Water-in-oil coalescence using glass fiber filters augmented with polymer nanofibers

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Abstract

Coalescence filtration is very effective for the separation of secondary emulsions that contain water droplets with diameters less than 50 μm. The factors that control the performance of coalescer filter media are fiber size and wettability. High wettability materials for water-in-oil dispersion promote coalescence. In this paper, new experimental results investigating the of performance non-woven filter media glass fiber augmented with polymer nanofiber are presented in relation to the relevant parameters (wettability, filter depth, flow velocity, and filter materials). A decrease in fiber size improves the overall separation efficiency of the process. (A blend of glass fibers with nanofibers having diameters of around 150 nanometers showed improvement of separation efficiency.) High wettability materials for water-in-oil dispersion promote coalescence. Lower flow rates perform better than higher flow rates. The intrinsic coalescence efficiency is observed to decrease with an increase in filter media thickness, though the overall separation improves with thickness.

1. Introduction

In recent years water-in-oil emulsion separation has received greater attention in the petroleum production. In many applications, dispersions of water drop sizes of less than 100 μm are very difficult to separate. The coalescence filter is an economical and effective for separation of secondary dispersions. There are three main steps of the coalescence process. First, the fibrous bed captures water droplets. Second, the collected water phase migrates through the bed and the droplets coalesce on the fibers. Third, the enlarged droplets are released from the fiber surfaces and are carried out of the medium by the oil flow. Coalescence performance depends on flow rate, bed depth, fiber surface properties, and drop size. Filter media with larger fiber contact areas generally perform better than media with lesser surface areas.

The wetting behavior of the water-in-oil or oil-in-water emulsion is considered important in determining the performance of the coalescence filter. Continuous, unwoven, woven, or chopped fibers of glass or polymers such as nylon and polyacrylonitrile (PAN) have increasingly important applications. Good wetting of fibers by the oil and water mixture and controlled fiber mixture interactions are necessary to obtain optimal composite mechanical properties (Schrader & Loeb, 1992). Many attempts have been made to determine the effect of the wettability on the coalescence process. However, investigators have different opinions of the effect of the wettability. Voyutskii et al. (1953) observed that an intermediate wettability gave the most effective separation, and concluded that for the best performance the filter should be sufficiently water-wetted to coalesce the water, but not so saturated as to produce excessive clogging by accumulated water. They found that the fiber contact surface is more important than pore size. Hazlett (1969) reported that the water droplets in water-in-oil emulsions must displace the oil film from the wet fiber for attachment to be effective. A water droplet easily displaces oil on hydrophilic surfaces. The displacement on a low energy surface such as polyethylene or Teflon should be considerably less. However, Clayfield et al., (1984) showed that coalescence efficiency is not related to the wetting property of the dispersed phase on the coalescence. They tested coalescence efficiency of seven different coated glass fibers and measured the critical surface tension.

There are three measures of filter performance that are of interest in this paper, and are summarized in Table 1. Capture efficiency, E, is the most commonly used measure, and in simply the ratio of particle captured by a filter to the particle challenging the filter. The capture efficiency is often related to the individual fiber efficiency and to the filter coefficient (Brown, 1993).

Coalescing filter media also have the mechanisms of droplet drainage. Sherony and Kintner (1971) define the overall coalescence efficiency, η_c, as a combination of capture efficiency and the fraction of collisions between drops that result in coalescence. The overall coalescence efficiency is related to the capture efficiency by

$$1-E=\exp\left\{-\frac{3(1-\varepsilon)S\left(1+\frac{d_u}{d_f}\right)}{4d_f(1-S)}\eta_c L\right\} \tag{1}$$

where

E = separation efficiency
ε = void fraction of the bed;
S = average saturation;
d_u = upstream average particle size (cm);
d_f = diameter of fiber (cm);
η_c = the overall coalescence efficiency; and
L = bed length (cm)

The overall coalescence efficiency, η_c can be determined from experimental values of E, ε, d_u, d_f and L.

The Third way to characterize the filter performance is with the quality factor, QF. The quality factor is defined by (Brown, 1993)

$$QF=\frac{-\ln\left(\frac{C_{out}}{C_{in}}\right)}{\Delta P} \tag{2}$$

For a monodispersed particle size and uniform filter properties, the logarithm of the penetration, $\ln(C_{out}/C_{in})$ is proportional to filter thickness. Similarly, the pressure drop is proportional to filter thickness through Darcy's law. Hence, the QF does depend upon thickness.

Flow rate is an important factor in water-in-oil dispersion flow as it controls the capture mechanism and capture probability of droplets, and the distribution of the dispersed phase. Many attempts have been made to determine the critical velocity defined as the velocity at which the effluent concentration of the dispersed phase exceeds a fixed value (Sareen et al., 1966, Spielman 1968, Fahim & Akbar, 1984, Othman et al., 1988 and Šećerov Sokolović et al., 1997). Many results show that the separation efficiency is influenced by the filter bed depth. Hazlett (1969) finds that an increased bed depth promotes coalescence, but there is a critical length beyond which the bed performance is not improved. Akber & Othman (1989) show a relationship between coalescer saturation profile and pressure drop and the optimum bed depths and the critical velocity.

Low flow rates, high operating temperatures, and increased coalescer bed depth increase coalescence filtration efficiency. A decrease in the fiber diameter also improves the overall efficiency of the filtration process. One of the important factors is surface area of the fiber.

Electrospinning provides a way to produce long polymer fibers with diameters in the range of 10-500 nm (Gibson et al., 2001). In our laboratory we routinely electrospin fibers from about 30nm up to a few microns in diameters. The electrospinning process is driven by the electrical forces on free charges on the surface or inside a polymeric liquid. When the electric field reaches a critical value at which the repulsive electric force overcomes the surface tension force, a charged jet of the solution is ejected from the tip of a cone protruding from a liquid drop of the polymer. As the jet stretches and elongates in the air, the solvent evaporates, leaving behind a charged polymer fiber that lays itself randomly on a collecting metal screen. Thus, continuous fibers are produced to form a non-woven

fabric (Doshi & Reneker, 1995). Non-woven fibrous materials composed of electrospun fibers have a large surface area and small pore size compared to commercial textiles, making them excellent materials for use in filtration applications (Deitzel et al., 2001). Figure 1 shows the relation between fiber diameters and surface area.

The objective of this work is to investigate the coalescence separation of water droplets in a water-in-oil mixture using glass fiber media and glass fiber plus electrospun nanofiber media. The goal is to experimentally study the relationship between pressure drop across the bed and separation efficiency. The results are analyzed using separation efficiency (E), quality factor (QF), and coalescence efficiency (η_c).

2. Experiment and Operating Procedure

A schematic diagram of the experimental apparatus is shown in Figure 2. A mixture of water-in-oil was used in the experiments, in which deionized water was dispersed in oil (visco 1487, diesel fluid). The visco 1487 (specific gravity = 0.83) is pumped from tank 1 by a peristaltic pump, at a constant flow rate through a mixing pipe (where the water drops are mixed with the oil), through the filter sample and into a settling tank and reservoir. The flow rate is controlled by selection of tube diameter size for the peristaltic pump. Flow rates for the peristaltic pump (Masterflex, Model L/S EW-07543-60) are from 100 ml/min to 290 ml/min.

The water-in-oil emulsion is produced in the mixing pipe. The water is pumped by a syringe pump through a hypodermic needle into the middle of the mixing pipe through which the oil flows. The mixing pipe is a Plexiglas tube with a 1 *mm* inside diameter and 11 mm outside diameter. The mixing pipe is inserted into the flexible tube between the pump and the sample holder. A very fine water-in-oil emulsion, in which 98 % of water droplets are less than 30 microns, is produced at the outlet of the hypodermic needle, due to the shear force created by the flowing oil phase.

Flow rates for the syringe pump (WPI, Model Sp101i) are from 0.001 µl/min to 1.175 *ml/min*. The water droplets produced in this way do not settle out of the oil for a reasonable length of time. The water droplets in the oil come into contact with each other in the filter sample and form larger coalesced droplets of water that are carried downstream to the settling tank. The larger drops are easily separated from the oil in the settling tank. Smaller drops that do not settle out by gravity are carried into the reservoir tank

The filter holder is made of Plexiglas. Machined into the holder is a cylindrical opening for placement of the filter test sample. The test sample is held in place by a stainless steel wire mesh screen. All of the sample filters have a diameter of 2.50 cm.

Sampling is conducted to measure the size distribution of water droplets at intervals of 10 to 25 minutes from the sampling taps positioned upstream and downstream, respectively, of the filter holder. Simultaneously, the pressure drop across the filter is measured using a manometer. The pressure drop reaches steady state in about 10 minutes. After that time, coalesced water breaks through the filter and appears in the filter effluent. The size distribution of water droplets are measured with a particle size analyzer (Hyac Royco BR8 particle counter, 8 channels, sizes from 1 to 150 microns).

Filter Samples

Filter samples are formed from glass fiber as supplied by the J.C. Binzer Company with no further treatment of the fibers. Measured quantities of fibers are dispersed in a dilute aqueous solution of sulfuric acid (pH 2.5) and Carboset 560 binder (acrylic polymer dispersed in water). The slurry is stirred 8 to 24 hours. The slurry is vacuum filtered onto a fine mesh screen in a mold with an inside diameter of 2.54 cm to form the filter samples. The applied vacuum pressure is from 100 to 160 kPa. To make the glass fiber filter augmented with polymer nanofibers, the nanofibers are added to the slurry of glass fibers and the filter sample is prepared as mentioned above. While still moist, the filters are removed intact from the screen and dried in an oven for about 120 minutes at a temperature of 150 °C. This heat treatment provides the filters with sufficient wet-strength for the coalescence study by thermal setting the Carboset 560. The filter porosities are measured using a pycnometer.

Electrospinning

Figure 3 shows a schematic diagram of the apparatus used in the electrospinning. The syringe is filled with polymer solution. The flow rate is controlled with a syringe pump (WPI, Model Sp101i). The needle with a 1.6 mm outside diameter, 1 mm inside diameter is connected to a power supply and charged to 20,000 volts (Gamma High Voltage Research, Model D-ES30PN/M692). The collecting surface is grounded to attract the charged fibers. A collecting surface is placed at a distance of about 20 cm from the tip of the needle of the syringe pump. The needle is charged to 20000 volts to charge the polymer solution and the polymer nanofibers are produced.

Three polymer solutions are used to make polymer nanofibers. The Nylon solution is prepared by dissolving 16 wt% Nylon-6 by mass in 84 wt% Formic acid. Fibers of Poly(meta-phenylene isophthalamide) (Polyamide) are dissolved in N,N-dimethylacetamide (DMAc) containing 4% lithium chloride (LiCl) at 60 °C to form a homogeneous solution with 16% polymer solution. The PAN solution is prepared by dissolving polyacrylonitrile 12% in 88% N, N-Dimethyl formamide at 80 °C to form a homogeneous solution. Diameters of the fibers and specific surface areas of the fibers are provided in Table 3. Electrospinning concentration, quantity of polymer solution, and flow rate of the syringe pump are provided in Table 4.

Contact angle

One method for determining the wettability of fibers is the Wilhelmy method. Another method for measuring the contact angle onto a fiber is by measuring symmetrical drops of liquid directly attached to a fiber and analytical expressions relating drop length, drop radius, and fiber radius to the contact angle to determine the surface free energy (Schrader & Loeb 1992). However, both methods are difficult to use to measure the contact angle on the polymer nanofibers (for instance, electrospun nylon fibers have 150 nm diameters).

Contact angles are used to predict wettability. The wetting tension τ, is calculated by

$$\tau = \gamma_{LV} \cos\theta \tag{3}$$

where γ_{LV} is the surface tension of the test fluid and θ is the contact angle at the liquid-vapor interface line (Vogler, 1998). We assume that the wettability of the polymer nanofiber is same as a surface coated with the polymer. A spin coater (Specialty Coating System, Model P-6000) is used for coating the surface of a glass slide. A small amount of polymer solution is applied on the rotating glass surface. Contact angles are measured by using a contact angle meter (Ramé-Hart, Inc., Model 100-00 Contact Angle Goniometer) and by calculating the slope of the tangent to the drop at the liquid-solid-vapor interface line.

3. Results and Discussion

3.1 Effect of polymer nanofibers

The main aim of this research is to investigate the effects of adding the polymer nanofibers to glass fiber media on filter media performance. Filter media and electrospnning data are summarized in Table 2 to Table 4. The total mass of polymer solution used to make the nanofibers was constant at 100 ul. Four different filter media have been examined. The plots of pressure drop and separation efficiency, E, for polymer nanofiber filters are shown in Figure 4. As the water-in-oil emulsion passes through the filter, a saturation profile builds up and the pressure drop increases steadily to a constant value. The results for the particle size distribution of upstream and downstream emulsion as measured by the Hyac Royco BR8 particle counter are shown in Figure 5. Separation efficiency, E is defined as follows

$$E = 1 - \frac{N_d}{N_u} \tag{4}$$

where N_u is the fraction of water droplets upstream and N_d is the fraction of water droplets downstream (Akagi et. al., 1990). Coalescence efficiency is defined as the fraction of dispersed water that is coalesced by the bed and hence effectively removed from the emulsion by gravity separation. The particles counted at the sampling port, after the settling tank in Figure 2, are those that did not get

separated. The design of the settling tank may influence these results; hence these results are compared between the experiments for relative performance.

The nanofiber filters had a better capture efficiency, but also had a higher-pressure drop. In these experiments Figure 6 shows that the increased surface area of the filter samples provides an improvement in the coalescence efficiency. Coalescence performance also depends on other factors such as wettability. The wetting behavior of the water-in-oil or oil-in-water emulsion is considered to be important in determining the performance of the coalescence efficiency. The area ratio of nanofiber is defined by

$$Area\ ratio\ of\ nanofiber = \frac{A_{nanofiber}}{A_{glassfiber}} \tag{5}$$

where the areas of the fiber are calculated by

$$A = \left(\frac{4}{d_f \rho}\right) \times w \tag{6}$$

and where ρ and w are density of the fiber and mass of the fiber in the filter sample.

3.2 Effect of wettability

Four types of different wettability filters were involved in the investigation. Table 5 summaries the surface tension of the test fluid, γ_{LV}, and the contact angle at the liquid-vapor interface line, θ. Table 6 summarizes the wettability results obtained for deionized water on the four materials of the fibers. Figure 7 has photographs of the drops of water on the glass and polymer surfaces. Glass fiber has the highest wettability of all of the materials tested.

The plot in Figure 5 shows that only droplets of the smallest diameter detectable appeared in the downstream dispersion. The 3 *mm* thick glass fiber plus polymer nanofibers filter has a larger surface area than the 5mm glass fiber filter. Whereas the best coalesce performance was found, as expected (Voyutskii et al. 1955, Hazlett 1969, Moses & Ng 1985, and Basu 1993), when using only the high wettability fibers. High wettability materials promote coalescence in water-in-oil dispersions. Table 2 summarizes the separation efficiency (E) and quality factor (QF) obtained for five filter media. Glass fibers have a higher wettability for water than the polymers used to make nanofibers. In general, the higher wetting fibers provide better coalescence. In this particular application, the best separation efficiency (E) was obtained with the glass fiber plus Polyamide nanofiber filter for the same size filter media and the combination of glass fibers and polymer nanofibers did not improve performance when compared through the quality factor (QF). However the glass fiber plus PAN polymer nanofiber filter has same separation efficiency (E) as the glass fiber plus Nylon polymer nanofiber filter which has the small diameter fiber and hence more surface area. The 5mm glass fiber filter, which has a smaller surface area compared to other media augmented with polymer nanofibers, has the best overall separation performance of all of the filters. This result shows that the wetting of the fibers by the dispersed phase promotes coalescence, and best coalescer filter media are made of high surface energy materials for water-in-oil dispersions.

3.3 Effect of filter depth

Two glass fiber filter depths of L=3 and 5mm have been tested. The plots of pressure drop and separation efficiency, E, Filters 1 and 4 are shown in Figure 4, and Table 2. The better capture efficiency by filter 5 compared to filter 1 can be explained by the dependence of capture on drop size and filter depth. Smaller particles are more difficult to capture and require thicker filters for a given capture efficiency. Equation (1) was applied to determine the coalescence efficiency η_c for the water-in-oil emulsion and listed in Table 2.

The coalescence efficiency decreases with an increase in depth as shown by the experimental results on Filters 1 and 5. This conclusion is in agreement with the findings of Mathavan and Viraraghavan (1992) and Moazed and Viraraghavan (2001), who found that the coalescence efficiency

in a peat bed and in granular organo-clay/antharacite mixture bed decreased with an increase in depth of the bed. For the coalescence process to be effective in a coalescer bed, the ratio of water drop diameter to fiber diameter should increase with an increase in filter depth. This trend was not observed in the 3~5mm depth glass fiber filters.

For the experiments conducted here the steady state values from the experiments are used to calculate the quality factor, and are listed in Table 2. Because of the design of the experiments, using a settling tank to remove the larger drops and the probability of re-entrainment, the quality factor calculated for this data is not a true representation of the filter performance, but represents the combined system performance.

The quality factor results show that the glass fiber only media perform better than the media with nanofibers. This is attributed to two factors: first, the glass fibers have a higher wettability than the polymer nanofibers, hence the glass fiber only media perform better to capture and coalesce to form larger drops, and second in the liquid phase filtration the large surface area of nanofibers significantly increase the drag force, causing the pressure drop to increase faster than the capture efficiency. This is the opposite to what can occur in gas phase filtrations where the nanofibers are small enough that the gas flows past the nanofibers in a slipflow mode (Graham, 2002).

High wettability polymers may perform better than the polymers used in this work. Also, the media with nanofibers may perform better than the glass fiber only filter media with drops that are submicron in size. Both of these topics are left for future work.

3.4 Effect of flow rate

Flow rate plays an important role in the filter media. It determines not only whether an emulsion will be generated in the mixing pipe, but also controls the migration behavior of the dispersed droplets. High flow rates (230 *ml/min*) produce a small mean size of water droplets in upstream and larger pressure drop across the filter media (see Figure 8). The graph in Figure 9 illustrates the experimental results of water particle size distribution of upstream and down stream of Filter 1. The separation efficiency for the experiment with flow rate of 230 *ml/min* is 54% compared to 71% below that for a flow rate of 100 *ml/min*. In these two ranges of fluid rate, the higher flow rate also requires a higher pressure drop, whereas at higher flow rates the filter efficiency significantly decreases.

4. Conclusions

In this work, the various factors such as materials, wettability, filter depth, and flow rate are evaluated for their affect on coalescence efficiency. A blend of glass fibers with polymer nanofibers having diameters of around 150 nanometers showed improvement of separation efficiency. Surface area varies with the diameters of the fibers and the amount of polymer electrospun into the filter media. The experimental data show us that even small additions of nanofibers increase in the capture efficiency of the filter media, but also cause an increase in the pressure drop. In general, the higher wetting fibers provide better coalescence. In this particular application the combination of glass fibers and polymer nanofibers did not improve performance when compared through the quality factor. The overall coalescence efficiency decreased with an increase in depth for the experimental conditions applied here. The flow rate has a significant effect on performance, with lower flow rates performing better than higher flow rates. As expected, thicker filters provide better coalescence but at higher pressure drops.

Acknowledgement

This work was supported by Ahlstrom Paper Group, Donaldson Company, Fleetguard, Hankison International, Hollingsworth and Vose, and Parker Hannifin. This work was also supported by the National Science Foundation grant CTS-9900949.

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Table 1. Comparison separation efficiency (E), quality factor (QF), and coalescence efficiency (η_c).

Factors	Advantage	Disadvantage	Use
E	Simple, Common use, direct evaluation of particle capture	Does not account for pressure drop and hence does not account for filter thickness	Can be correlated to compare overall capture efficiency
QF	Combines E and pressure drop to eliminate dependence on the filter thickness	Assumes filter capture coefficient, is not dependent on non- linear mechanisms.	Used to compare media of different thicknesses and to optimize design
η_c	Accounts for the mechanism of coalescence	In present form applies to monosized fibers.	Used for coalesce media of one fiber size

Table 2. Physical property of filter media

Parameter	Filter 1	Filter 2	Filter 3	Filter 4	Filter 5
Added Material	None	PAN	Polyamide	Nylon	None
Weight, g	0.18	0.24	0.22	0.24	0.32
Length, mm	3	3	3	3	5
Porosity	0.940	0.962	0.973	0.985	0.940
Area Ratio of Nanopolymer, %	0	3.79	14.97	10.02	0
Efficiency, %	71	75	77	75	92
S *	0.019				0.017
η_c **	0.445				0.072
Quality Factor	2.25	1.02	1.03	0.80	3.93

* Average filter saturation
** Efficiency of coalescence

Table 3. Fibers and their diameters (Rangarajan, 2001)

Parameter	Approximate average diameter,	Specific Surface area
Glass fiber	1.5 μm	1.21 m^2/g
PAN nanofiber	500 nm	6.76 m^2/g
Polyamid nanofiber	150 nm	22.6 m^2/g
Nylon nanofiber	250 nm	14.3 m^2/g

Table 4. Data of electrospinning

Polymer Solution	PAN	Polyamide	Nylon
Concentration, wt %	12	16	16
Quantity, μl	100	100	100
Flow rate for electrospining, μl / min	21.4	3.6	3.6

Table 5. Physical property of used fluid

Type of fluid	Visco 1487	Deionized water
Specific gravity	0.832	1
Surface tension (at 28 °C), dynes/cm	28.5	71.0

Table 6. Contact angle and wettability with water at 28 °C, and filter separation efficiency

Polymer Solution	Glass	PAN	Polyamide	Nylon
Contact angle, θ, degree	0	38.6	52.1	46.4
Wettability, dynes/cm	71.0	55.4	43.6	49.0
Separation Efficiency (E), % 3mm filter	71	75	77	75
Separation Efficiency (E), % 5mm filter	92			

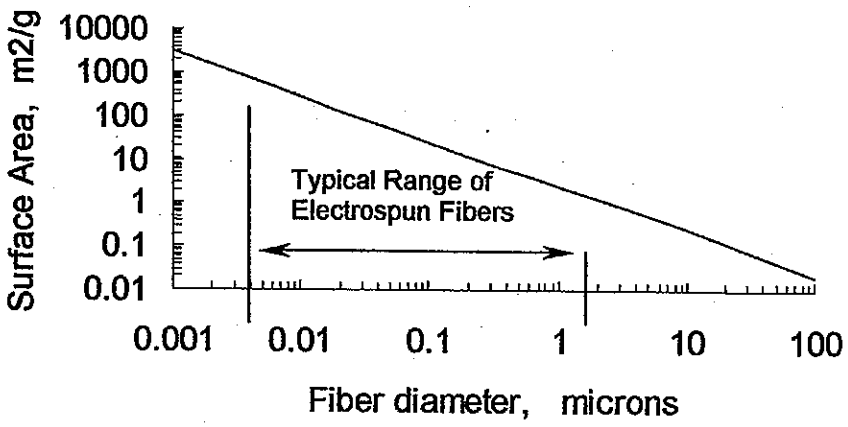
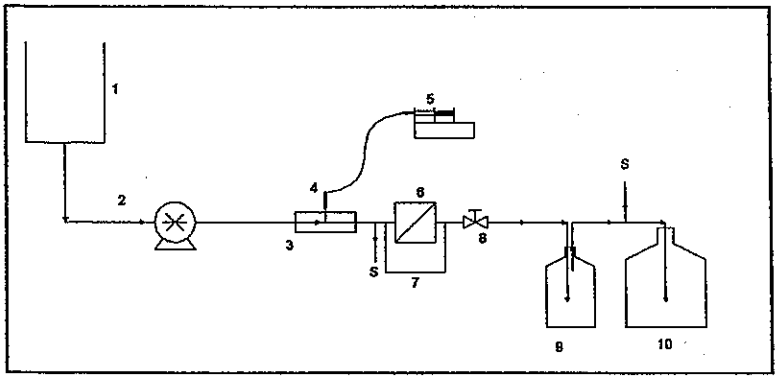


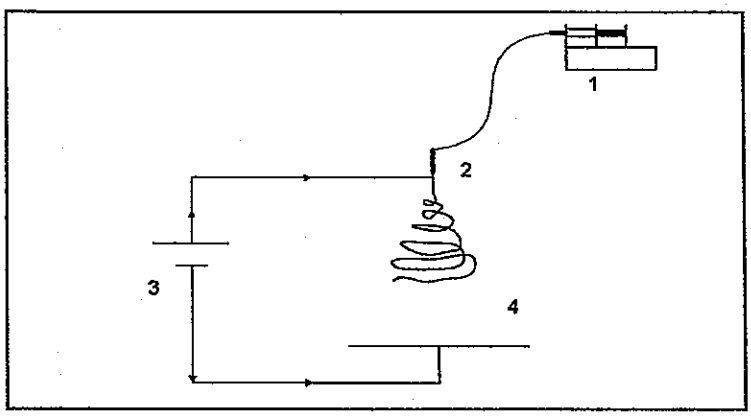
Figure 1. Nylon Fiber diameter vs. Surface area for a polymer with density of 1120 kg/m^3. electrospun fibers are typically submicron up to a few microns in diameters.



1. Oil Tank
2. Peristaltic pump
3. Mixing pipe
4. Hypodermic needle
5. Syringe pump
6. Filter Sample holder
7. Manometer
8. Valve
9. Settling tank
10. Reservoir tank

S. Sampling points

Figure 2. Experimental apparatus.



1. Syringe pump
2. Hypodermic needle
3. Power supply, 20000 volts
4. Collecting surface

Figure 3. Experimental apparatus of electrospinning.

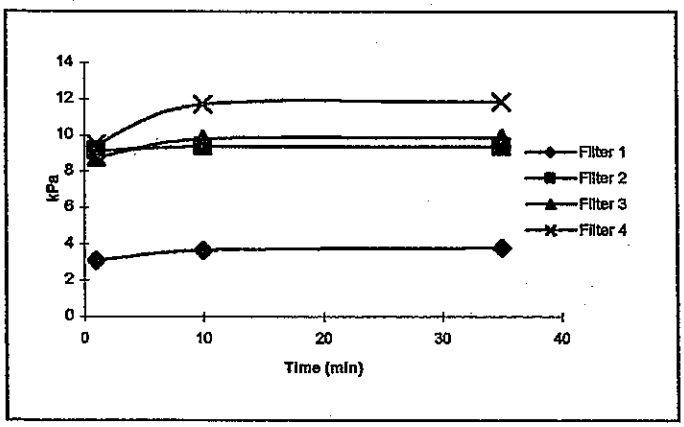


Figure 4. Pressure drop vs. time for different filter media listed in Table 2.

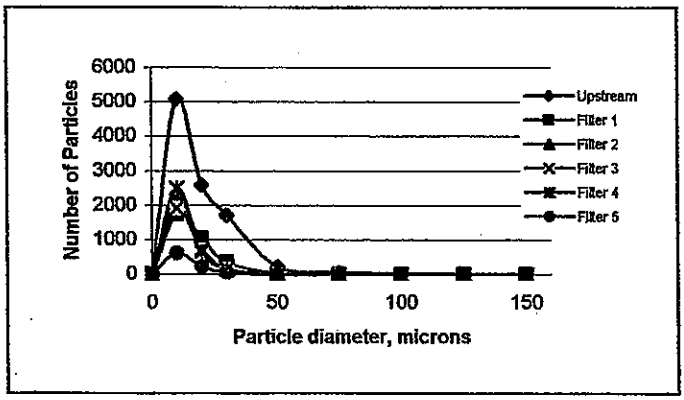


Figure 5. Particle size distribution of water droplets upstream and downstream of the five filters listed in Table 2.

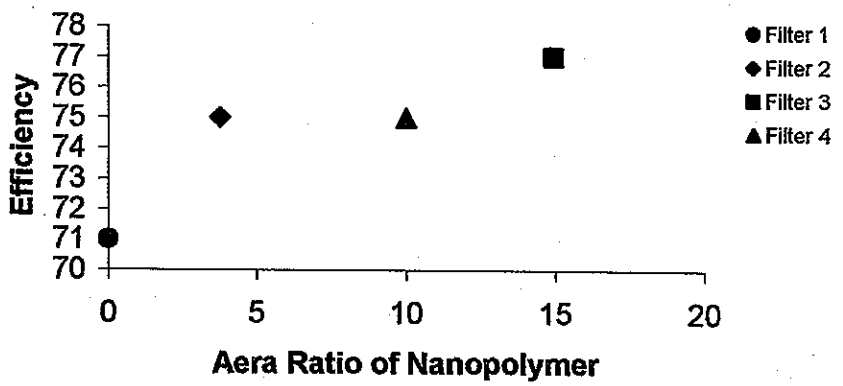


Figure 6. Effect of nanofibers on filter performance. The data are plotted as the area ratio of polymer nanofibers vs efficiency for the four filters of the same thickness.

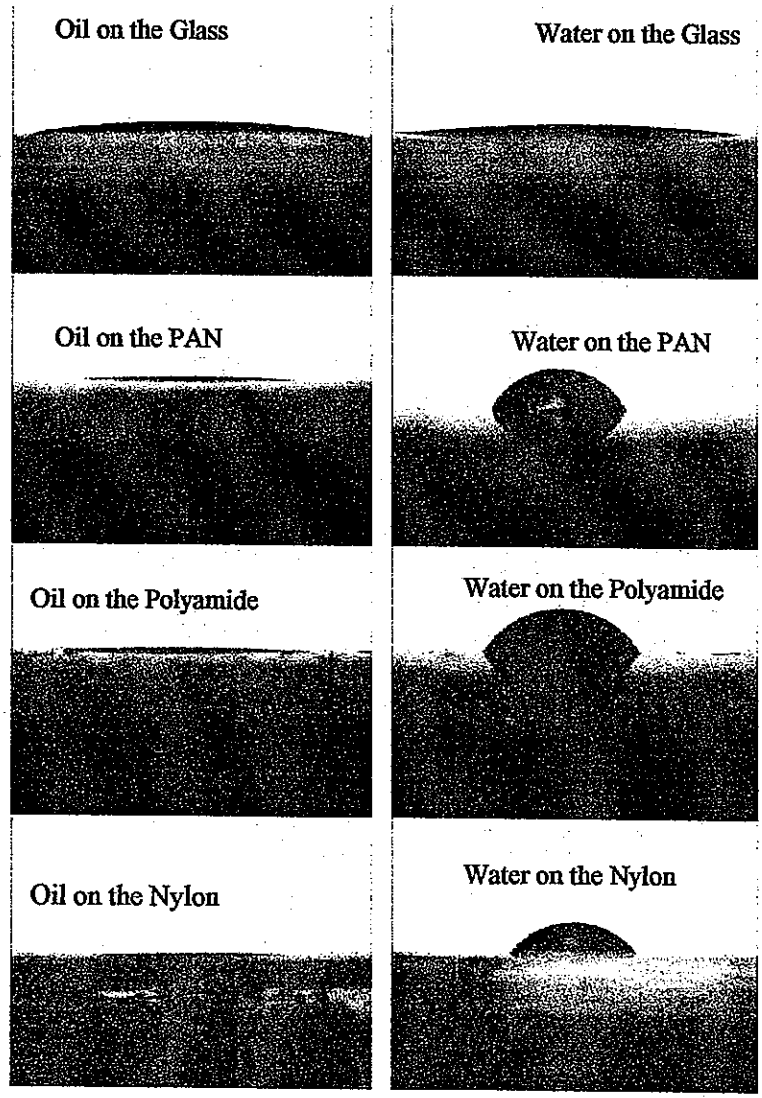


Figure 7. Oil and water drop on four different surfaces using the drop shape method.

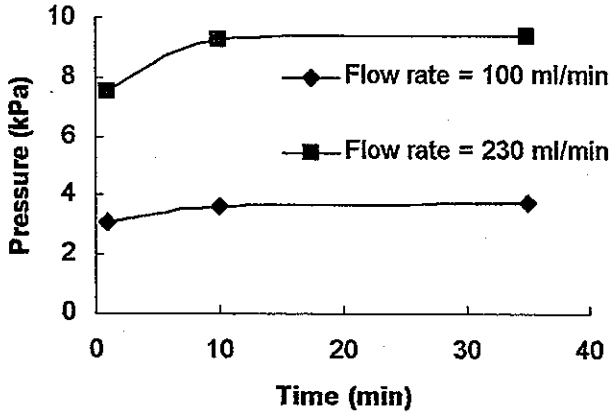


Figure 8. Pressure drop vs. time for different flow rate for Filter 1.

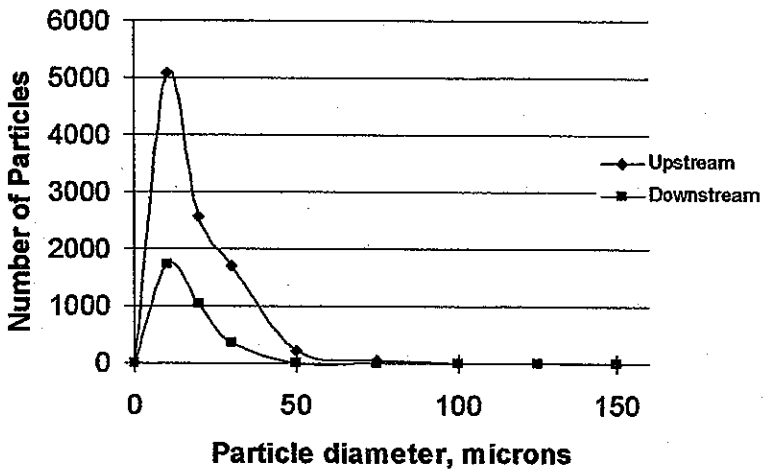


Figure 9. Particle size distribution of water droplets upstream and downstream of Filter 1.