

CHARACTERIZATION OF DEEP BED FILTER MEDIA FOR OIL REMOVAL FROM PRODUCED WATER

C. Hank Rawlins, PhD, P.E., eProcess Technologies
Ariane E. Erickson, Montana Tech of the University of Montana

Abstract

Deep bed media filtration is used as a tertiary treatment step (after hydrocyclones and flotation cells) to remove fine oil droplets and solids from oilfield water before reinjection disposal or to remove particulate material from surface waters used for waterflood injection. Oil and solids injection requirements are typically <5 ppm concentration at <2 micron diameter. The preferred media for produced water filtration is walnut or pecan shell, however the use of this media has evolved by trial & error and the fundamental mechanisms that occur during separation and media cleaning have not been investigated. A project was initiated to fill in this knowledge gap by analyzing the physical, mechanical, chemical, and hydrodynamic properties of six types of granular filtration media (12/20 mesh size): garnet, silica, anthracite, pecan shell, English walnut shell, and black walnut shell. Comparison of bed packing, oil wetting, attrition resistance, particle strength, and fluidization has led to an understanding of the preferred filtration media for separation of fine oil droplets and solids.

Oilfield Deep Bed Media Filter Design and Use

Generally, deep bed media filters find use as the tertiary or polishing stage in the produced water treatment systems. In a typical treatment system, produced water exits the bulk separators (i.e. primary treater or free water knockout) with 1000-2000 ppm oil-in-water (OIW) content. The water will be near wellhead temperature, and from ambient to near wellhead pressure. For offshore installations the primary treatment step is a deoiler hydrocyclone, which reduces the OIW to the range of 29-100 ppm followed by a secondary treatment stage using a hydraulic flotation cell to meet overboard discharge requirements of <29 ppm OIW. Onshore facilities may use a corrugated plate interceptor (CPI) separator or American Petroleum Institute (API) separator as the primary treatment, possibly followed by mechanical flotation cells as the secondary treatment. Onshore tertiary treatment to <5 ppm OIW in cases of very stringent regulations or disposal

well injection is achieved with deep bed granular media filters. These filters often use walnut and/or pecan shells, and are generically called walnut shell filters or nut shell filters. In addition to produced water treatment, the walnut shell filter may be used to treat surface waters or brine well supply waters for solids removal before injection. In all cases the walnut shell filter serves as the final polishing step to reduce oil and solids concentration to very low levels and very fine particle size to protect the disposal or re-injection well.

The following design boundary conditions are given for a generic walnut shell filter used in an oilfield installation. These values can vary widely across various geographic regions, but serve as a nominal point for process design.

- Water flow rate: 5000-75000 BPD (145-2190 GPM) for a single vessel
- Water temperature: Ambient to 150°F
- Operating pressure: Atmospheric to 50 psig (3.4 bar)
- OIW content: <50 ppm inlet (from deoiler/flotation outlet), <5 ppm outlet (injection quality)
- Solids content: <25 ppm inlet, <5 ppm outlet (injection quality)
- Particle size inlet: <20 micron oil droplets, <10 micron solids
- Particle size outlet: <2 microns oil and solids
- Media: Walnut shell (primary), pecan shell (secondary)

A flow schematic of a vertical walnut shell filter is shown in Figure 1. While there are several patented or proprietary designs offered by walnut shell manufacturers, the flow schematic shown is for illustration purposes only. It is loosely based on US patent 4,826,609. Water with oil and solids enters near the top of the vessel through a distribution nozzle. The bulk flow of water is downward, where ideally a plug flow regime of the water flows through a bed of granular filter media. This media may be 36"-72" in depth and span the full diameter of the vessel. The nominal flux rate is 10-16 gpm/ft². The media is retained by a screen, which allows the cleaned water to flow through and report out the bottom of the vessel. Initial pressure drop across the bed is approximately 1 psi, and as the differential climbs to 15-20 psi, a backwash cycle is initiated. During

the backwash cycle a portion of the inlet flow is used to scrub the media by liquid agitation, while the bulk is diverted elsewhere. The backflow concentrate containing oil and solids empties through the backwash outlet.

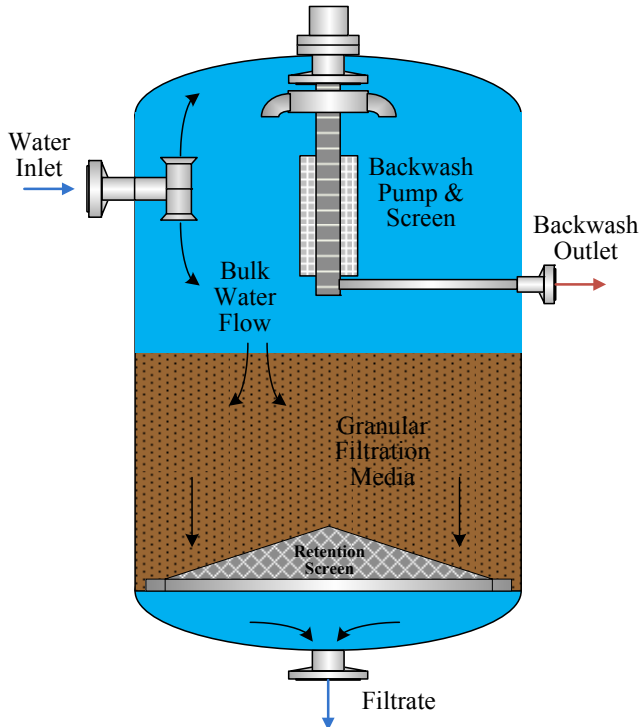


Figure 1. Schematic of flow pattern for deep bed granular media filter.

Walnut Shell as a Filter Media

The preferred media for oilfield produced water filtration is walnut or pecan shell, however, the use of this media has evolved much by trial & error. The fundamental mechanisms that occur during separation and backwashing in terms of oil-media, solids-media, and media-media interactions have not been thoroughly investigated. The amount of published information regarding oil removal from produced water by walnut shell media is negligible leading to an incomplete knowledge base for defining this unit process. The current investigation seeks to understand the oil-solids-media mechanisms occurring during produced water filtration in order to define a set of scientific principles for selecting and sizing a walnut shell filter in a wide variety of process condition.

More information is available from the patent lineage than from technical papers. The earliest published mention of walnut shells as a filter media comes from Hirs (U.S. patent 3,953,333) in 1975 which is a development from polymeric or mineral filter media. This use is further elucidated in U.S. patent 3,992,291 (Hirs, 1975) which

claims black walnut shells possess the capability of coalescing oil from contaminated water, however they have a relatively weak affinity for that oil thus are easily backwashed. The measured oil retention per unit volume of walnut shells was less than sand, anthracite, or PVC. Another key factor claimed in the patent, and subsequently in current use in marketing walnut shell filters, is that black walnut shells have a modulus of elasticity of 170,000 psi and relatively non-abrasive when compared to sand or coal. The determination of the modulus of elasticity value, or its connection to separation or backwash performance has never been clarified. Many other patents have been filed on specific vessel or system designs to simplify operation and backwash; however these two patents originate the technology.

Several investigators have studied the operation of walnut shell filters with mixed results. The earliest technical papers are from Rahman et al.^{1,2} and states the mechanism for filtration is direct interception or adsorption. Coal based filters are too oil wet and require high backwash rates, however walnut shells are water wet (hydrophilic) which require less backwash agitation. However he then states that walnut shells have high oil adsorption characteristics (oleophilic) which is contradictory. Kenawy et al.³ states that oil coalesces on the grains and accumulates in the bed becoming entrapped within the media interstices. Evans et al.⁴ mentions that crushed walnut or pecan as capable of achieving 5 mg/l oil-in-water concentration, but does not detail the mechanisms involved. Mantilla et al.⁵ details a remote land-based produced water treatment system using a walnut shell filter, and provides data showing an removal efficiency of 21 mg/l solids and 5 mg/l oil down to 4 mg/l solids and 1 mg/l oil. He also mentions that barium sulfate is being removed by the filter. Blumenschein et al.⁶ details a pilot scale study of treating steel mill effluent and shows that the filter can handle 26 gpm/ft² with a solids loading rate of 11 lb/ft² at 85% removal efficiency, however oil removal efficiency was not detailed. Lastly, Srinivasan et al.⁷ tested the oil sorption capacity of black walnut shells media using a variety of industrial oils. They found that oil sorption decreased with increasing oil viscosity and interfacial tension, with overall sorption capacities of 0.5-0.8 g/g. They also state that walnut shells have excellent surface characteristics for coalescing and filtration and superior resilience to attrition.

Only one of the seven papers mentions any technical depth on the properties of walnut shell media, and the rest provide anecdotal results of case studies. This leads to the conclusion that the fundamental mechanism that makes walnut shell as the preferred produced water filtration media is not understood. The two statements made repeatedly by the papers, are that walnut shells are “water wet” and have “high oil adsorption/sorption capabilities.” The two statements are essentially in conflict. Water wet

means that walnut shells are hydrophilic, which in turn makes the oleophobic. An oleophobic material will then repel oil, and not be able to capture that oil by adsorption. While adsorption can have physical or chemical form (physisorption or chemisorption) in either case, oleophobicity will impart a repulsive force to prevent coating of the walnut shell by oil.

By anecdotal evidence it is known that walnut shell media removes oil from produced water, so some capture mechanism(s) must be present. As oil is typically in liquid form, it is hypothesized that purely mechanical capture by deep bed filtration is insufficient. The oil must be captured by the surface of the walnut shell media by weak physisorption or chemisorption, capillary force between or within nut shell particles, or by some degree of oleophilicity. The exact mechanism requires further investigation. The degree to which walnut shell media can capture dissolved as well as suspended hydrocarbons also deserves investigation.

In addition to oil capture, walnut shell media is effective at suspended solids removal. This follows traditional (deep) media filtration theory of particle bridging. The degree to which walnut shell as a media is better than sand or diatomaceous earth is not quantified, or by what other mechanisms improves the capture. Anecdotal evidence also suggests that walnut shells play a role in removal of dissolved solids, including heavy metal contaminants (i.e. they have been tested in acid mine waste waters). The degree to which they serve in this capacity also deserves investigation.

Information regarding detailed mechanical properties of walnut shells was not forthcoming. One paper states walnut shells have a hardness of 3 on the Mohs scale, which puts them in league with calcite and copper. The common statement regarding walnut shell media is that they have a high modulus of elasticity which gives them a high resilience to attrition during the backwash cycle. The modulus of elasticity is a poor mechanical property for use in attrition or abrasion resistance. Brass, copper, and titanium have a modulus of elasticity an order of magnitude higher than the value stated for walnut shells (i.e. 1,700,000 psi vs. 170,000 psi), but these materials have very poor resistance to wear, abrasion, or attrition. Teflon (PTFE) has approximately 2.5 times less modulus of elasticity (75,000 psi), but it has excellent abrasion and attrition resistance. A combination of hardness and toughness provides a better indication of attrition resistance, and comparative values can be measured from a self abrasion index test. This value for walnut shell, pecan shell, and other common media should be accurately quantified to provide the end user with more appropriate selection criteria.

Characterization of Granular Filter Media

As the granular media comprise the main operating constituent of the filter, the first step in understanding the performance of this unit process is to characterize the media being used. The main focus of this investigation is to analyze common industrial granular filtration media to determine the physical, surface chemistry, mechanical, and hydrodynamic properties. These properties can be used to rank oil and solids filtration effectiveness, flow path, maintenance, media life, and overall filter design.

Six media materials were identified as common to deep bed media filtration for water, and were procured from various vendors. English walnut and pecan shell was procured from a Texas reseller, black walnut from a Missouri producer, silica from a construction retailer, and garnet and anthracite from a local Butte water treatment company. All materials were specified with a target mesh size of 12/20 (1700 microns x 850 microns). The English walnut, black walnut, pecan, and silica were delivered at that specification; however garnet was only available at 14 mesh (1420 microns) and anthracite at 10 mesh (2000 microns). All material was received in bulk form and packed in bags (paper bags for English walnut, black walnut, pecan, and silica, solid plastic bag for anthracite, and woven plastic mesh bag for garnet). Characterization and analysis was done on as-received material unless otherwise stated. Each test was conducted with three specimens taken from the media received, and the results presented are the average of the measured values.

Physical Properties of Granular Filter Media

Physical property determination was undertaken to understand the basic structure and properties of the various granular media. The properties measured include weight percent moisture loss, particle size distribution, apparent density, true density, bulk density, specific surface area, pore size distribution, optical microscopy, and scanning electron microscopy. Data from key analyses are provided.

Particle Size Distribution

A grab sample of approximately 250 grams was selected from each media bag. The initial weight was recorded, and the material sieved on a nested sieve stack for three minutes. Eight inch Tyler mesh screens of size 10, 12, 14, 16, 18, 20, and pan were used. The actual micron size from each sieve was recorded. After sieving the individual weight fraction from each sieve and the total sample weight was recorded to determine % loss. The combined 10-20 mesh fractions, -20 mesh fraction, and +10 mesh fraction were bagged separately for subsequent testing or analysis.

All weights were recorded to 0.00 grams accuracy. The results are shown in Figure 2.

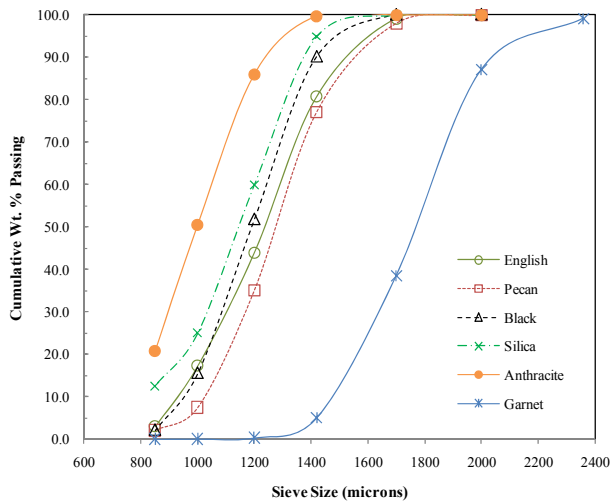


Figure 2. Particle size distribution of six media types specified at 12/20 mesh.

The biological media (English walnut, black walnut, and pecan) had similar size distributions with <2-3% of the material smaller than 20 mesh. The D_{50} values varied from 1200-1300 microns and the slope (D_{90}/D_{10}) values were similar. Silica had a similar slope, but the amount of fines (<20 mesh) was much larger at 12.5%, which decreased the D_{50} to ~1100 microns. Garnet had the lowest amount of fines (0.01%); however the D_{50} value is ~1800 microns, which is much larger than all other material. Anthracite was at the opposite end, with the smallest D_{50} at ~1000 microns, and a high amount of fines near 21%. For comparison of subsequent analysis, silica is the media with the closest particle size distribution to the biological media.

Apparent and True Density

An approximate 5 gram specimen from the 10-20 mesh material from the particle size analysis was selected and weighed. The apparent density was determined using a glass immersion pycnometer with 18 MΩ water as the immersion media. A second approximate 5 gram specimen from the 10-20 mesh material was selected, pulverized in a ring & puck mill to 100% passing 200 mesh, and the true density determined by the same method. All weights were recorded to 0.0000 grams accuracy. True density is a measure of the material density without any voids, while apparent density is the density of the granular media as-received with voids included. The results are shown in Table 1.

The apparent density is used to determine how individual particles will behave in water. The English walnut, black walnut, and pecan shells have similar

apparent density, which is lower than the inorganic materials. Anthracite is a bit higher, followed by silica and garnet. Measurement of true density provides an indication of the void space in the granular material. Of the biological media, black walnut has about half the void space compared to English walnut and pecan shell. Silica and garnet have little to no void space indicating a near fully dense material.

Table 1. Summarized Results for Apparent and True Density Tests

Media	Specific Gravity		Void %
	Apparent	True	
English	1.34	1.44	7.1
Pecan	1.34	1.46	8.3
Black	1.39	1.45	3.8
Silica	2.63	2.64	0.4
Garnet	4.14	4.17	0.6
Anthracite	1.60	1.67	4.5

Bulk Density

A glass 1000 ml graduated cylinder was selected and the weight recorded. Approximately 100 ml of media material (grab sample) was added and the cylinder tapped 10 times on the flat portion of the cylinder. The process was repeated with 100 ml of material and 10 taps until 900 ml was reached. The weight of the cylinder with dry material was recorded to determine dry bulk density and open volume % dry (using apparent density). Using a large burette, tap water was added until reaching the level of the media, and the amount of water (ml) was recorded along with the weight of the cylinder (media plus water) and resulting level of water/media mixture (height may change due to absorption swelling). The cylinder was then inverted onto a 600 micron (U.S. 30 mesh) sieve, and the media and water removed. The free water was allowed to drain through the sieve. The net wet weight of the media was recorded to determine the water retention quotient (i.e. adsorbed + absorbed water in g water/g media). All weights were recorded to 0.00 grams accuracy. The results are shown in Table 2.

The dry bulk density followed the same trend as apparent density, with the biological materials exhibiting the lowest values, and garnet the highest values. The dry open volume % though was nearly the same, regardless of material type. This is because of the narrow size distribution leading to similar packing efficiency. The water retention quotient is a combination of absorbed water into the pores of the granular media plus adsorbed water on the surface. Garnet, followed by silica, had the lowest water retention due to low surface absorption. Of the biological materials the water retention quotient approximately followed the % void space (Table 1) with black walnut having the lowest amount of water absorbed.

Table 2. Summarized Results for Bulk Density Tests

Media	Bulk Density (kg/m ³)	App. Density (kg/m ³)	Open Vol. % Dry	Water Ret. (g/g)
English	772	1336	42.2	0.50
Pecan	729	1338	45.6	0.48
Black	832	1392	40.2	0.40
Silica	1570	2631	40.3	0.15
Garnet	2496	4144	39.8	0.06
Anthracite	902	1600	43.6	0.33

Specific Surface Area

A sample (~5 grams) of 10-20 mesh media was selected and analyzed by BET surface area analysis using a Quantachrome Monosorb MS-13 surface area analyzer. This method measures the quantity of gas adsorbed on a solid surface. The sample is put into a quartz sample cell that is then submerged into liquid nitrogen. An adsorbate and inert carrier gas (30% N₂ + 70% He) are passed over the sample and the change in thermal conductivity of the flowing mixture is used to determine the partial pressure of the adsorbate and ultimately the weight of adsorbate required to cover the surface with one molecular layer. From this method the total surface area (m²) is determined and using the weight of the sample the SSA (specific surface area, m²/g) is determined. All weights are recorded to 0.0000 gram accuracy. The results are shown in Figure 3.

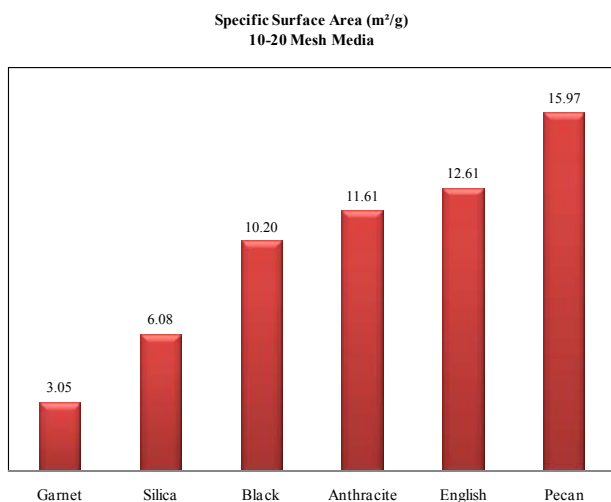


Figure 3. Specific surface area (m²/g) of six filter media with 10-20 mesh size.

The specific surface area measurement shows that garnet has the least surface area at 3 m²/g, while silica has twice that value. That is because silica has more cracks and surface imperfections (seen in optical microscopy) and a more irregular shape. Black walnut shell has the lowest SSA of the biological materials which corresponds to

smoothest surface. Pecan has the highest value of SSA showing that it has the most irregular surface features.

Surface Chemistry Properties of Granular Filter Media

Both surface chemistry and bulk chemistry of the granular filtration media were undertaken. As filtration of suspended solids and oils is primarily a mechanical phenomenon, the bulk chemistry properties is not critical for comparison, but was undertaken to understand the structure of the inorganic materials. X-ray diffraction (XRD) showed the silica to be composed primarily of quartz and the garnet to be composed primarily of almandine (Fe₃Al₂(SiO₄)₃). X-ray fluorescence (XRF) showed minor amounts of aluminum and potassium in the silica and magnesium and manganese in the garnet. Combustion analysis showed the anthracite to contain 79% carbon with 0.8% sulfur, while the three biological materials all contained 36-38% carbon and 0.08% sulfur.

Though filtration of suspended solids and oils is primarily due to inertia and blocking, the surface chemistry as related to oil adhesion is important for removal of the oil by fluidization and backwashing. Surface chemistry analysis included sessile drop test and oil retention quotient.

Sessile Drop Test

Approximately 5 grams of each media material was mounted in a two-part hardening epoxy, then sanded and polished with 600 grit paper using a non-aqueous lubricant (ethylene glycol). The polished surface was cleaned with alcohol and allowed to dry overnight at ambient conditions. Each mount was visually inspected to confirm the presence of exposed full diameter particles. The epoxy mount was placed on a Ramé-Hart contact angle goniometer which enables magnified visualization of a static (sessile) drop and determination of contact angle. The contact angle indicates the degree of wetting by the fluid in the drop. A 0° degree contact angle indicates fully wet, while 180° indicates no wetting. Using a syringe with 33 gauge needle a single drop of 18 MΩ water, n-Dodecane (API 58), and Mobil Velocite #6 (API 35) were each put onto different exposed particles. The contact angle for each fluid type was measured and recorded. The results are shown in Table 3.

All media showed a finite contact angle with water, with pecan the lowest (most wet) and English walnut shell the highest (least wet). However, the range of values did not vary significantly, and all materials were at least partially wet by water. For both oils tested, all materials exhibited a zero degree contact angle, showing that they are all completely wetted by oil.

Table 3. Contact Angle for Water and Two Oils with Filtration Media

Media	18 MΩ Water	Dodecane	Mobil Velocite #6
Silica	35	0	0
Garnet	35	0	0
Anthracite	44	0	0
Pecan	23	0	0
English	51	0	0
Black	31	0	0

Oil Retention Quotient

Three test oils were used in the oil retention quotient test; 99% dodecane, Mobil Velocite #6, and Tectyl 802A. These oils represent light, medium, and heavy API oil, respectively. These oils were characterized for density, viscosity, and surface tension (air-oil). Density measurement was accomplished using a quartz pycnometer, viscosity measured with a Brookfield DV-II+ Pro viscometer with appropriate spindle, and surface tension using a SITA t60 bubble pressure tensiometer. Table 4 shows the results of the fluid characterization.

Table 4. Fluid Properties of the Three Test Oils

Oil	Density @ 22°C (g/cm³)	API Gravity	Visc. @ 22°C (cP)	Surf. Tens. (dyne/cm)
Dodecane	0.747	57.9	1.3	38.2
Velocite	0.848	35.4	15.9	29.7
Tectyl	0.916	23.0	292.4	32.5

For dry media testing, approximately 25 grams of 10-20 mesh sized material was weighed out and placed into a clean pan. The media was covered completely with the test oil for 20 minutes. The mixture was then poured on a 25 mesh screen, spread into a monolayer, and allowed to stand for 5 minutes while oil drains by gravity. The oily media was removed from the screen and weighed on filter paper. The procedure was repeated for the three test oils. For wet media testing, approximately 25 grams of the 10-20 mesh material was weighed out, placed in a beaker, and completely submerged with tap water for one hour. The excess water was removed and the media filtered through a vacuum filter for five minutes to remove any free water. The wet media was transferred onto a dry piece of filter paper to record the saturated media weight. The wet media was then subjected to the same oil immersion test as the dry media. All weights were recorded to 0.00 grams accuracy. The results are shown in Figures 4 and 5.

The three test oils were selected to provide a range of API densities. Dodecane is a light condensate with 58 API gravity, Mobil Velocite #6 is a medium weight oil with 35 API gravity, while Tectyl is a heavy oil with 23 API gravity. The dry silica and garnet media showed

approximately one-half the oil retention compared to the biological media. Of the biological media, pecan shell had the highest oil retention overall. If oil retention is indicative of oil filtration capacity, pecan shell holds the most oil. Wetting the media prior to exposure to oil decreases the amount of oil retained for the light and medium oils, but had little effect on the heavy oil. Overall, garnet had the lowest oil retention quotient, while pecan had the highest.

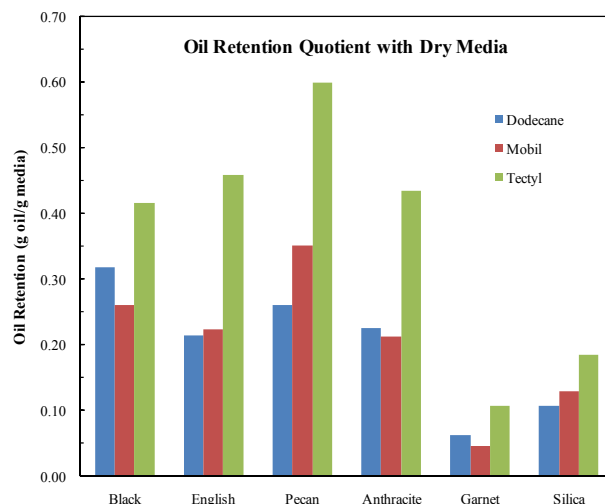


Figure 4. Oil retention quotient of three test oils using dry media.

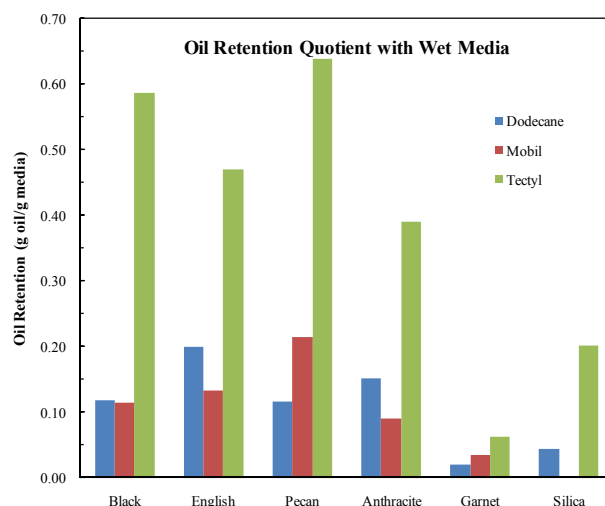


Figure 5. Oil retention quotient of three test oils using wet media.

Mechanical Properties of Granular Filter Media

The mechanical properties were measured mainly to understand the effect of physical handling of the media

during agitation (fluidization + backwash). Mechanical property analysis included self attrition index (wet) and crush strength.

Self Attrition Index (Wet)

Approximately 300 ml of material was dried and sieved at Tyler mesh size 10, 20, and pan in 150 ml lots. The +10 mesh and -20 mesh material was discarded. 250 ml of the material (non-compacted) was measured into a graduated cylinder, the weight determined, and the material wet sieved at 20 mesh to ensure all -20 mesh material was washed through. The -20 mesh material was captured, dried, and weighed. The excess water was drained from the +20 mesh material, and then the material was added to a 1000 ml Nalgene bottle. 250 ml of tap water was added to the bottle, the lid put on, and the lid taped. The bottle was agitated on a Thermolyne shaker table at 325 RPM for 1 hour. The slurry was wet sieved at 20 mesh and the -20 mesh material was captured, dried, and weighed. The +20 mesh fraction was returned to the Nalgene bottle, 250 ml water added, and the bottle sealed. The shaking and sieving procedure was repeated at +9 hours, +20 hours, and +30 hours. After last run, the slurry was wet sieved at 20 mesh and the +/- 20 mesh material captured, dried, and weighed. The -20 and + 20 mesh dried fractions were saved for optical analysis. All weights were recorded to 0.000 grams accuracy. The results are shown in Figures 6 and 7.

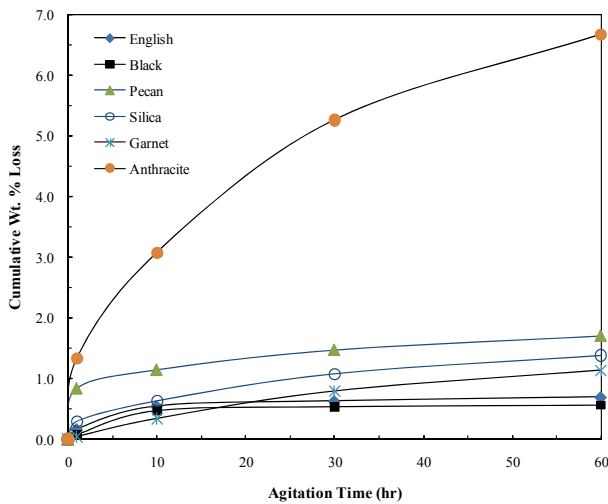


Figure 6. Results from self-attrition test of six granular media.

Self-attrition index is a representation of the particle-particle interaction that occurs during fluidization and backwash. Ignoring interaction with the vessel walls, most of the particle wear arises from particle-to-particle impact during these events. This test was to provide an index of the

wear rate from particle-particle interaction (self-attrition). The index is not a scaling factor to determine exact material life in a deep bed media filter but represents a ranking of material to compare wear life. Factors that affect self-attrition include density (which drives inertial forces), hardness, and toughness. Anthracite exhibited the highest wear at all times, and continued to show wear at 60 hours. Silica and garnet showed about 20% of the wear of anthracite, and the wear continued even at 60 hours. Of the biological material pecan had the highest wear, at more than three times the walnut shell media, and continued to wear at 60 hours. English and black walnut shell showed a very similar trend with English wearing about 15% more than black at all times. The key trend is that both walnut shell media reached a plateau at +10 hours and showed little to no wear after that time. Overall black walnut shell showed the greatest resistance to self-attrition both in magnitude and trend.

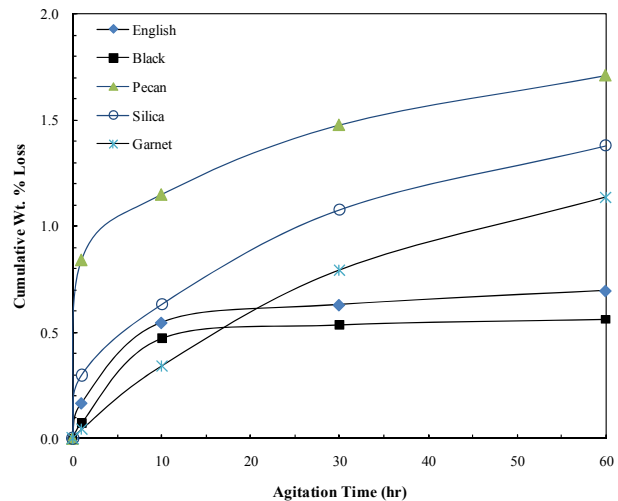


Figure 7. Results from self-attrition test of six granular media (less anthracite).

Hydrodynamic Properties of Granular Filter Media

The hydrodynamic properties were measured to determine the flux rate parameters for filtration and fluidization, including the pressure/flow relationship.

Fluidization

For the silica and garnet tests, approximately 48 inches of dry media was measured out. For the anthracite, black walnut, English walnut, and pecan, approximately 41 inches of dry media was measured out and soaked in tap water for one hour. All media was then added to the column, fluidized with tap water, and allowed to settle to determine

the media's initial wet height. Media height and inlet pressure was recorded at each target flow (flux) rate.

Figure 8 shows a schematic of the test apparatus built and used for fluidization flux testing. The main column was built of 4 inch schedule 40 clear PVC and uses 24 mesh and 4 mesh (304 SS) screens to hold the media. Water supply and drains from the column were through ¾ inch diameter garden hose, while all 1 inch lines were PVC. The flow control valve was a manual globe valve (class 125, 1 inch), the pressure indicator was 0-30 psi bourdon tube type, and the flow indicator was a digital flowmeter/totalizer with 1 inch nylon body and 0.3-3 GPM capacity.

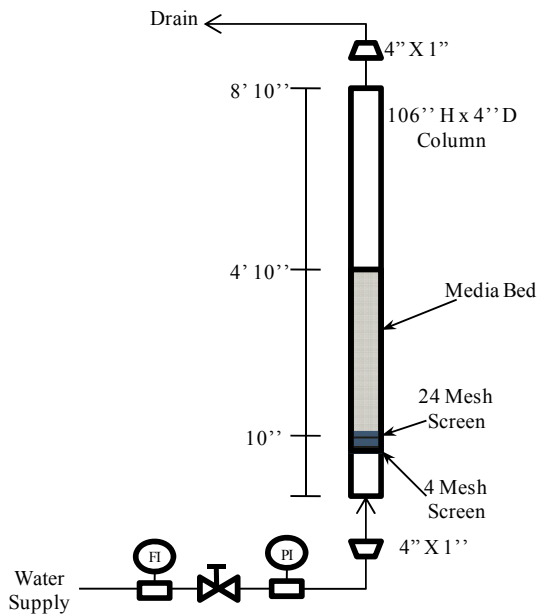


Figure 8. Flow schematic of test apparatus for fluidization measurement of granular media.

Figure 9 shows the fluidization flux versus bed height increase (%), and Figure 10 shows the fluidization flux pressure. Fluidization flux is a key parameter for designing a backwashable deep bed media filter. The 4 inch column was meant to simulate a small cross-sectional area of a larger diameter filter. The wall effects/friction will be much more pronounced in a 4 inch column, but the data acquired should provide sufficient design parameters for fluidization of the media. Figure 9 shows that fluidization, as determined by bed height increase, is a function of media density. Fluidization is defined at the point where the bed begins to increase in height due to upward flow of the fluid, and for quantitative comparison a 1% increase in bed height is defined as the onset of fluidization. Garnet (4.14 apparent s.g.) required a flux of ~26 gpm/ft² to fluidize, while silica (2.63 apparent s.g.) required ~12 gpm/ft². This is nearly a directly proportional correlation. The next lowest was

anthracite (~4.7 gpm/ft² flux), followed by black walnut (~4.5 gpm/ft²), while pecan and English walnut were nearly identical (~3 gpm/ft²). The fluidization flux requirement for the biological media is significantly less than the mineral-based media, which can be directly attributed to a lower cost (energy) design.

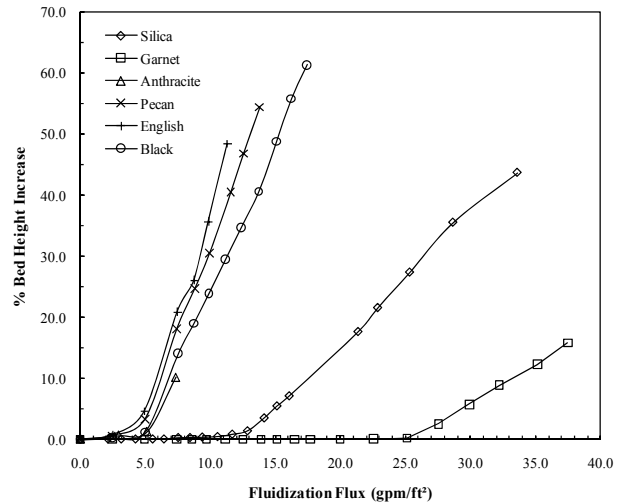


Figure 9. Fluidization flux versus % bed height increase for six media types.

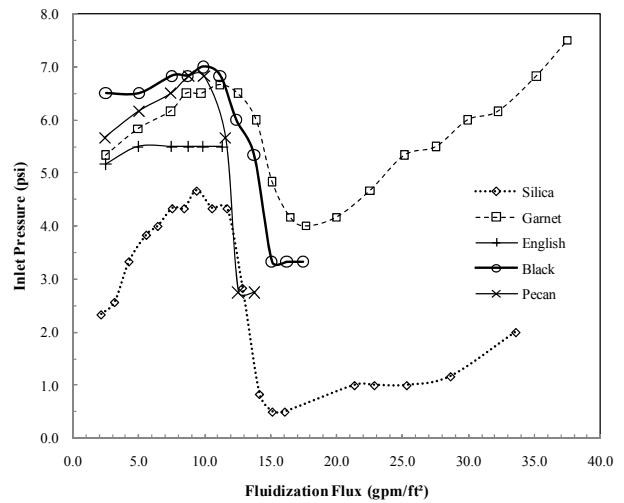


Figure 10. Fluidization flux versus inlet (back) pressure for five media types.

The pressure requirement to fluidize, shown in Figure 10, is based on static head of the fluid, resistance head of the packed bed (due to particle interlocking), and friction head due to flow. The static head of the fluid will be a function of the fluid level height in the vessel, and in this case was ~3.8 psi for a 106 inch column full of water. All the media followed a similar trend of 5.0-6.5 psi at onset of

flux, which slowly increased to a maximum at 10 gpm/ft² flux. The silica test had a siphon vacuum in the overflow line, thus the absolute values were artificially low. It took ~10 gpm/ft² to overcome bed resistance, then the pressure dropped to a minimum at ~15 gpm/ft² as the bed broke free. The increase in pressure after this point was due to friction of the slurry flow in the column (pipe). It is interesting to note that the required pressure to break the bed resistance is nearly constant however the bed height is a function of media density.

Pressure/Flow Relationship

The pressure required to achieve a specific flux rate is a key sizing parameter of a deep bed media filter. The flux rate versus pressure drop for the six granular media was measured using a 4 inch column. Each media was pre-soaked in water for at least one hour then a specific height measured into the 4 inch diameter column. Each media was tested at 24 inch, 48 inch, and 72 inch bed height. Water was introduced into the top of the column at a measured flow rate, and the resulting pressure drop determined. For each test the pressure drop was measured as the head of water above the media bed. As the media was suspended on a screen to allow free space below the bed (e.g. no back pressure from piping), the height of water measured above the bed will give the pressure drop across the specific bed depth.

Figure 11 shows a schematic of the test apparatus built and used for measuring the pressure/flow relationship. The main column was built of 4 inch schedule 40 clear PVC and uses 24 mesh and 4 mesh (304 SS) screens to hold the media. Water supply and drains from the column were through 3/4 inch garden hose, while all 1 inch lines were gray PVC. The flow control valve was a manual globe valve (class 125, 1 inch) and the flow indicator was a digital flowmeter/totalizer with 1 inch nylon body and 0.3-3 GPM capacity.

Figure 12 shows the water flux (gpm/ft²) versus pressure drop (psi) for six media with a nominal 72 inch bed height. The pressure drop for all media and all flow rates tested was very low. Within the normal flux range experienced (12-16 gpm/ft²) the pressure drop for all media was <1 psi. Silica and anthracite exhibited a slightly higher pressure drop because of particle shape and size. These two media types were flat and angular and had the smallest particle size, and when packed together in a bed increased the resistance to flow. The three biological media (English walnut, black walnut, and pecan) all exhibited very similar pressure drop characteristics. The pressure drop for all media showed a linear relationship with flux.

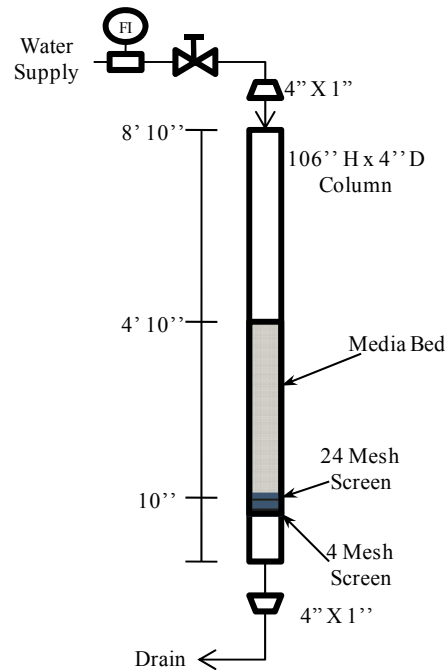


Figure 11. Flow schematic of test apparatus for flux versus pressure drop measurement of granular media.

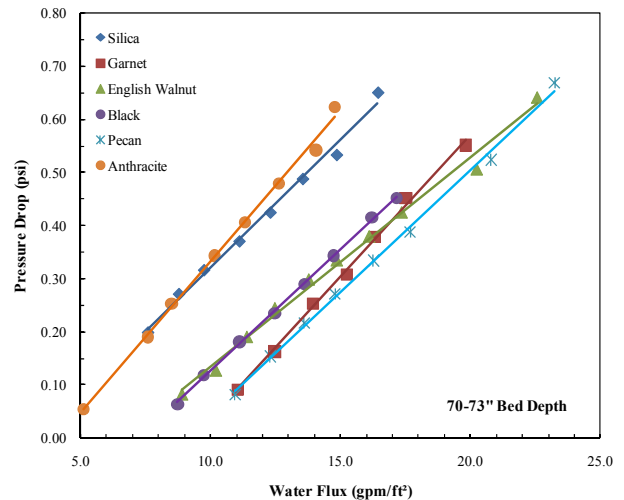


Figure 12. Pressure drop versus filtration flux for all six media types.

Recommendations for Granular Filter Media Selection

Assuming all media tested will capture solid particles at a similar efficiency (which is a function of filter particle inertia and media particle packing) and that the capture of oil droplets is a function of surface chemistry (i.e. oil wetting), then pecan shell would have the highest filtration

effectiveness and garnet the least. Black walnut and English walnut had slightly less oil wetting quotient than pecan.

In terms of fluidization flux, the amount of water required to expand the media bed is a function of media apparent density. English walnut, followed closely by pecan and black walnut, requires the least amount of water to fluidize (3-5 gpm/ft²), while garnet requires the most (>25 gpm/ft²). When backwashing to clean the media, the particles are agitated violently and are subject to inter-particle impact which is termed self-attrition. The most self-attrition resistant material is black walnut followed closely by English walnut, which both reached a plateau of 0.5% loss after 10 hours of self-attrition (simulates 30 days of normal backwash cycle use). All other media continues to lose weight due to self-attrition, even after 60 hours of testing (~180 days of use). The pressure drop through all new media was <1 psi at all filtration flux rates expected in field use. This pressure drop will increase as the filtration pores plug with particulate material.

From the key parameters measured, which include oil wetting, fluidization flux, self-attrition resistance, and pressure drop, the best overall material as a single media is black walnut. It has high oil wetting, low fluidization flux, and low loss due to abrasion. The filtration efficiency in many applications can be improved however by adding pecan shell to the black walnut shell to increase the oil removal capability. Pecan shell does have a slightly higher media wear rate compared to black walnut shell, but the increase in oil removal efficiency should offset this factor. The ratio of walnut:pecan shell mix, both in terms of quantity and size distribution, as well as the homogeneity and structure of the mixture, is the subject of a subsequent research project.

References

1. Rahman, S.S., A.M. Ezzat. "Recent advances in the filtration of oil field brines and injection waters." *Presented at the Eleventh Annual Energy-Sources Technology Conference and Exhibition, Drilling and Production Technology Symposium*, ASME, 1988, 43-48.
2. Rahman, S.S. "Evaluation of filtering efficiency of walnut granules as deep-bed filter media." *Journal of Petroleum Science & Engineering* 7.3 (1992): 239-246.
3. Kenawy, F.A., M.E. Kandil. "Comparative evaluation between a modified CFP separator and all other available oil-water separation techniques." *SPE Paper 46817 Presented at the 1998 SPE International Conference on Health, Safety, and Environment in Oil and Gas Exploration and Production*, Caracas, Venezuela, 7-10 June 1998.
4. Evans, P., K. Robinson. "Produced water management-reservoir and facilities engineering aspects." *SPE Paper 53245 Presented at the 1999 SPE Middle East Oil Show*, Bahrain, 20-23 February 1999.
5. Mantilla, I., P. Quintero. "Comprehensive system for treatment and injection of produced water: field cases." *SPE Paper 63169 Presented at the 2000 SPE Annual Technical Conference and Exhibition*, Dallas, TX, 1-4 October 2000.
6. Blumenschein, C.D. "Walnut shell filtration for oil and solids removal from steel mill recycle systems." *AISE Steel Technology* 78.4 (2001): 33-37.
7. Srinivasan, A., T. Viraraghavan. "Removal of oil by walnut shell media." *Bioresource Technology* 99 (2008): 8217-8220.