Other granular "nutshell" media have been used, including English walnut, pecan, coconut, apricot hull, and palm kernel; however, black walnut shell (BWS) is still preferred.

The fundamental mechanisms that occur during nutshell filtration and backwashing in terms of oil-media, solids-media, and media-media interactions have received little attention in the literature. A study by Rawlins and Erickson (2010) provides a literature review of walnut shell filter analyses. Investigations by Rahman et al. (1988, 1992), Kenawy and Kandil (1998), Evans and Robinson (1999), Mantilla and Quintero (2000), and Blumenschein (2001) provide anecdotal testing results in various applications but no elucidation of the mechanisms involved. Srinivasan and Viraraghavan (2008) provided the only technical analysis and found that oil sorption decreased with increasing oil viscosity and interfacial tension and that walnut shells exhibit excellent surface characteristics for coalescing and filtration. The method of oil droplet capture during filtration was not studied.

Rawlins and Erickson (2010) provided the first comparison of the physical, chemical, and hydrodynamic properties of granular filter media. They investigated six types of media: garnet, silica, anthracite, pecan shell (PS), English walnut shell, and BWS. The best medium for oil capture, as determined by wet retention testing, is PS, followed by walnut shell. A comparison of the average quotient (grams of oil per grams of media) for three test oils reveals that PS held 0.32 g oil/g medium, whereas English and black walnut held the same amount at 0.27 g/g (~15% less than PS).

All media exhibited a finite contact angle with water, ranging from 23° for pecan to 31° for black walnut to 51° for English walnut (Rawlins and Erickson 2010). A contact angle of 0° indicates full wetting and 180° indicates full repulsion. The range of values did not vary substantially, and all materials were moderately wetted by water. All materials were completely wetted by the test oils (0° contact angle). The nut shell medium is strongly oleophilic and moderately hydrophilic.

During backwash scrubbing, the particles are agitated violently and subjected to inter-particle impact, a process called self-attrition. The material most resistant to self-attrition is black walnut, followed closely by English walnut. Both walnut types reached a plateau of ~0.5% loss after 10 hours of self-attrition, simulating 30 days of normal backwash cycle use. All other media continued to lose weight even after 60 hours of testing (~180 days of use).

A comparison of the tested parameters, including oil wetting, fluidization flux, self-attrition resistance, and pressure drop, indicated that black walnut was the best overall medium. This material exhibits high oil wetting, a low fluidization flux, and low self-attrition.

Hensley (1987, 1990) claimed that a mixture of pecan and walnut shells provided improved filtration for process water containing solids and oil. The preferred shell mixture is 80% PS and 20% BWS. The BWS serves to increase filter bed porosity, whereas the PS provides the primary filtration duty. Further results of mixed-bed testing have not been published in the literature.

Engineered and synthetic media have been designed to improve deep-bed media filters for the produced water industry. Examples of new media include surface-modified black walnut with an oleophilic polymer (Xedia Process Solutions 2016) and fully synthetic polymer granules (Schlumberger 2016, Siemens 2016). These synthetic media are claimed to increase oil-carrying capacity in the filter bed, maintain filtration performance at high feed oil concentrations, and improve filtration performance under standard conditions. The remainder of this study focuses only on standard, unmodified, nutshell media for filtration of oil from water.

Flux in Nutshell Filters

The primary sizing criterion for media filters is the filtration flux. Stated in terms of flow rate per unit area, the most common unit for flux is gpm/ft². Municipal water clarifiers (deep-bed media filters with sand) have a low flux of 2-6 gpm/ft² (Hudson 1981). Media with low flux values, in combination with coagulants, are used to capture very small bacteria particles. The design flux for simple graded bed sand/anthracite filters for oilfield use is also low at 2-3 gpm/ft² (Bradley 1987). Bradley (1987) claimed that a multimedia high-rate deep-bed filter with anthracite and multiple sizes of garnet, in conjunction with a polyelectrolyte coagulant, has flux values of 15-20 gpm/ft². This design is for solids removal only because oil is very difficult to remove after being adsorbed onto anthracite. The nutshell media filter with BWS has a stated flux of 10-15 gpm/ft², with data given for a flux of 10.6 gpm/ft² (Bradley 1987).

The initial design patents by Hirs (1976a, 1976b) do not recommend a design flux value; however, later patents by Hensley (1987, 1990) recommend a flux of 11.1 gpm/ft². Rahman (1988) tested walnut shell filters on produced water at 8 gpm/ft², whereas Blumenschein (2001) tested the filters on steel mill effluent water at 13-26 gpm/ft². The latter application was for solids removal only; oil removal efficiency was not stated.

Tyrie (2011) provides a breakdown by medium and application of flux for deep-bed media filters. Sand has a low flux of 2 gpm/ft² for oil and solids removal. Adding anthracite increases the flux to 6-8 gpm/ft². Switching to garnet plus anthracite increases the flux to 8-12 gpm/ft². BWS has a flux of 11 gpm/ft².

Some discrepancies exist regarding the recommended flux for a BWS filter used to remove oil and solids from produced water. The literature reviewed shows a limit of 10-11 gpm/ft². The latest Hensley patent (1995) increased this recommended limit to 13.5 gpm/ft². This latter value has become the industry-accepted maximum flux; however, the conditions under which the nutshell filter can perform to this degree have not been reported. The maximum flux provides the diameter of the nut shell filter for a given flow rate and is thus the key factor in determining equipment size.

Laboratory Testing for Oil Droplet Removal

Verification of the maximum flux that permits efficient oil removal was tested in a laboratory nut shell filter flow loop. The goal of the testing was to determine if the nutshell filter could operate at a flux of 13.5 gpm/ft² while providing an outlet oil concentration of <5 ppmv. BWS and PS filters were tested, and the performance of the laboratory unit was evaluated by quantitative measurement of outlet oil-in-water concentrations under specific conditions.

Equipment Setup.

Figure 3 shows the test flow loop setup. The nut shell filter was built using a 4-inch-diameter clear PVC pipe, with inlet and outlet lines scaled as appropriate. This diameter was chosen to minimize wall effects while permitting rapid testing of numerous variables. Feed water (fresh or saline) was sourced from a storage tank and delivered to the filter through a poly line and gear pump. The gear pump was operated at a fixed speed and was equipped with a recycle line to return excess flow to the feed tank. A globe valve and turbine flowmeter on the inlet line were used to set the flux. A peristaltic pump injected oil into the feed line, and the oil and water were passed through a static mixer to create a dispersion. Although the droplet size is an important process parameter for the evaluation of filter performance, it was not directly measured in this study. Accurate droplet size must be measured in-situ (not through sampling), and an in-line droplet size measurement device was not available during our research program. Inlet water entered the top of the column, and then passed through a 48-inch bed of granular media. A retaining screen of appropriate mesh was used to hold the filter bed in place. Outlet water was collected into a poly line and directed to the drain. Samples for oil concentration measurements were collected from the outlet line.

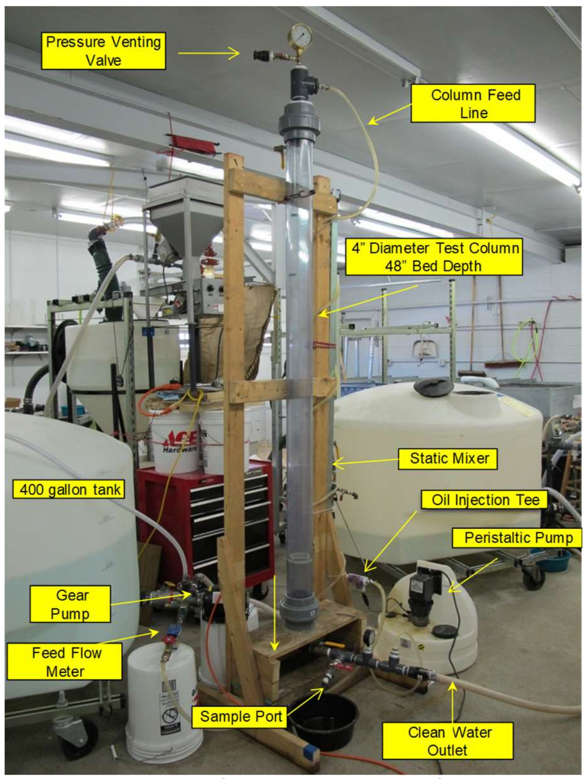

Fig. 3 - Photograph of laboratory nut shell filter test loop.

Fresh-water tests were conducted using a continuous pass through of water. Testing with saline water required recycling of the liquid. To prevent build-up of oil in the feed tank during water recycling, the outlet stream was passed through an oil-adsorbent-packed fiber bed. Samples were periodically collected from the recycle stream to ensure that the oil content remained <1 ppmv (below the detection limit of the instrument).

Test Variables and Materials.

Table I lists the test parameters and fluid properties. Unrefined Bakken crude was used as the test oil. Saline water was made from tap water and bulk salt (NaCl). Oil and water properties were measured in the lab prior to testing. Nut shell filter media (black walnut and pecan) were the same materials as previously reported (Rawlins 2010). Oil concentrations (inlet and outlet) were measured with a Turner

Designs TD-500 D oil-in-water meter. This unit has a measurement range of 0-1000 ppmv with 0.1 ppmv resolution.

Table 1 – Variables Investigated in Nut Shell Filtration Tests

Variable	Units	Value Range
Controlled		
- Column Diameter	inches	4
- Bed Depth	inches	48
- Run Time	minutes	360
- Oil Type	-	Bakken Crude (unrefined)
- Oil Density	kg/m³	846 (35.8 API)
- Oil Viscosity	cP	7.6 (at 65°F)
- Temperature	°F	Ambient (65°F)
- Water Density (fresh)	kg/m³	1007
- Water Viscosity (fresh)	cP	1.01 (at 65°F)
- Media Preparation	-	Pre-soaked, screened, and settled
Independent		
- Media Type	-	Black walnut shell (BWS) Pecan shell (PS)
- Inlet Oil Concentration	ppmv	50, 100
- Flux	gpm/ft²	7.0, 10.0, 11.0, 11.5, 12.5, 13.5
- Salinity	wt.%	0%, 10%
- Water Density (at 10% salinity)	kg/m³	1080
- Water Viscosity (at 10% salinity)	cP	1.25 (at 65°F)
Dependent		
- Oil outlet concentration	ppmv	As-measured

Test Procedure.

Prior to starting a test, the flow lines and the empty filter were flushed with clean water for 15 minutes. The selected test medium was soaked in fresh water for 1 hour and then sieved at the appropriate bottom sieve size to remove any fine particulates. For example, the 12/20 mesh media was wet-washed at 20 mesh. An appropriate quantity of medium was charged into the column, with fresh media used for each test. The medium was packed by flushing the medium upward with fresh water and then tapping the column with a mallet as the media settled. Packing was performed to remove entrained air bubbles in the media bed. With the medium in place, clean water was flushed through the bed at the test flux for 10 minutes to remove any remaining fines.

The variables in Table I provided seventeen test configurations for testing oil filtration; including medium type, medium size, flux, oil concentration, and water salinity. Each filtration test ran for 360 minutes. After oil injection was started, inlet and outlet samples were collected at 30-minute intervals. Upon completion of a test run, the filter was fully cleaned and set up for the next run.

An additional test was conducted for oil penetration rate and quantification. The experimental setup followed the same protocol as filtration testing and used unrecycled fresh water. This test used 12/20 BWS, 12.0 gpm/ft² flux, and 100 ppmv oil. The test ran for 360 minutes, and inlet and outlet samples were collected at 30-minute intervals. The injected oil was mixed with a UV dye to allow visual tracking of the oil as it accumulated within the filter bed. Upon completion of the test, the filter bed was left in the column and the head space water was withdrawn without disturbing the medium in the bed. The filter medium was removed from the column in six-inch sections for quantitative oil measurement. Each six-inch section was placed into a stirring vessel, and two liters of hot water was added. The mixture was stirred vigorously with a paddle mixer to release the adsorbed oil. After stirring, the mixture was allowed to settle, with the oil rising to the top and the medium settling to the bottom. The oil layer was decanted to measure the volume using a graduated cylinder, and this value was converted to weight using the oil density. The filter medium was dried in an oven, and the two measurements were used to calculate the weight percent oil on the dry media.

Results

The key findings from the testing are presented. **Figure 4** shows the oil filtration performance of BWS medium from 7.0-13.5 gpm/ft² flux. All tests shown used fresh water (0% salinity), 12/20 medium size, and 100 ppmv inlet oil concentration. This medium provided <5 ppmv outlet oil at a flux of ≤11.5 gpm/ft². Increasing the flux to 12.5 gpm/ft² resulted in performance deterioration. The target outlet oil levels were achieved during the first half of the test period but increased to 10 ppmv at 6 hours. The maximum flux showed sporadic breakthrough of oil, with spikes as high as 45 ppmv and an average of ~25 ppmv. At these conditions, a flux of 13.5 gpm/ft² would not provide the desired filtration performance for BWS at 12/20 mesh.

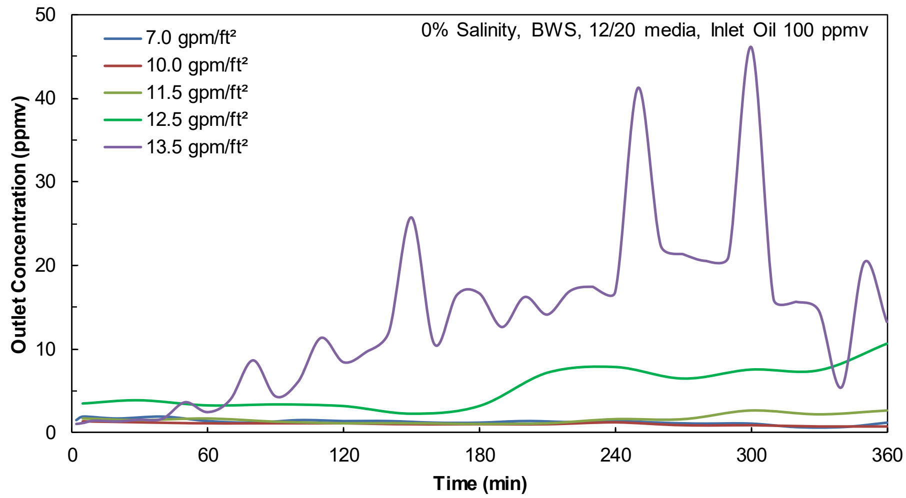

Fig. 4 - Comparison of flux for BWS with fresh water, 12/20 medium size, and 100 ppmv oil inlet concentration.

PS at 11.0 gpm/ft² flux showed slightly worse performance compared to BWS; however, both media satisfied the outlet criterion during the 6-hour test period, as shown in **Fig. 5**. The 13.5 gpm/ft² flux showed oil breakthrough for PS, similar to what was observed with BWS. The performance was sporadic, with spikes as high as 45 ppmv for BWS and 35 ppmv for PS; the average for both was ~25 ppmv. Under these conditions, a flux of 13.5 gpm/ft² would not provide the desired filtration performance for either medium at 12/20 mesh. No clear benefit in oil removal was observed for PS over BWS.

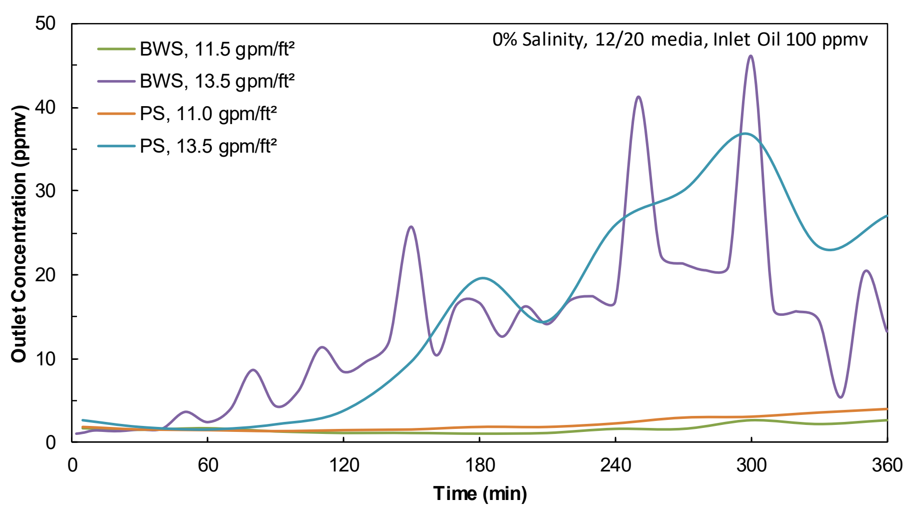

Fig. 5 - Comparison of flux for BWS and PS with fresh water, 12/20 medium size, and 100 ppmv oil inlet concentration.

Figure 6 shows a comparison of the mixed medium, 80% PS + 20% BWS, which has been claimed to enhance filtration performance (Hensley 1987, 1990). Under the same test conditions as **Fig. 4** with 13.5

gpm/ft² flux, the media mixture performed worse than either single medium, with spikes as high as 50 ppmv oil and an average of ~35 ppmv.

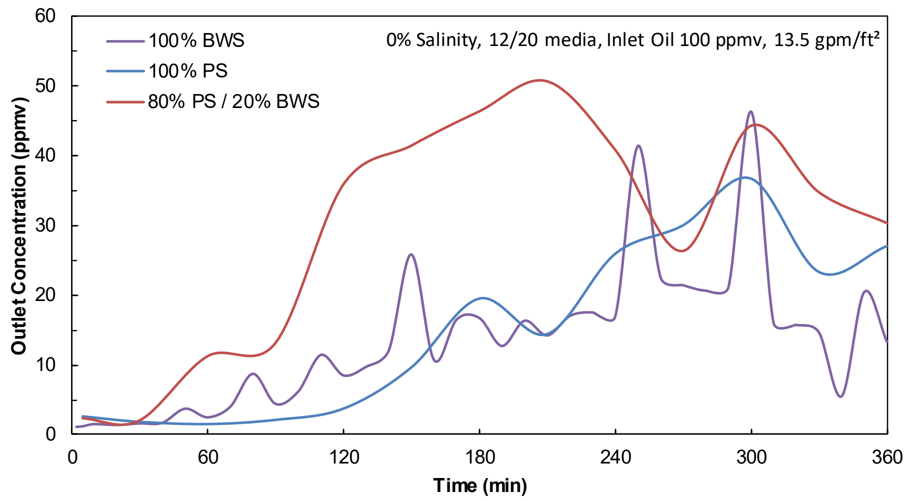

Fig. 6 - Oil filtration performance of mixed media (80% PS/20% BWS) in fresh water at 13.5 gpm/ft² flux.

Decreasing the medium size improved the oil removal performance, as shown in **Fig. 7**. Tests using smaller-mesh media were conducted in fresh water at 13.5 gpm/ft² flux and 100 ppmv inlet oil. Neither the 8/12 nor the 12/20 medium achieved <5 ppmv oil outlet under these conditions. The 8/12 medium resulted in an initial breakthrough of up to 25 ppmv, after which the performance was similar to that of the 12/20 size. However, at the end of the test run, the 8/12 medium resulted in an oil outlet of ~45 ppmv. Smaller media, 20/30 size, delivered excellent performance under these conditions, providing <5 ppmv outlet for the entire test duration. At 13.5 gpm/ft² flux, the 20/30 medium can satisfy the desired criterion in fresh water.

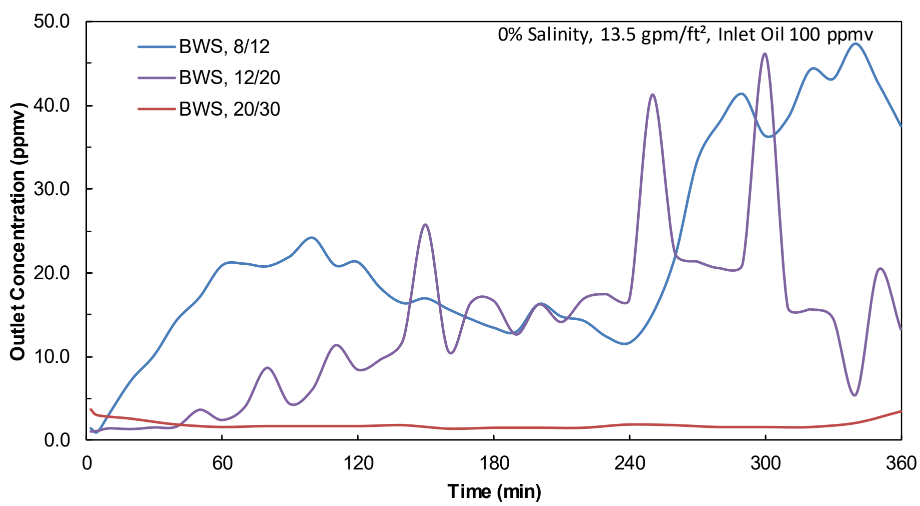

Fig. 7 - Comparison of medium size for BWS operating in fresh water at 13.5 gpm/ft² and 100 ppmv inlet oil.

Figure 8 shows that increasing water salinity improved the filtration performance. These experiments compare 12/20 BWS at 13.5 gpm/ft² flux in fresh and saline (10 wt.% NaCl) water. The fresh water data are the same as those shown in **Fig. 4**. Increasing the salinity prevented the outlet oil spikes observed in the case of fresh water and lowered the average outlet oil concentration. At 100 ppmv inlet oil, the outlet averaged 5 ppmv for most of the test period, then gradually increased to 10 ppmv. At 50 ppmv inlet oil, the outlet averaged 3 ppmv and ended at 7 ppmv. When fresh water was used, 13.5 gpm/ft² flux resulted in oil filtration failure of the 12/20 BWS medium. However, as most produced waters have a high salt content, this flux can be approached more confidently. A flux of 13.0 gpm/ft² is recommended as a conservative limit for 12/20 medium size in most oilfield cases.

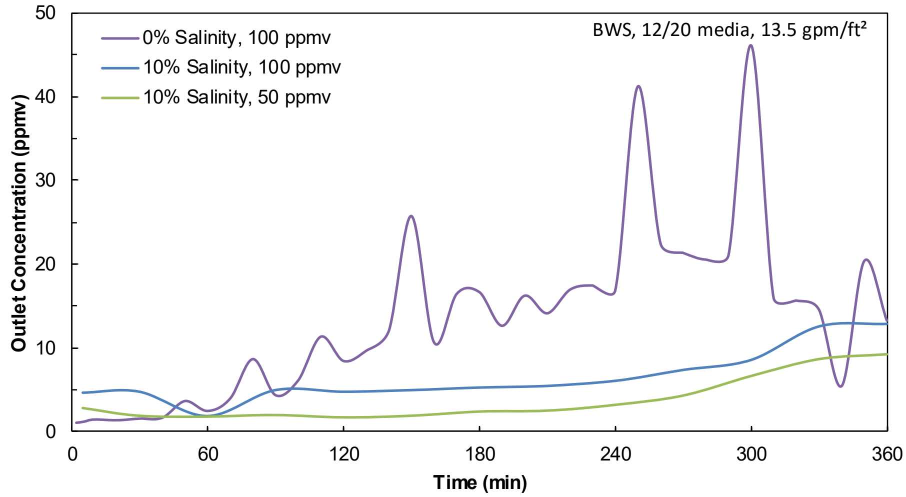

Fig. 8 - Oil filtration performance of BWS in fresh and saline (10 wt.%) water at 13.5 gpm/ft² flux.

A comparison of pressure drops through the 48-inch media bed for several medium sizes and types is shown in **Fig. 9**. These data are for fresh water at 13.5 gpm/ft² flux and 100 ppmv oil. For BWS, the pressure drop was inversely proportional to the medium size. The 8/12 medium (coarsest size) generated a very low pressure drop of ~1 psi for the 6-hour test. Decreasing medium size to the most commonly used 12/20 increased the pressure drop to ~2.5 psi, whereas the smallest BWS of 20/30 mesh generated a substantial increase in pressure drop (14 psi at the 6-hour mark). The 20/30 BWS did provide <5 ppmv oil at the outlet for the test duration (at 13.5 gpm/ft² flux) but did so at the expense of a significant pressure drop. PS exhibited the lowest pressure drop of all the investigated media and decreased during the test period. This result indicates that PS does not pack as tightly as BWS, consistent with the result previously reported after conducting hydraulic analysis of individual media (Rawlins 2010, Fig. 12).

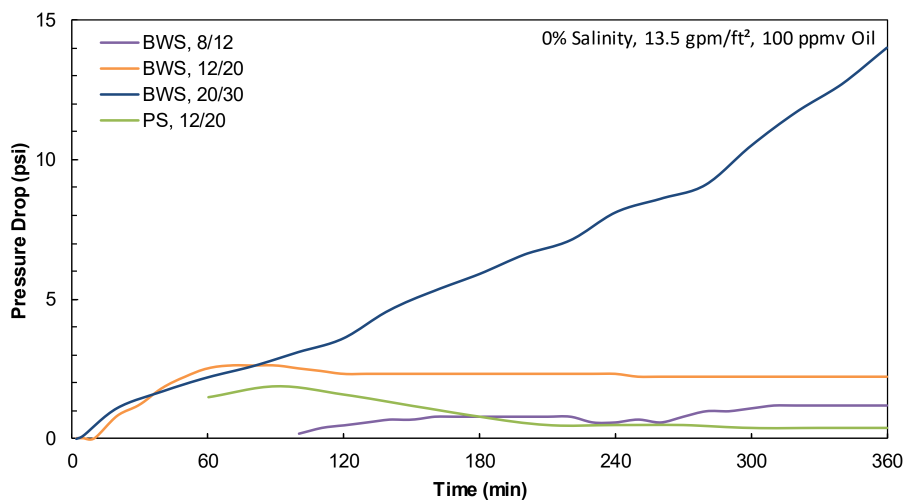

Fig. 9 - Comparison of pressure drop for BWS and PS at 13.5 gpm/ft² flux in fresh water with 100 ppmv oil.

Figures 10-12 show the results from the oil penetration tests. These tests were conducted at 12.0 gpm/ft² fresh water with 100 ppmv oil inlet concentration and with 12/20 BWS medium. This flux value was chosen to push the media filter beyond the target value of 11.5 gpm/ft² in **Fig. 4**, but below the 13.5 gpm/ft² non-performing point. The outlet oil concentration remained below 5 ppmv for the test duration.

The oil for this test was mixed with a dye to render the droplets visible under UV light. **Figure 10** shows two photographs from the testing; the oil appears as near-white particles. The left photo was taken at 45 minutes, when the oil droplets appeared as small discrete particles. The right photo was taken at 360 minutes; the collected oil is shown as large "blobs" similar in size to the filter medium.

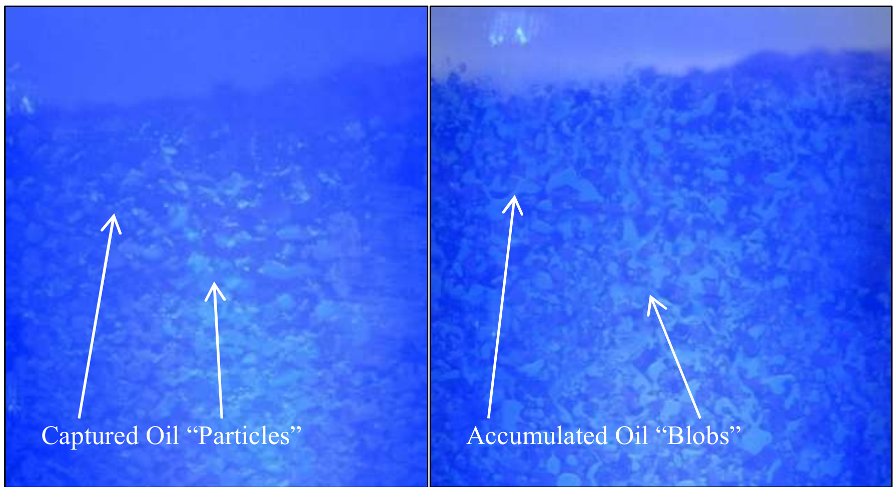

Fig. 10 - Photographs showing oil accumulation at the media interstices. (Left) Oil as particles at 45 minutes of testing; (right) oil as blobs at 360 minutes of testing.

A clear, nearly level interface of oil as it moved down the bed was measurable starting at ~95 minutes. This interface moved downward as the test proceeded, and timed measurements yielded the rate. **Figure 11** shows the interface level (inches from the top of the bed) versus time. The starting point was 11 inches, which increased to 23 inches over the test duration. Following a power-law fit, full breakthrough of 48 inches should occur at 20.9 hours.

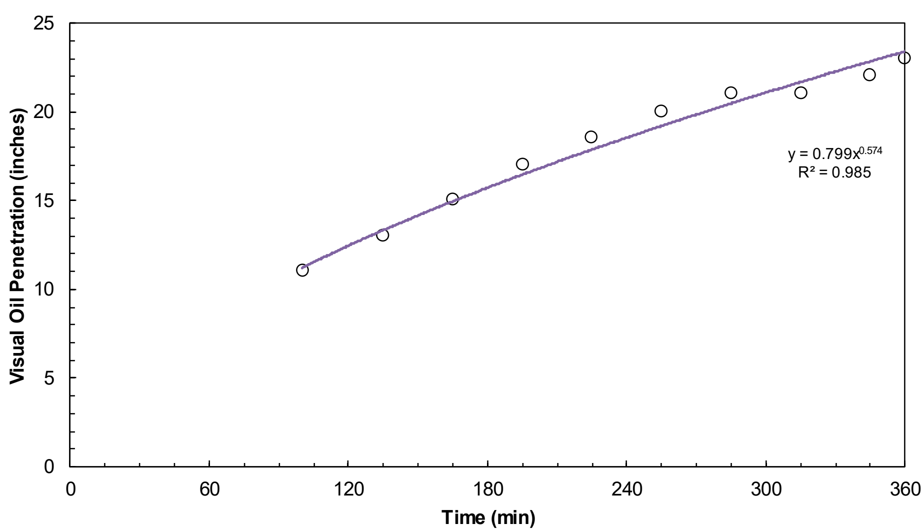

Fig. 11 - Penetration (inches from top) of oil interface in a 12/20 BWS medium bed at 12.0 gpm/ft² flux, fresh water, and 100 ppmv oil inlet concentration.

Figure 12 shows the quantity of oil captured within the media bed, as measured at the end of the test run. As described in the test procedure, 6-inch sections of the media were removed and analyzed for oil content. The amount of oil, as weight% oil on media (dry basis), is reported for each 6-inch section. These data are shown for the midline of each 6-inch section (i.e., 3 inches, 9 inches, 15 inches, etc.) as the bed penetration depth. All oil (>99%) was accumulated within the top 21 inches of the media bed, which correlates to the visual interface data in **Fig. 11**. These data indicate that any interactions between the oil and vessel material or wall effects were negligible during testing. Oil capture in the media bed advanced at nearly a level plug-flow interface.

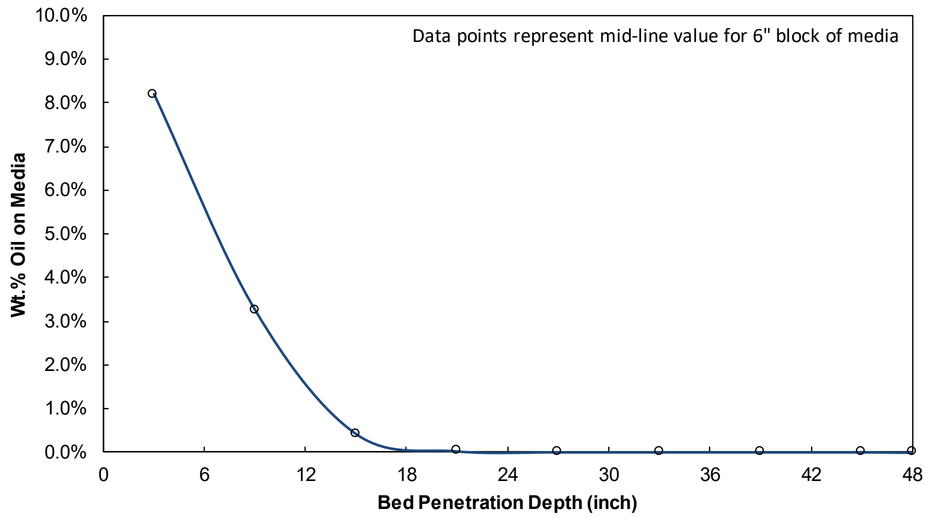

Fig. 12 - Oil concentration at the midpoint of each 6-inch section upon completion of the test run.

Discussion

Several design and process parameters were investigated to determine the highest flux that provides <5 ppmv oil outlet. A comparison of these parameters is listed as follows.

Flux.

In the baseline configuration (12/20 medium, 100 ppmv inlet oil, and 0% salinity) with BWS, fluxes of 7.0-13.5 gpm/ft² were investigated, with 12.5 gpm/ft² flux maintaining <5 ppmv outlet for 6- and 18-hour test durations. A higher flux resulted in >5 ppmv outlet prior to the end of the 6-hour test. Decreasing the medium size to 20/30 mesh kept the oil outlet at <5 ppmv at 13.5 gpm/ft² flux. Higher fluxes were not tested, as the pressure drop with 20/30 medium was deemed too high for normal operation (17 psi at 6 hours). Pressure drop for the 12/20 medium remained <3 psi for the test duration.

Medium Type.

Under the baseline conditions, the PS medium showed no improvements compared to BWS. The results obtained when the flux was reduced to 11.0 gpm/ft² with PS medium were similar to those obtained with BWS at the same flux. In addition, mixed media (80% PS and 20% BWS) at 12/20 mesh and 13.5 gpm/ft² flux showed no improvement in performance.

Salinity.

Increasing salinity improved oil removal efficiency. BWS at 12/20 mesh and 13.5 gpm/ft² flux in fresh water reached >5 ppmv at ~70 minutes. Increasing salinity to 10% under the same conditions eliminated oil breakthrough spikes and increased the operating time to ~200 minutes.

Inlet Oil Concentration.

Reducing inlet oil concentration delayed the breakthrough of oil from the media bed. Inlet oil concentrations of 100 ppmv and 50 ppmv were compared using 10% brine with 12/20 BWS at 13.5 gpm/ft² flux. At 100 ppmv inlet, the outlet oil concentration remained below 5 ppmv for 200 minutes; by contrast, at 50 ppmv inlet, the outlet oil concentration remained below 5 ppmv for 320 minutes, corresponding to a 60% increase in operating time. A smaller medium size (20/30 BWS) at 13.5 gpm/ft² flux showed negligible differences in performance between the two inlet oil concentrations.

Oil Penetration.

BWS medium under baseline conditions demonstrated suitable efficiency (<5 ppmv oil outlet) at 12.0 gpm/ft² flux. At this flux, an oil penetration depth of 23 inches occurred over 6 hours of testing. Extrapolating the advancement of the oil interface suggested that the oil interface would proceed to a bed depth of 48 inches after ~21 hours. Oil droplets initially act as discrete particles that are trapped by straining. As filtration proceeded, these droplets coalesced to form large pools of oil that collected within the interstices between the media particles.

This quantitative assessment of oil penetration in the media bed compares well with the visual observations. Although trace amounts of oil were visible through the entire bed depth, the concentration decreased to <0.1 wt.% at 23-inch penetration. Approximately 95% of the oil was contained in the first 12 inches of the media bed and >99% in the first 18 inches of the bed.

Recommendations for Oil Filtration

Based on the test results, the following parameters are recommended for the design and operation of a nutshell filter for oil removal from produced water:

1. For freshwater operation, the maximum flux for BWS media is 12.5 gpm/ft² for 12/20 mesh and 13.5 gpm/ft² for 20/30 mesh.
2. As water salinity increases, these flux values can be increased by 0.5-1.0 gpm/ft².
3. PS shows no performance advantage over BWS; in addition, given the higher attrition rate of PS, BWS is preferred.
4. Mixed media (i.e., BWS and PS) does not result in a performance improvement per the limited testing performed here. Anecdotal data from field operations suggest that mixed media does have advantages; this topic requires further analysis.
5. Decreasing media size (i.e., from 12/20 mesh to 20/30 mesh) does offer better filtration performance at high flux but with a substantial increase in pressure drop.
6. Oil penetration into the media bed progresses via a near plug-flow profile. Full breakthrough should occur at 20 hours for 100 ppmv inlet oil and at 30 hours for 50 ppmv. Daily backwash of an operating nutshell filter is recommended.

Particulate Solids Filtration

Testing for particulate solids filtration was appended to the test program upon completion of the oil filtration work. Solids filtration was done with either BWS or PS media, and with or without oil in the feedstream. This program was not as comprehensive as that undertaken with the oil removal, and a summary of the work and results are provided.

Equipment Setup and Materials.

The same equipment and flow loop as previously described was used for all solids removal testing. IMSIL® microcrystalling silica (99% SiO_2) – grade A75 – was used for all testing. This grade has a mean particle size of 12 microns and a density of 2.65 g/cm³.

Test Variables and Analysis.

The test variables included media type (BWS or PS), brine concentration (0% or 10% salinity), media size (12/20 or 20/30), sand concentration (100 or 200 ppmw), and inlet oil concentration (50 or 100 ppmv). All tests were done at a flux of 13.5 gpm/ft². Outlet samples were analyzed for particle size analysis using a Multisizer™ 3 Coulter Counter® or Microtrac S3500 particle size analyzer, and for concentration using 0.2 µm Millipore™ filter samples.

Summary of Results.

A summary of results from the testing is listed in Table 2. These results are to be considered as a first-order estimation, and not definitive. Further testing with multiple runs is required.

Table 2 – Summary of Results from Particulate Solids Filtration

Media Type	Media Size	Brine Wt. %	Flux (gpm/ft²)	Sand Inlet Conc. (ppmw)	Oil Inlet Conc. (ppmv)	Separation Size, D_{98} (µm)
BWS	12/20	0%	13.5	100	0	7.1
BWS	12/20	10%	13.5	100	0	6.1
BWS	20/30	10%	13.5	100	0	4.6
PS	12/20	10%	13.5	100	0	4.4
BWS	12/20	10%	13.5	100	50	4.7

Based on the initial test data the following generalizations can be concluded;

- Increasing salinity, decreasing media size, and adding oil to the inlet stream all allow capture of finer sand particles
- PS has better capture efficiency than BWS
- The average solids capture size with a nutshell filter at a variety of conditions is nominally 5 microns

Further Work

The present work was undertaken at the eProcess Technologies flow loop lab in Butte, MT, USA. Sponsored students from Montana Tech University conducted all testing. Future work to be conducted on the nutshell filter flow loop - upon securing further student sponsorship - includes oil droplet size effects, oil filtration analysis with engineered nutshell or synthetic media, backwash testing, and treatment of the backwash waste stream.

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Nomenclature

BWS = *black walnut shell*
ppmv = *parts per million by volume*
PS = *pecan shell*
PVC = *polyvinyl chloride*
UV = *ultraviolet*

SI Metric Conversion Factors

°API141.5/(131.5+°API)			=	g/cm³
cP	x1.0	E-03	=	Pa·s
°F(°F-32)/1.8		E-01	=	°C
gpm/ft²	x6.790 233	E-04	=	(m³/s)/m² = m/s
inch	x2.54	E+00	=	cm
psi	x6.894 757	E+00	=	kPa

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