MIXED HYDROPHILIC / HYDROPHOBIC FIBER MEDIA FOR LIQUID-LIQUID COALESCENCE

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MIXED HYDROPHILIC / HYDROPHOBIC FIBER MEDIA FOR LIQUID-LIQUID COALESCENCE

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ABSTRACT

Separation of liquid-liquid dispersions is important industrially and environmentally. Fibrous coalescing filters are used to separate liquid-liquid dispersions. Wettability of the fibers significantly influences the filter performance. The wettability of the filter media depends on surface properties of fiber materials and porosity of the filter medium. The wettability of filter media and interfacial tension of the liquid are the important factors in designing a coalescing filter media. It is generally accepted that the intermediate wettability gives best filter performance. However, the effect of wettability on filter performance is not well understood, especially at low interfacial tension liquids. In addition, wettability characterization techniques are not widely applied for fibrous filter media.

The goal of this work is to study the effect of wettability on filter performance and by evaluating filter media of varying wetting properties. In this work, filter media were prepared by mixing hydrophilic and hydrophobic fibers to control the wettability and to improve the performance of the filter media. The mixing of fibers was achieved by designing filter media in different geometries. The different geometries developed and evaluated are mixed, layered and radially layered hydrophilic/hydrophobic fiber media. The wettability of the filter media was characterized using modified Washburn’s equation and expressed as the Lipophilic to Hydrophilic (L/H) ratio. Water and Viscor-1487 oil were used as test liquids for the wettability characterization. The developed geometries
resulted in variation in wettability. The filter media were tested in liquid-liquid coalescence experiments in which fine emulsion was generated by dispersing water droplets in the Viscor-1487 oil.

The experimental results show that wettability of filter media can be controlled by constructing a media with different geometries and choosing a right composition of hydrophilic/hydrophobic fibers. The pressure drop of filter media was reduced with increase in L/H. The performance of filter hydrophilic/hydrophobic filter media was improved as compare to only glass fiber (hydrophilic) media. However, improvement in quality factor is mainly due to reduction in pressure drop. The best filter performance was achieved with layered and radially layered structures made of glass and polypropylene fibers having L/H between 2 to 10. The media developed with glass and electrospun polypropylene fibers shows significant improvement in separation efficiency but also increase in pressure drop. The results and approach of this work will be useful in designing and developing filter media for many industrial filtration applications.

*Keywords:* Filter media, coalescence, wettability, modified Washburn’s equation, filter performance, interfacial tension.
DEDICATIONS

My beloved parents
Smt. Usha S. Kulkarni
Late Mr. Shashank V. Kulkarni
And
My beloved grandmother
Late Smt. Vasundhara V. Kulkarni
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CHAPTER I

INTRODUCTION

1.1. Motivation

Separation of liquid-liquid dispersions is of significant importance industrially and environmentally. In liquid-liquid dispersions, two immiscible liquid phases are present in which one liquid phase is finely dispersed in other liquid phase. The fibrous coalescing filters are the effective and economical way for separation of such finely dispersed immiscible phases [1-4]. Examples includes but not limited to,

- Removal of water from liquid fuels in automotive industry[5]
- Removal of water from aviation fuels [2, 4]
- Removal of water from hydrocarbons and petrochemicals [6, 8]
- Dewatering of crude oil [9, 10]
- Removal of oils and hydrocarbons from water and wastewater [11-14]

Emulsions and dispersions are common sources of contaminants in chemical and petrochemical processes. Water present in hydrocarbons and process fluids generates stable emulsion and cause products to be off specification [7]. The liquid fuels contain particulate matter, water, wax and biological contaminants. The water can enter into fuels
during transfer, bulk storage, rainfall etc.; however, it is mainly a result of condensation of atmospheric moisture within the tank [4]. Water causes problems like corrosion of sensitive engine components, microbial growth, degradation of fuel quality and burning efficiency. Effect of water is more complex and separation of water from liquid fuels is difficult as compare to other contaminants. The fuels have different additives as lubricity agents, corrosion inhibitors, wax dispersants, antifoam etc. These additives can be classified in one category of “surfactants”, which reduces the interfacial tension of fuel and separation water becomes more difficult [2, 7, 15]. The use of biodiesels and their blending in diesel fuels also lowers the interfacial tension of fuels [15, 16]. Biodiesels contains as much as 0.5 % of water [17]. Along with decreasing the interfacial tension, surfactants coat the fiber surface and can change the wettability of the fiber surface. This leads to reduced performance of coalescing filters.

Water is present in fuels as free water, emulsified water and dissolved water [18]. The free water is generally separated by gravity settling or other mechanical operations. The emulsified water having drop size less than 100 μm, known as secondary emulsions, are difficult to separate. The fibrous coalescing filters are commonly used to separate such finely dispersed secondary emulsions. Using fibrous coalescing filters, the fine emulsions of droplets below 100 μm are transformed into enlarged drops which can be easily separated by gravity settling or any other mechanical operations [1].

The filtration industry is in the need of long term solutions for separation of liquid-liquid dispersions, which can be achieved by improved design and development of coalescing filters. The understanding the effects of wettability and interfacial tension on filter performance are the important aspects of design of coalescing filter.
1.2. Coalescence filtration

In the coalescence process, the droplets are captured on fibers, the collected water droplets migrate through the fiber media and coalesce to form enlarged drops that are released from the fiber surfaces [1, 2]. The performance of coalescing filters is measured in terms of separation efficiency and pressure drop. The separation efficiency is dependent on properties of the emulsion (e.g. composition, density, viscosity, drop size etc.). It also depends on the filter properties including the fiber surface wetting properties, fiber size, fiber orientation, porosity, binder content, and filter bed length [11-13, 21-26]. The continuous phase flow rate is important as it controls the mechanism of droplet capture on the fibers, inertia force available for drop-drop collision and coalescence, and the drag force on the coalesced drops to cause the drops to migrate through the filter. In liquid-liquid coalescence, wettability of filter media significantly affects the filter performance [21-27]. The effect of wettability of filter media is more significant when interfacial tensions between liquids are low. The wettability of fibrous filter media depends on surface properties of fibers and porosity of the medium. Wettability of fibers can influence the capture of droplets on fiber surface, migration of droplets on fiber surface, enlargement of droplets, time for which droplets stay on fiber surface and drainage and release of drops from filter medium.

1.3. Problem statement

Despite having a significant influence on filter performance, wettability has given a minimal attention in literature. The effect of wettability on filter performance is not
well understood when interfacial tensions are low. There is not enough data available in literature to understand the effect of wettability and interfacial tension on the performance of coalescing filter media.

The wettability of a solid surface is generally characterized using the contact angle method. The nonwoven fibers, fiber mats and fiber sheets are termed as hydrophilic or hydrophobic based on the contact angle of the original material on flat surface. There are different techniques which are developed to determine wettability of nonwoven and textile materials based on phenomena of wicking and capillarity. However, wettability characterization techniques are not widely applied to determine the wettability of fibrous coalescing filters.

The properties of liquid fuels can vary based on location, weather and season. The liquid fuels with different interfacial tension are used in different applications. In separating emulsion and dispersions, interfacial tension of the liquids and their wetting behavior with the porous medium are important aspects in designing an efficient coalescing filter media. The design of filter media of required wettability for fuels with variation in interfacial tension is presently limited to the selection of fiber material or specific type of coating on fibers. A common practice to control the wettability is by selecting a material from which the fibers are made that has an intermediate wettability, or by applying a coating, such as a silane compound, that gives the surface of the fiber structures the desired intermediate wettability. The difficulty here is finding the right material or coating that has the best wettability for a given application. This approach does easily provide for incremental changes in wettability.
1.4. Hypothesis

In water-in-oil coalescence, water is the dispersed phase and oil is the continuous bulk phase. In the coalescence process, the water drops coalesce to form large drops on hydrophilic fibers [22]. These water drops occupy the pores formed by fibrous structure. Eventually, increased water hold up inside filter media reduces porosity and permeability and increases saturation. The increase in saturation leads to excessive pressure drop. If fibers are hydrophobic, the water drops do not adhere to the surface of the fibers and limited or no coalescence occurs at the fiber surface [22]. These general observations are summarized in Figure 1.1. Research results show the best filter performance is obtained at the intermediate wettability [22, 24, 25]. Therefore, to achieve best filter performance, the filter should be sufficiently water-wetted to form enlarged water drops but not too water-wetted to increase saturation and pressure drop. The available data and literature do not provide design information on what is the optimum wetting range for the best filter performance.
This work has three hypotheses based on mixing or blending of hydrophobic and hydrophilic fibers in the construction of filter media.

1. Hypothesis One:

The First hypothesis of this work is that by constructing filter media of mixed hydrophilic and hydrophobic fibers one can control the wettability of the filter medium and hence can control the filter performance for liquid-liquid coalescence. The hydrophilic fiber generates large drops and drops that tend to stay on hydrophilic surface for extended period of time. The hydrophobic fibers aid in drop migration and drainage, thereby reducing saturation and increasing permeability. In mixed hydrophilic/hydrophobic fiber media, the hydrophobic fibers can divert the water drops towards
hydrophilic fibers and enlarged drops will form on hydrophilic fibers. The enlarged drops are easier to separate using downstream devices such as gravity settling.

The mixing of hydrophilic and hydrophobic fibers can be achieved by designing a filter media in different fiber structural geometries. The different geometries result in wettable and non-wettable flow paths and variation in wettability.

2. Hypothesis Two

The second hypothesis of the work is that wettability of the filter media can be varied by constructing filter media in different geometries of layers of wetting and non-wetting fibers.

3. Hypothesis Three

The third hypothesis of this work is that the wettability of the filter media can be controlled by choosing the right composition of hydrophilic and hydrophobic fiber materials in media. The media with required wettability can be designed for particular application.

The goal of this research work is to study the effect of wettability on filter performance by evaluating filter media of different wetting properties. The filter media with varying wetting properties can be achieved by varying composition of hydrophilic/hydrophobic fiber materials in a particular geometry. The optimum wetting range for the best filter performance can be achieved by evaluating performance of filter media of a range of different wetting properties.
1.5. Research objectives

The primary objectives of the research work are:

- Design and develop different geometries of filter media using hydrophilic and hydrophobic fibers.
- Develop filter media in different compositions of hydrophilic/hydrophobic fibers to achieve media with different wetting properties.
- Develop and apply appropriate wettability characterization technique for fibrous filter media e.g. Modified Washburn’s equation, contact angle etc.
- Evaluate performance of developed filter media for liquid-liquid coalescence.
- Study the factors affecting the wettability of the filter media.
- Study the effect of wettability on filter performance.
- Study the effect of interfacial tension on filter performance.
- Analyze and predict the appropriate materials and optimum wettability range for liquid-liquid coalescence.
- Set-up a theory to account for pressure drop and saturation as function of position of filter media with effect of wettability.

1.6. Approach

In this work, filter media were developed by mixed hydrophilic and hydrophobic fibers in different compositions to achieve range of wetting properties and attempt to achieve optimum wettability range for best filter performance for liquid-liquid
coalescence. In designing such media, the materials can be in the form of micro and nano-sized fibers, fibrous sheets, synthetic pulps or any form of material, which will aid in coalescence and optimizing the wettability of the filter media. Materials were selected based on their surface property, compatibility with liquids used, availability, and possible applications in industry. The media were evaluated for their wettability using Washburn’s wettability characterization technique, which is described in chapter IV. The filter performances were evaluated using liquid-liquid coalescence experiments. The Viscor 1487 (Viscor oil) (Rock Valley Chemical Company, IL, USA) was used as the oil phase for liquid-liquid coalescence experiments and as a test liquid in wettability characterization. Viscor oil is a calibration fluid having properties similar to diesel fuel. The interfacial tension of fuel was lowered by using a surfactant and experiments were performed with a selective filter media among the filter media tested. The experimental results will be helpful to understand the effect of wettability and interfacial tension on filter performance.

1.7. Benefits of work

There are over 900 companies involved in filter media manufacturing, design and distribution for many different applications [28, 29]. The research results from this work; including filter media design, development, testing and characterization; can have significant economic impact on the filter industry.

The results of this work will directly impact the automotive industry, especially in fuel filter design. The results will also impact the aerospace industry, similarly for
aviation fuel filter design. The results may be useful for the liquid-liquid separations in petrochemical industry. The similar approach can be applied to separation of aerosols from air or gas stream based on wettability of the liquid. The results obtained will be helpful to fiber and media manufacturers in understanding wettability of the different fibers and fiber structures. The broader impacts of wettability characterization results include textile industry in characterizing absorbing or non-absorbing materials. Although the work is focused on separation of water from liquid fuels, the principles and approach can be applied to the separation of oil from water. The results can be useful in designing filters for separation of oils and hydrocarbons from produced water, industrial water, and wastewater.

1.8. Dissertation outline

CHAPTER I explains the motivation behind the work, problem statement and hypothesis of work, research objectives and approach of the work and benefits of the work.

CHAPTER II reviews the literature on sources and applications of liquid-liquid coalescence and separations, theory and literature on coalescence filtration, wettability, interfacial tension and electrospinning.

CHAPTER III explains the design and preparation of different hydrophilic/hydrophobic filter media geometries.

CHAPTER IV presents the wettability characterization technique, wettability characterization results of all hydrophilic/hydrophobic filter media.
CHAPTER V explains the liquid-liquid coalescence set-up and coalescence experimental procedure.

CHAPTER VI presents the experimental results of all hydrophilic/ hydrophobic fiber media and studies the effect of wettability and interfacial tension on filter performance.

CHAPTER VII presents the conclusion of the research work based on wettability characterization and coalescence experiments.

CHAPTER VIII presents the recommendation for the future work on liquid-liquid coalescence.

APPENDIX A describes the model set-up of two-phase flow through fibrous filter media for pressure drop and saturation as a function of filter bed length considering the relative permeability of wetting and non-wetting phases in porous media.

APPENDIX B describes the nomenclature of symbols used in the model.

APPENDIX C presents the use of Jorin ViPA instrument for upstream and downstream sampling and results with mixed media are presented.

APPENDIX D presents the regression analysis of L/H and fraction of polymers fibers used in the mixed and layered filter media.
CHAPTER II

LITERATURE REVIEW

The chapter reviews the applications and liquid-liquid separation, coalescence filtration theory, effect of wettability and interfacial tension on liquid-liquid separation and electrospinning.

2.1. Applications liquid-liquid filtration and separation

Liquid-liquid dispersions are encountered in many industrial applications and separation of those finely dispersed immiscible liquids is important for efficient operation of the system, removal of contaminants, maintaining the quality of products and environmental protection. Some of industrial applications are explained below.

- Automotive industry – Separation water from liquid fuels

   The liquid fuels contain particulate matter, water, wax and biological contaminants. The water can enter into fuels during transfer, bulk storage, rainfall etc.; however it is mainly a result of condensation of atmospheric moisture within the tank [18]. The water is present in fuels as free water, dissolved water and emulsified water. The emulsified water is the major reason of concern because it combines with chemicals in fuels such as sulfur and chlorine, to form corrosive
compounds and corrodes sensitive engine parts. The corrosion is the main reason for diesel engine failures and maintenance issues [21]. The dissolved water does not have significant effect on fuel systems but at low temperatures it can freeze and could cause problems. In addition, water promotes microbial growth in engines resulting in blockage of fuel filters and injection systems. The wax is problematic in cold conditions, which also blocks the fuel filters. The separation of water is more difficult and has more complex effects as compare to other contaminants. In addition, different additives present in fuels reduce the interfacial tension of fuels and problem of separation become more difficult [14, 30-33].

- Aviation Industry – Separation of water from liquid fuels

    The safe and efficient operation of aircraft requires fuel which is clean and dry. Water is a most serious hazard as compare solid contaminants. The solubility of water varies with temperature. The water can freeze out at any point in the fuel system and become a source of mechanical difficulty. All the contaminants should be removed from the fuel before refueling the tank. The primary tendency of free water is to precipitate under low temperatures. As an aircraft operates at different altitudes and at ground level, it consequently experiences extreme variations in temperature. The precipitated free water can freeze into ice when aircraft operates at below 30F. The density and viscosity of jet fuels such Kerosene and JP-4 is higher than automotive fuels and settling of contaminants take a longer time [2, 34].
- Petroleum refineries and petrochemical process

  Refineries use liquid and gas coalescers extensively for various applications. The typical refinery applications are: amine sweetening, hydrotreating naphtha, caustic treating, catalytic reforming unit, alkylation, salt extraction from hydrocarbons, protection of catalysts from water contamination in packed bed and fluidized bed reactors, removal of carried-over caustic from caustic treatment processes, removal of carried-over amine from LPG, reduction of sodium/potassium levels in gasoline [35].

  Along with the refinery applications coalescing filters are largely used in the petrochemical industry where typical applications includes but not limited to separating fuel oils (including heavy fuel) from heavy oil as well as separate organic phase from liquid phase. Coalescers are also used to separate water and acids from hydrocarbons, thereby preventing corrosion and saving of inhibitor in packed bed reactors. Other applications include separation of salt from turbine fuels in order to avoid corrosion of turbine blades, filtration and water separation from turbine lube oils (Turbine oil conditioning) and regeneration of process-liquids. Point of use coalescers are widely used for quality control of fuel oils and fuels; removal of water haze and solids before fuel loading [36]. Today water contamination in refinery fuels is a bigger problem than solid contamination in refineries.

- Produced water applications

  Coalescing filters are used for crude oil exploration applications. They are used for high efficiency separation of oil and solids from injection water, high
efficiency separation of oil for produced water disposal, high efficiency separation of oil for desalination water disposal, high efficiency separation of oil from wash-down, oil separation from surface water of sealed areas and also automatic tank de-watering of crude oil to fuel oils, gasoline, separation of hydrocarbons from process water, enabling a recirculation of the water, elimination of hydrocarbon settling in the process etc. [37-38]. Instead of complicated stripping and purging procedures the use of coalescers is preferred; which are more efficient, simpler and produce less waste water [37].

- Wastewater treatment

The waste water industry uses filters for removal of chemical and biological waste and also toxic chemicals from the waste water, saving of polyelectrolytes and flocculants, water savings and improvement of wastewater treatment plant downstream [38]. In the water industry membrane, ultrafiltration, nanofiltration and reverse osmosis are the predominantly used technologies. The coalescers are mainly used for removal of dispersed oils from water.

2.2. Emulsions and stability of emulsion

The two immiscible liquids in which one is finely dispersed in another forms a dispersion. The droplet size of the dispersed phase in a continuous phase and settling time of the dispersed phase can define the mixture as either emulsion or colloid. In colloidal solutions, the dispersed phase can be solid or liquid and it does not settle for long time [39]. In emulsions, the dispersed drops larger than 100 µm settle quickly. However,
settling of very fine drop below 100 µm takes longer time [25]. The interfacial tension has significant influence on stability of emulsions. Liquids with interfacial tensions below 20 mN/m tend to form stable emulsion. The emulsions of water-in-oil having an interfacial tension above 20 mN/m has larger coalesced water drops which can be easily be separated by coalescing filters [6]. The fuels such as ultra low sulfur diesel, blends of biodiesels and fuels with additives may have an interfacial tensions below 10 mN/m. The liquid viscosity has a significant impact on the droplet coalescence. The droplets must overcome the higher drag forces exerted by the continuous phase viscous fluid to reach and colloid with another drop [2]. It is difficult to separate water drops from high viscosity liquids. The temperature of an emulsion also affects the separation process but primarily through modification of the fluid properties. Higher temperatures increase the concentration of dissolved water which can not be separated by liquid-liquid coalescers [4]. At lower temperatures the water can phase separate or can be removed by using coalescers.

2.3. **Coalescence Filtration**

As the emulsion passes through the filter medium the droplets are captured in the depth of the fiber medium. There are several mechanisms that contribute to the capture of droplets [40]. Submicron size droplets and particles are captured by the mechanisms as shown in Fig. 2.1. These mechanisms are called the single fiber capture mechanisms. The capture on a single fiber is considered to be dependent upon the local flow conditions around the fiber and when the filter properties are uniform the capture on a single fiber is
a representation of the entire filter medium. The main assumption of the single fiber concept is that all fibers are identical [41]. The main capture mechanisms observed are inertial impaction, direct interception, diffusion and gravitational deposition. The particle may also follow a mechanism of electrostatic deposition, but it is mainly dominant in solid particle capture. The single fiber mechanism has been widely accepted by the filtration industry as it leads to comparable results with experiments. All of these mechanisms occur simultaneously at any point of time. One or more of these mechanisms dominate depending on the droplet size of the liquid.

2.3.1. Capture Mechanisms

![Single fiber capture mechanisms](image)

Figure 2.1. Single fiber capture mechanisms (a) Inertial impaction, (b) Direct interception, (c) Diffusion.
a. Inertial impaction: A particle with higher density moves in a line less curved than that of the streamline with a higher probability that the particle will be captured by impaction. Impaction occurs when the particle traveling in the flow stream deviates by virtue of its inertia and collides with the fiber. This type of filtration mechanism is predominant with high gas velocities and dense fiber packing of the filter media is present. When a dense or large particle is transported in the gas stream, the particle’s inertia may be large enough that the particle’s trajectory will deviate from the air streamline as the air bends around a fiber. If the particle’s path carries the particle within one particle radius to the fiber surface, the particle will collide with the fiber and become captured. Particles or droplet usually larger than 1 µm follow this mechanism of particle capture.

b. Direct interception: In direct interception, a particle (or water droplet) following the streamlines of the laminar flow can be captured by a fiber because both the particle and fibers have finite sizes. If the radius of the particle is greater than the distance between the streamline which contains the particle and the fiber; the particle collides with the fiber and is hence captured. Streamlines further than one particle radius away from a filter fiber will not contribute to the interception mechanism. Particles in the range of 0.3-1 µm in diameter usually follow the air streamline; they will intercept a fiber if the distance of stream line approaches to less than the particle’s radius to the fiber surface.

c. Diffusion: A particle moving with a fluid tends to depart from the streamline due to collisions with the fluid molecules. This motion is random and gives the particles a motion similar to molecular diffusion. For small particles, with
diameters in the range of 0.1 µm, diffusion is the predominant capture mechanism. Diffusion is predominant with low gas velocities and smaller particle. Particles smaller than 0.3 µm have very little mass. As a result they travel in random motion superimposed upon the stream line flow due to collisions with air molecules, similar to molecular diffusion. The random motion increases the likelihood that they will come in contact with the fibers and will be captured. The capture of water droplets by this mechanism in the liquid fuels would decrease with higher viscosity of liquids; however the lower flow velocities would increase the efficiency of this mechanism of water coalescence in fuels. The diffusion coefficient is related to the viscosity, temperature and particle size. The influence of water droplet diameter is identified to be an important factor in the effectiveness of interception mechanism [32, 33, 40].

d. Gravitational Deposition: At low gas velocities, the larger diameter droplets may separate from the fluid stream due to the influence of gravity before reaching the medium. The larger diameter follows a vertical motion in the fluid stream. When droplet reach at the fiber, because of the vertical motion, it may get be captured by gravitation deposition and fluid stream pass through filter.

While the capture mechanisms described here are most effective on particles on a certain size range, an increase or decrease in the air velocity can cause the capture efficiency to vary [40]. The comprehensive view of fibrous filtration and its theory, efficiency of fibrous filters, comparison of fibrous filters with other filtration, loading characteristics of solid particles, and forces controlling the filtration process is given by
Brown [40] and Davies [42,43]. Stenhouse and Trotter [44] studied the loading of submicron solid particles on fibrous filters.

2.3.2. Factors affecting coalescence filtration

The separation efficiency of filter media for liquid-liquid separation is dependent on the properties of the dispersion (e.g., composition, density, viscosity, droplet diameter) and fiber bed properties including fiber sizes, fiber orientation, wetting properties of fibers and the filter [11-13, 21-26]. The surface tension of liquid and emulsion flow rate are also has significant influence on performance of the filter media. Saturation and pressure drop of the filter media are important parameters which need to be controlled to optimize filter life. Filter life and the filter media performance can be improved by lowering the saturation and pressure drop while maintaining high capture efficiency. Various parameters affect the coalescence filtration process as well as coalescers. They are listed as follows. These parameters are important in designing the coalescing filter media [32-33].

- Pore size impacts performance of coalescing filters. Coalescers perform well when pore size is decreased. Pore size can be reduced by reducing fiber size from micron to submicron or nanofibers. Nanofibers enhance the efficiency of coalescers as they have large surface area to volume ratio and smaller pores.

- Face velocity impacts on overall performance of coalescers as it controls the capture mechanism and probability of droplet capture. Coalescers have higher performance at lower face velocities.
• Droplets size of dispersed phase affects performance of the coalescers. Dispersed phases with larger mean droplet size are found to coalesce more easily.

• Interfacial tension is one of the important factors which affect performance of coalescing filters. Emulsions with higher interfacial tensions coalesce better than lower interfacial tension.

• Viscosity of the continuous phase affects performance of the coalescer. Lower viscosity of continuous phase helps in coalescence better than higher viscosity.

• Dirt and surfactant tend to reduce the chance of coalescence of dispersed phase.

• Influence of electrostatic potential barrier have a negative effect and can prevent coalescence.

• Intermediate values of surface energy of fibers perform better than high or low surface energy fibers in coalescing filters. This is generally accepted but very limited data are available in literature.

2.4. Wettability

2.4.1. Wetting Phenomena

The wetting phenomena is generally described by the displacement of solid-air interface with the solid-liquid interface. A small liquid droplet brought in contact with solid surface, it spreads over solid surface. The wetting behavior of the liquid with solid surface is characterized by contact angle. The forces in equilibrium on the drop at solid-liquid boundary, are shown in Figure 2.2. and described by Young’s equation [45,46]:

21
Figure 2.2. Liquid drop on a solid surface at equilibrium.

\[ \cos \theta = \frac{\gamma_{sv} - \gamma_{sl}}{\gamma_{lv}} \]  \hspace{1cm} (2.1)

Where, \( \gamma \) is the interfacial tension between the various combinations of solid, liquid and vapor; the subscripts \( s, l, \) and \( v \) stands for solid, liquid and vapor respectively. The \( \theta \) is the contact angle of liquid with solid surface.

If \( \theta = 0^\circ \) then liquid completely spread on solid surface and surface is called as hydrophilic. If \( \theta \leq 90^\circ \) then solid surface is mostly hydrophilic and if \( \theta \geq 90^\circ \) then solid surface is hydrophobic. The parameter called spreading coefficient \( S \) which gives the difference between the surface energy (E) of solid surface when it is dry and wet[47].

\[ S = [E_{solid}]_{dry} - [E_{solid}]_{wet} \]  \hspace{1cm} (2.2)

\[ S = \gamma_{so} - (\gamma_{so} - \gamma_{l}) \]  \hspace{1cm} (2.3)

Where, \( \gamma_{so} \) is the surface tension of vapor free dry solid surface and \( \gamma_{l} \) is the surface tension of liquid.
2.4.2. Effect of wettability on coalescence

In fibrous filter media, when water droplets in the oil stream come in contact with a fiber surface, it either attaches to the fiber surface, partially attaches to the surface or do not attach to the fiber surface. The Figure 2.3 explains the behavior of water drop on fiber surfaces different wetting properties.

![Diagram of water drop behavior on fiber surface]

If the fiber surface is hydrophilic, then water drops form a thick film on the surface. If the surface is hydrophobic then water drops do not attach to the surface and simply get carried over with flow.

Shin [22] studied the drop attachment on silane coated glass rods. The glass rods were coated with three different silanes APTS (3-aminopropyltriethoxysilane), CES (2-carboxymethylthioethyltrimethylsilane), FTS (heptadecafluoro-1,1,2,2-tetra-

Figure 2.3. Behavior of water droplet on fiber surface with different wetting properties.
hydrodecyltrichlorosilane) having water contact angle on flat surface 39°, 62°, 104° respectively. The uncoated glass rods had a water contact angle of nearly zero degrees. In his experiments with flow of petroleum ether and water over coated and uncoated glass rods, it was observed that water drops form thick film around the uncoated glass rods (hydrophilic) and the drops attached to the glass rods for long time. The water drops did not adhere to the FTS (hydrophobic) coated glass rod and drops were carried away with the flow without any coalescence. The water drops on APTS and CES coated rods (intermediate wetting) surface formed thinner films and coalescence was observed.

The background research and industrial knowledge consider that intermediate wettability gives the best filter performance. Wetting of water-in-oil or oil-in-water in a filter is considered to be important in coalescence efficiency of filter media.

Hazlett[19] reported that the water droplets should displace the oil film from a wet fiber surface in water-in-oil emulsion. A water droplet easily displaces oil on a hydrophilic surface. The displacement of the oil phase on hydrophobic surface is less than hydrophilic surface.

Sareen and coworkers [33] performed experiments with packed bed of different fibers with oil-in-water emulsions as a primary objective and also with water-in-oil emulsion. The fibers used were cotton, Dynel, glass, polyethylene, polypropylene, and Teflon. The mixed packed beds of cotton-glass, cotton-Dynel and cotton-Teflon were also tested. The cotton, glass and Teflon fiber bed showed coalescence when dispersed phase was lighter than continuous phase. The Dynel, polyethylene did not show the complete coalescence. Mixed-fiber beds, composed of cotton and glass wool and cotton
and Dynel, were found to be efficient coalescing media for secondary emulsions. The fiber sizes varied between 8 to 46 µm.

Magiera [48] studied the effect of surface energy of fibers of an octanol/water system. The glass, stainless steel and Teflon (PTFE) were used the study. It was reported that, the glass and stainless fiber coalescer showed good separation efficiency but hydrophobic coalescer made of Teflon fibers did not result in a good separation. The separation efficiency declined with decrease in surface energy of fibers. It was observed that smaller drops do not adhere to fibers and larger drops wet the surface very well. It was also observed that droplets were only in contact with the glass and stainless steel fibers.

Basu [27] studied the water-in-oil and oil-in-water coalescence on glass and Teflon disks. He concluded that contact angles of about 30° of a dispersed phase in presence of continuous phase favors the coalescence of dispersed phase and forms a thin film at the downstream surface of a disk filter. The contact angle between 50° and 90° of dispersed phase in the presence of a continuous phase, resulted in the disk being partially wetted by the dispersed phase and a thin film was collected at the downstream surface leaving some of the drops at the surface. If the contact angle of the dispersed phase is 140° or greater then no coalescence was observed on the surface. Basu recommend that materials of appropriate wettability can be selected to achieve higher coalescence efficiency.

Kocherginsky [49] studied the effect of hydrophilic and hydrophobic membranes on demulsification of water-in-oil emulsion containing surfactants. It was observed that
hydrophilic membranes were more efficient than hydrophobic membranes for separation of emulsion containing surfactant di-2-ethylhexyl phosphoric acid.

Clayfield [50] studied the effect of wettability of different silane coating on glass fibers on coalescence of oil-in-water system. The coalescence efficiency of silane coatings of different surface energy (16 to 39mN/m) were studied. It was found that coalescence efficiency of similar magnitudes of surface energy silanes differed significantly. It was concluded that coalescence efficiency can not be correlated with surface energy. The difficulty in comparison of the results of coalescence performances was the available fibers were different in fiber size, surface roughness and porosity.

Bitten [51] studied the coalescence of water drops in aviation fuels on single fibers perpendicular to flow based on photographic observation. The untreated glass, Teflon, nylon and Decron fibers were used in the study. The rate of growth of droplets was much higher for glass fibers as compare to plastic fibers. It was also concluded that rate growth of water droplets and size of drops produced on plastic fiber was small and plastic fibers were not useful for practical separation of water from fuel.

Shin and Chase [1] found that the addition of small amount of polymeric fibers improves filter performance, however higher wetting fiber gives better filter coalescence but also cause an increase in pressure drop. The addition of optimal amount of hydrophobic submicron sized polystyrene fibers showed improvement in capture efficiency without increasing the pressure drop [26].

Moorthy [24] studied the effect of surface energy of silane coated glass fibers on coalescence filtration of dispersed water drops in Viscor 1487 oil. The filters of glass
fibers were coated with APTS, CES and FTS silanes. The coalescence efficiency of different silane coated filters were compared with uncoated glass filter media. The APTS coated filter media showed the highest coalescence efficiency and also higher performance. It was reported that intermediate wettability gives best filter performance.

Bansal [52] studied the effect of fiber bed properties on oil-in-water emulsion separation and filtration performance. The effect of pore size, permeability, wettability and porosity on filter performance were investigated. The wettability of fibers were varied by using different hydrophilic and hydrophobic coatings. The nano particles of 25nm were attached to the fibers to achieve certain degree of surface roughness. It was concluded that, at similar surface energies, the pore size has a significant impact on filter performance. The smaller pore size gives better separation. The coalesce should be preferably wetted by dispersed phase to achieve better droplet capture and coalescence. The coalescence efficiency depends on influent droplet size, residence time of drops in filter media, surface wettability, pore size, fiber size and porosity of the medium.

Some researchers suggested that the superabsorbent materials like polysiloxanes and polyester are useful for separation of water or oil from organic compounds [53]. The oleophilic filter comprised of glass fibers and Teflon fibers is efficient for separation for oil-in-water dispersions [54].

2.5. Effect of interfacial tension on coalescence filtration

The molecules at the surface of a liquid and in the bulk of the liquid experience attractive forces to the surrounding molecules. The forces around molecules in the bulk of
a liquid are balanced due to the symmetry of the same molecules surrounding a given bulk liquid molecule. However, molecules at the surface of the liquid experience asymmetry of forces due to different molecules in the gas above the surface compared to the bulk molecules of the liquid. The surface tension is a measure of force experienced by liquid molecules at the surface required to bring the asymmetric forces into equilibrium. In emulsions and dispersions, when two liquids are present, the interfacial tension between two liquids is a measure of the attractive force between each phase for its own species. The each phase is repelled by the other phase at the two-liquid interface. Thus, interfacial tension is the surface energy per unit area or force per unit length (dynes/cm). It is also defined as work required to create additional surface area.

2.5.1. Surfactants

Surfactants have molecules containing polar heads (hydrophilic) and non-polar tails (hydrophobic). When added to the liquid fuels, the surfactant molecules orient in a way that hydrophilic head attach to the water molecule and hydrophobic tail to the hydrocarbon molecule. The effect is to expand a surface area between the water and hydrocarbon phases, and in effect, reducing the interfacial tension.

The surfactant molecules orient at the water interface and form a monolayer. Once the monolayer of surfactant molecules is saturated, additional molecules of surfactants aggregate and form micelles. This is known as critical micelles concentration (CMC). After this stage, addition of surfactant does not reduce the interfacial tension and
remains constant. The interfacial tension determines the stability of emulsion. The interfacial tension below 20 dynes/cm generates stable emulsion [6, 7].

2.5.2. Effect of surfactants on coalescence filtration

Surfactants added to the liquid fuels and hydrocarbons reduce the interfacial tension of the liquid. Although the topic is of significant importance very limited literature and data is reported in literature.

Wines [6, 7] studied the effect of surfactants on industrial scale coalescers. The surfactant molecules can coat the surface of the fibers and change the wettability of the fiber surface. This causes an inefficient running of coalescence filters and leads to shorter life and frequent replacement of coalescence filters. Subsequently, higher operating and disposal cost. Wines proposed a polymeric coated media having selective wettability for high water efficiency and lowered effect of surfactants.

Dahlqvist [55] studied the effect of different surfactants on thin and thick filters with different pore sizes. It was found that thick filters with smaller pores perform better as compare to filters with larger pores. The rate of coalescence decreased after the addition of surfactants and the effect was enhanced with deeper filter media.

Chen [56] studied the stability of diesel emulsions using sorbitan monooleate (SM) emulsifier. The variables including emulsifier dosage, ratio of oil to water, temperature, stirring intensity and mixing time were studied. The results show that
optimum conditions are emulsifier dosage of 0.5 %, oil to water ratio 1:1, temperature 30 °C and stirring intensity 2500rpm.

Giribabu [57] studied the adsorption of nonionic surfactants at air-water and water-toluene interfaces and its effect on coalescence time of air-bubbles and water droplets. The three surfactants Tween 20, Triton X-100 and Span 80 were used in the study. The interfacial tension was found decreased with addition of surfactants and reached a saturation value. The time of coalescence increased with increase in concentration of surfactants.

Bessee and Hutzler [58] reviewed the effect of additives and biodiesel blends on water separation efficiencies of coalescers and separators for diesel and ultra low sulfur diesels. It was reported that additives in major diesel fuels can reduce the separation efficiencies significantly. The coalescer technology, on average, has higher separation efficiency than other available technologies.

Hughes [59] attempted a study of effect of wettability of fibers on separation of water from aviation fuels when surfactants were present. The growth of water droplets were observed. The observations reported that trend in fiber wettability as function of surfactant exposure do not follow currently proposed mechanism of droplet capture such as interception and attachment of water drops. It was also concluded that observed coalesce failures involves droplet release stage rather than interception.

Schnable [60] and Edmondson [61] reports that filter media with hydrophobic properties was beneficial to improving separation efficiency of water from diesel,
kerosene, jet fuels and hydrocarbons containing strong surfactants. The hydrophobic fiber materials resist the surfactant coating and efficient coalescence was obtained.

2.6. Electrospinning

Electrospinning is a simple process that uses an electric field to make polymer nanofibers of submicron and nanometer sizes. In electrospinning, solid fibers of small diameters are produced as a result of stretching of the electrified jet [62]. Generally, the suspended drop starts to stretch and forms a Taylor cone as a result of an electric field being applied to the solution [63-64]. A droplet at the tip of the syringe needle, when electrified, experiences two types of electrostatic forces- the electrostatic repulsion between the surface charges and the Coulombic force by external electric field [62]. The distortion of this solution drop is caused by the balance of the repulsive forces induced on the drop due to the charge distribution and the surface tension of the liquid. When the voltage reaches a critical value, the electric force overcomes the surface tension and viscoelastic forces of the deformed drop in the suspended polymer solution formed at the tip of the syringe, and a jet launches [64, 65]. The electrified jet undergoes stretching and bending, eventually forming into a continuous thread. After the jet travels through the air, the polymer fibers are produced by the evaporation of the solvent and are collected at an electrically grounded target. The surface morphology of the electrospun fiber is affected by many parameters, such as the polymer concentration, applied voltage, spinning distance, air friction, gravity, temperature, and ambient parameters [62-71]. In electrospinning, the spinning of fibers is achieved largely by the charges placed on the jet
by the applied field, which move with the jet. A schematic of the electrospinning process is shown in Figure 2.4.

Figure 2.4. Schematic of single jet electrospinning.

The electrospinning setup mainly consists of a syringe, a high voltage power supply and a collector. The polymer is usually dissolved in suitable solvent. Nanofibers in the range of 10 to 2000 nm diameter can be achieved by choosing the appropriate polymer solvent system [62,73]. The syringe is filled with the polymer solution. A syringe pump is used to pump the solution at a constant flow rate. The polymer solution is electrified by applying a high voltage, usually in the range of 1-30 kV. The fibers are deposited onto a collector which is grounded. The diameter of the deposited fiber can be
controlled through electrospinning parameters like solution viscosity, applied voltage, distance between the syringe needle tip and collector surface and many others [62].
CHAPTER III

HYDROPHILIC/HYDROPHOBIC FIBER MEDIA DEVELOPMENT

3.1. Introduction

The objective of the work is to develop coalescing filter media by mixing hydrophilic and hydrophobic fiber materials. This chapter focuses on development of filter media using hydrophilic and hydrophobic materials in different geometries and compositions. The materials used, filter preparation techniques developed for different types of filter media and geometries are described.

3.1.1. Materials and their properties

The materials used in the filter preparation, their properties and source are shown in Table 3.1. The fiber materials were selected based on their surface properties, fiber diameter and their compatibility with Viscor oil and water. The fibers selected are commonly used in the filtration industry for different liquid-liquid separation applications.
Table 3.1. Materials used for filter preparation.

<table>
<thead>
<tr>
<th>Material</th>
<th>Fiber size (µm)</th>
<th>Specific gravity</th>
<th>Melting point, °C</th>
<th>Surface nature (Contact angle, deg)</th>
<th>Source/ Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microglass</td>
<td>2-5</td>
<td>2.500</td>
<td>600</td>
<td>Hydrophilic (0 deg)</td>
<td>Hollingsworth &amp; Vose</td>
</tr>
<tr>
<td>Polypropylene short cut</td>
<td>~7</td>
<td>0.900</td>
<td>166</td>
<td>Hydrophobic (104 deg) [74]</td>
<td>Minifibers Inc.</td>
</tr>
<tr>
<td>Polyester short cut</td>
<td>~7</td>
<td>1.380</td>
<td>247</td>
<td>Hydrophobic (80 deg) [75]</td>
<td>Minifibers Inc.</td>
</tr>
<tr>
<td>Polypropylene nanofibers</td>
<td>300nm to 1000nm</td>
<td>0.900</td>
<td>166</td>
<td>Hydrophobic (&gt;150 deg)</td>
<td>Electrospinning (In lab)</td>
</tr>
<tr>
<td>FTS silane ((heptadecafluoro-1,1,2,2-tetra-hydrodecyl)trichlorosilane)</td>
<td></td>
<td></td>
<td></td>
<td>Hydrophobic (104 deg)</td>
<td>Gelest Inc.</td>
</tr>
</tbody>
</table>

Nonwoven filter media typically have binders (glues) to hold the fiber structure together. Being of similar chemical materials, the binders often have surface properties similar to the fibers that they stick to which aids in the construction of the medium and its wetting properties. While making filter media with combination of two different types of fibers with different surface properties, the binders suitable to each type of fiber materials.
can be used [76, 77]. The amounts of binders blended with the fibers are in proportion to the amount of each fiber type. Because the fibers are well blended, when the binders glue the fibers together and the two types of fibers become interlocked, forming a continuous filter medium structure.

Megasol S50 (Wesbond Corporation, WV) and polyethylene synthetic pulp fibers (Minifibers Inc.) were used as binder materials for microglass and polymeric fibers respectively.

3.1.2. Vacuum molding process

A schematic of the vacuum molding process for forming the filters is shown in Figure 3.1. The filter mold of 2.25cm diameter was used to construct a disk shape filter media. This filter mold was attached at the bottom of the vacuum molding tank and connected to vacuum pump through a storage tank. The desired amount of fiber slurry was transferred into the vacuum molding tank. Constant mixing or agitation was applied to keep fibers in the slurry dispersed. The vacuum pump was operated at 100 to 500 mm of mercury pressure. The slurry was sucked with moderate vacuum pressure through a fine mesh screen and collected in storage tank. A filter paper was placed on metal screen to collect the fibers. The fibers settle and cake builds up on the filter paper and the water passes through. The wet filter cake was removed from the molds. The slurry collected in the storage tank was drained out to empty the tank.
3.1.3. Glass fiber filter media preparation

The B-glass fiber media are commonly used in filtration industry for many different filtration applications including liquid-liquid coalescing filters for separation of water from fuels and hydrocarbons. The selection of fiber size is important for specific applications and is generally proportional to the range of particle size to be separated.

Figure 3.1. Schematic of vacuum molding set-up.
from continuous phase fluid. For high efficiency liquid-liquid filtration, micron and submicron sized fibers are preferred.

The glass fiber media was prepared using the Vacuum molding process. The steps involved in the preparation of glass fiber media are as follows.

• Preparation of slurry:

First, 0.5 grams of microglass fibers of 2-5 microns were soaked in 1 L of filtered water for about 20 min. The soaked fibers were blended in a mechanical blender to make a dispersed fiber slurry. The slurry was then transferred into a mixing tank and diluted to 3 L of water and well dispersed using a mechanical stirrer for 20 min. The Westar+ starch of 0.015 gm was added to slurry with continued stirring of 5 min. The 0.5 ml of Megasol S50 binder was added to slurry and stirred for an additional 5 min.

• Vacuum molding

The slurry of glass fibers was prepared with binder and was vacuum filtered on a filter paper and fine mesh through the 2.25 cm filter mold.

• Heating and drying

The filter media was removed from the filter mold when wet and then heated in oven at 120 °C for 2 hrs.
3.2. Different geometries of filter media

3.2.1. Mixed hydrophilic/hydrophobic filter media

In a mixed hydrophilic/ hydrophobic fiber media, the two fiber types are well blended or mixed so that within a small sample volume of the filter medium both types of fibers are present, as indicated in Figure 3.2.

![Figure 3.2. Mixed hydrophilic/hydrophobic fiber media.](image)

The first challenge encountered in making mixed fiber media was the preparation of a mixed fiber slurry. As specific gravity of glass fibers is higher than water and because of their hydrophilic nature, they submerge and easily disperse in water using a mechanical stirrer. However, the PP fibers are hydrophobic and are less dense than water. The PP fibers tend to float on the surface of the water. The mechanical stirring of several hours was unsuccessful in blending both types of fibers in water. In addition, once stirring was stopped, the hydrophobic (PP) fibers easily separated from the glass fibers and floated to the water surface. The hydrophobic PP fiber slurry required longer agitation times than the glass fibers because air trapped among the fibers and the fibers tended to form clumps in water.
A second challenge was encountered during the vacuum molding process. As happened in the slurry preparation, the hydrophobic fibers floated on the water surface as soon as they were transferred into the vacuum molding tank. The vacuum process produced media as a stack of two separate layers of glass and polypropylene fibers. Continuous agitation within vacuum molding tank was useful in partially dispersing the hydrophobic fibers with the glass fibers but a better operation was needed. With a mixture of hydrophobic and hydrophilic fibers, the hydrophilic fibers always ended up as a layer on the bottom of the filter sample in the mold.

To achieve better mixing of fibers and simultaneous vacuum filtration of both types of fibers, the vacuum molding process was modified with a centrifugal pump to circulate the slurry back into the slurry tank as shown in Figure 3.3 and Figure 3.4 (a). Figure 3.4 (b) shows the storage tank to collect the vacuum filtered slurry and Figure 3.4 (c) shows the filter mold. The centrifugal pump (March MFG., Inc, HP=1/40) was placed so that part of the circulated slurry flowed through the mold while the rest of the slurry cycled back to the mixing tank.
Figure 3.3. Schematic of vacuum molding assembly modified with centrifugal pump.
Figure 3.4. (a) Vacuum mold assembly with centrifugal pump, (b) Storage tank with vacuum pump (c) filter molds with 2.25 cm hole inside diameter.

To prepare a mixed fiber filter media two separate slurries of glass and polypropylene fibers with desired composition were prepared in 2.5 liter of water. The glass fiber slurry was prepared with starch (ratio of 0.03 gram starch per 1 gram of glass fiber) and Megasol S50 binder (Wesbond Corporation) (ratio of 1 ml of Megasol per 1 gram of glass fiber). The polypropylene fiber slurry was prepared with polyethylene
synthetic pulp fibers (Minifibers Incorporated) (ratio of 0.20 gram polyethylene fibers per 1 gram of polymer fiber) as a thermal binder. First, the hydrophobic fiber slurry was transferred into the vacuum mold tank and circulated using the pump. After about 3 minutes of circulation, the vacuum was applied and the glass fiber slurry was added to the tank with a flow rate of about 150 ml/min. This is achieved through multiple trial and error experiments and manual observation of near uniform mixing of both types of fibers. Care was taken to not operate the centrifugal pump without any slurry. After vacuum molding, the wet filter was taken out from the mold and heated at 125 °C for 3 to 4 min. to initially set the polyethylene thermal binder. The filter samples were next heated in an oven for 2 hrs at 120 °C for drying and to complete setting of the binders. The SEM image of mixed filter media before and after heating at 125 °C is shown in Figure 3.5. The polyethylene binder melts after heating and helps in binding polypropylene fibers inside the filter media.

Figure 3.5. SEM image of mixed glass and polypropylene media (a) before heating (b) after heating.
The filter media were prepared with different compositions of glass and polypropylene fibers i.e. glass fiber composition of 80%, 60%, 40%, 20%. The filter media were disk shaped having 2.25 cm diameter. Each medium consisted of 0.5 grams of fibers and binder material was added in proportion to each fiber type.

3.2.2. Layered hydrophilic/hydrophobic filter media

A second approach to make the hydrophilic/hydrophobic filter media with varying wetting properties was by layering thin mats of fibers of different types. This is shown in Figure 3.6. With this type of media, experiments were carried out with flow perpendicular to the each layer. The layered media had a jump in wettability at the interface of two different layers. The layered media can also be prepared with varying composition of fibers to achieve media with different wetting properties. The thickness of each layer varied with the amounts of fibers to obtain the fiber ratio compositions of filters. Layered hydrophilic/hydrophobic media were constructed using vacuum mold process.

Figure 3.6. Layered hydrophilic/hydrophobic filter media.
Two separate slurries of glass fibers and polypropylene fibers were prepared with recipes similar to those described previously for the mixed fiber filter media. Approximately 250 ml of each slurry were alternately vacuum filtered through a fine mesh screen in the filter mold 2.25 cm diameter, to build up the filter in thickness of alternating layers of glass and polypropylene fibers.

In each filter sample, the exit or outlet layer was a layer of glass fibers and the inlet layer was a layer of polymer fibers. The filter samples were constructed of 10 equal thickness layers of glass fibers and 10 equal thickness layers of polymer fibers. By varying the thicknesses of the respective glass and polymer fiber layers the media were prepared with varying glass fiber compositions (80%, 60%, 40%, and 20%). Each filter sample contained about 0.5 g total of fibers and the filter thicknesses varied between 1.0 cm to 1.2 cm with variations in the fiber layer thickness between 0.4 mm to 0.6 mm, depending on the mass of fibers.

The wet filter samples were removed from the mold and a thin layer of binder was applied on the outside cylindrical surface of filter to hold all the fiber layers in place. This was done carefully without disturbing the disk shape of the filter media. The wet filter samples were heated at $125^\circ$C for 3 to 4 min. to initially set the polyethylene thermal binder. The filter samples were subsequently heated in an oven for 2 hrs at $120^\circ$C for drying and to complete setting of the binders.

Similarly, layered media of glass and polyester (Minifibers Inc.) fibers were prepared in different compositions of glass fibers (80%, 60%, 40%, 20%).
3.2.3. Radially layered hydrophilic/hydrophobic filter media

A third way to construct media of varying wetting properties was by making layers such that the fluid flow was parallel to the boundary between the layers. The media are constructed with thin mats, so that all layers are aligned in circular or spiral pattern as shown in the Figure 3.7. With these media the fluid flows parallel to the cylinder axis which is parallel to the boundaries between the layers. Similar to layered filter media, this type of media has a jump in wettability at the interface between different layers.

Figure 3.7. Radially layered hydrophilic/hydrophobic fiber media.

To prepare radially layered filter media, thin composite mats of hydrophilic and hydrophobic fibers were prepared using a lab scale sheet former as shown in Figure 3.8. The sheet former (Labtech Instruments Inc.) operates on a simple phenomena of gravity settling. The sheet former assembly consists of a cylindrical slurry tank, 6 inch metal screen (standard 150 mesh opening), filter sheet mold, drain valve and drain valve handle, and a storage tank (sump) at the bottom of the assembly connected to drain valve.
using a pipe. The slurry tank has an additional openings and pipes at the sheet mold to manage the overflow of the slurry. The slurry tank has a latch to seal the tank with sheet mold. It has a rest to support the tank when it is tilted in the opened position. The sheet former has additional components including manual stirrer, two 6 inch diameter metal couch plates coated with Teflon, and a roller (couch roll) to press the fiber sheet.

Figure 3.8. Lab scale sheet former and components: (a) Sheet former assembly, (b) Slurry tank with metal screen, (c) two metal plates and metal screen.
The fine metal screen of 6 inch diameter is placed on the sheet mold and tank is sealed with latch. The required amount of slurry is poured into the slurry tank. The manual stirrer is used to disperse the fibers in the tank. The slurry is drained by opening a drain valve using drain valve handle. The fibers are collected on the screen and water passes through. The water is collected in the sump at the bottom of the assembly and then drained out in a sink. The sheet of fibers is formed on a metal screen. The metal screen is taken out from the tank. The wet fiber sheet is transferred to one of the Teflon coated metal plates and the second metal plate is placed on top of the wet fiber sheet. The fiber sheet between the two metal plates is pressed using a roller. The pressing of fiber sheet can alter the porosity and permeability of sheet and therefore use of it is based on the user’s requirements. The Teflon coating help prevent the sheets from sticking to the plate and it facilitates easy removal of the sheets from screen after pressing. The fiber sheet is subsequently transferred onto a filter paper and heated in oven.

Two separate slurries of glass fibers and polypropylene fibers with desired composition were prepared in 2.5 L of water. Similar recipes were used as described previously with similar proportions of binders. First, the glass fiber slurry was transferred into the cylindrical slurry tank of the sheet former and second the polypropylene slurry was added. The mixed slurries were kept in the tank without any mixing for 5 minutes so that polypropylene fibers separate from the glass fibers and float toward the water surface. The drain valve was opened and fibers were settled on the metal screen. The fiber sheet was taken out and heated in oven at 125 °C for 1 to 2 minutes to initially set the polyethylene thermal binder. The filter samples were then heated in an oven for 1 hr at 120 °C for drying and to complete setting of the binders. The slurry recipes were
modified different compositions of glass and polypropylene fibers to create sheets of fibers with varying glass fiber compositions of (80%, 60%, 40%, 20%). Each composite sheet was composed of 2 grams of total fibers and thickness of each layer is varied as amount of fibers in each sheet varied. Figure 3.9 shows the images of prepared composite sheets. Figure 3.9(b) shows water drops bead up on the hydrophobic side of the sheet. The drops bead up at different locations of fiber sheets which shows that the distribution of hydrophobic fibers is uniform. The water drops do not pass through from the hydrophobic side of the sheet. When applied to the hydrophilic side of the sheet the water drops are absorbed by the fibers.

Figure 3.9. (a) Prepared composite glass and polypropylene fiber sheet, (b) water drops (colored red with food coloring) form beads with high contact area on the hydrophobic side of the sheet.

These fiber sheets were cut into strips of 9cm length and 1 cm wide. A total of five strips from the same sheet were manually rolled to create a radially layered media
with 2.25 cm diameter. The weights of the strips was varied between 0.119 grams to 0.126 grams. The amount of fibers in the filter media were proportional to the amount of fibers in original sheet. Thus, the filter media were constructed with different compositions of glass and polypropylene fibers, i.e. the amount of glass fibers in filter media (80%, 60%, 40%). Similarly, radially layered glass and polyester fiber filter media were prepared in different compositions.

3.3. Hydrophobic electrospun fibers mixed filter media

The filter media were prepared by mixing polypropylene electrospun fibers with glass fibers. The polypropylene fibers were generated using a single jet electrospinning process. The polypropylene fibers were prepared in different fiber sizes (i.e. average diameters of 300nm, 600nm, 900nm) with variation in polypropylene solution concentration. These polypropylene electrospun fibers were added to glass fiber media in different area ratios (0.5, 1 and 2), where area ratio is defined as

\[
\text{Area ratio} = \frac{\text{Area of nanofibers}}{\text{Area of glassfibers}} \quad (3.1)
\]

where area of fibers are calculated by

\[
A = \frac{m}{\rho} \times \frac{4}{d_f} \quad (3.2)
\]

and where \(m\), \(\rho\) and \(d_f\) are the mass, density and diameter of the fibers in the filter medium.
3.3.1. Electrospun Polypropylene fibers

The polypropylene (syndiotactic, Mw 174 000, Melt index 2.2g/10min (230 C/2.16kg ASTM D 1238)) was dissolved in Cyclohexane/Acetone/N-Dimethylformamide (DMF) by weight (8/1/1) and heated in oven at 70 °C to make 1%, 2% and 3% PP solutions [78]. Syringes, tubings and needles were heated in oven at 70 °C for 30 minutes before electrospinning.

The syringe was filled with PP solution and pumped, using syringe pump (WPI, Model Sp101i), at a flow rate of 25 ml/hr. The syringe was connected to standard hypodermic 21 gauge needle through 1/8” Teflon tubing. The needle was charged at 25 KV using a power supply (Gamma High Voltage, Model D-ES30PN/M692). The grounded collecting surface to collect the charged fibers was placed at 25 cm from the tip of the needle. The lab-scale Electrospinning set-up is shown in Figure 3.10. The average fiber sizes for 1%, 2% and 3% PP solutions were 300nm, 600nm and 900nm respectively. The SEM images of the polypropylene nanofibers are shown in Figure 3.11.
Figure 3.10. Electrospinning set-up used for making PP nanofibers.

Figure 3.11. SEM image of polypropylene electrospun fibers. The fiber mats had some beads as shown in this image.
3.3.2. Filter media preparation

The glass fiber slurry was prepared with 0.5 grams of glass fibers in 2.5 L of water with starch (ratio of 0.3 gm of starch per 1 g of glass fibers) and Megasol S50 binder (ratio of 1ml of Megasol S50 per 1 g of glass fibers) (Wesbond Corporation). The desired amount of PP electrospun fibers was chopped in a mechanical blender with 0.5L water and mixed with glass fiber slurry. The mixed slurry was vacuum filtered through a fine mesh screen in a mold with inside diameter 2.25 cm, to build a filter cake. The mixed slurry was continuously stirred to ensure that both type of fibers were vacuum filtered simultaneously. The wet filter samples were removed from the mold and heated in oven for 2 hrs at 120 °C for drying and setting of binder. The SEM image in Figure 3.12 shows the polypropylene nanofibers mixed with glass fibers.

![SEM image of polypropylene nanofiber mixed glass fibers in filter media.](image)

Figure 3.12. SEM image of polypropylene nanofiber mixed glass fibers in filter media.
3.4. Mixed media with silane coated fibers

The silane coupling agents were used to modify the wettability or surface energy of the fiber surfaces without changing the structural properties of the media (fiber diameter and packing). The silanes formed mono or multilayer coatings on the surface. Moorthy (Moorthy K., MS thesis, Effect of Surface Energy of Fibers on Coalescence Filtration, The University of Akron 2007.) used three different silanes to vary the wettability of the glass fiber filter media. The FTS ((heptadecafluoro-1,1,2,2-tetra-hydrodecyl)trichlorosilane) silane was used in this work to lower the surface energy of the fibers.

The coupling mechanism of the organofunctional silanes depends on a stable link between the organofunctional group (Y) and hydrolyzable groups (X) in compounds of the structure X₃SiRY. The organofunctional groups provide the reactivity with the polymer; the hydrolyzable groups are intermediates that form the silanol groups for bonding the mineral surfaces [24].

The covalent bond between the FTS molecule and the glass is relatively stable. Based on the coating time variation in wettability can be achieved.

3.4.1. Vapor phase deposition

A glass fiber sheet of 2 grams of fibers were prepared using a lab scale sheet former without any binder material. The formed sheet was cleaned under a stream of N₂ gas. The glass fiber sheet was cut into a 10 cm x 10 cm square and cleaned with the stream of N₂ gas a second time. The mixture of silane solution was prepared in a glass
Petri dish with 3 gm of paraffin oil and 200 µl of FTS silane, which was placed in a desiccator. The glass fiber sheet was suspended above the silane solution and the desiccator was closed. A glass slide was twice cleaned with deionized water and acetone, heated in an oven at 120°C, and cleaned under N2 stream. The glass slide was placed in a desiccator along with fiber sheet. The glass fiber sheet and the glass slide were in the desiccators together for a deposition time of 1 hr.

3.4.2. Contact angle measurement

After the coating, the contact angles for water and viscor oil on the coated glass slide were measured using a drop shape analyzer (Kruss Instruments, Germany). The average contact angle values on coated glass slides were 106° (±3.72) and 66° (±3.34) for water and Viscor oil respectively. The reported contact angle values are the average of four experiments with ± one standard deviation and each experimental value was the average of six measurements at different locations of glass slide. Example images of drops from which the contact angles of water and viscor oil were measured are shown in Figure 3.13.

3.4.3. Filter media preparation

The filter samples were prepared with different compositions of coated and uncoated glass fibers (i.e. the ratios of uncoated glass fibers to total fibers were 80%, 60%, 40%, and 20%. The slurries of desired amounts of coated and uncoated glass fibers
were prepared in 3 liter of water. For 0.5 grams of total fibers 0.015 grams of starch and 0.5 milliliters of Megasol S50 was added. The mixed slurry was vacuum filtered through a fine mesh screen. Wet filter samples were heated at 120°C for 2 hrs.

Figure 3.13 Contact angle of (a) water and (b) Viscor oil on FTS coated glass slide.
CHAPTER IV
FILTER MEDIA CHARACTERIZATION AND MODIFIED WASHBURN’S EQUATION

This chapter focuses on characterization of filter media for porosity, permeability, and wettability. The objective is to develop and apply an appropriate technique for characterization of wettability of the filter media. The Washburn’s method, based on capillary rise of liquid in porous media, is used to characterize the filter media. The wettability of the filter media is expressed as Lipophilic to Hydrophilic (L/H) ratio. The water and Viscor oil are used as test liquids and L/H represents the relative wettability of filter media with water and Viscor oil. This chapter covers the wettability characterization technique, the experimental results of wettability characterization of filter media with different geometries and compositions, and the effect of porosity and permeability on filter media performance.

4.5.1. Wettability of fibrous filter media

The wettability of a solid surface was used to be generally characterized by the contact angle [45]. However, it is difficult to observe and reproduce contact angles of a liquid drop on the porous surface of a fibrous filter medium. In fibrous filter media,
wettability is a relative adhesion or spreading of two immiscible fluids on solid fiber material. When two immiscible phases are present, then wettability of the media is a measure of preferential wetting of one liquid relative to other liquid [79, 81]. Wetting is a complex process because a fibrous medium is composed of numerous tiny pores formed by solid fibers. A more appropriate characterization method is the rate of a liquid drawn into the porous medium due to capillary forces. The approach of Washburn’s method idealizes the filter media is bundle of parallel uniform cylindrical capillaries and the liquid penetration into the pores of the porous media is related to the contact angle of the liquid in the pores [82, 99]. The filter sample is suspended above the test liquid so that the bottom surface of the filter is in contact with the free surface of the liquid. The test liquid is placed on weighing balance and the initial decrease in weight on balance ensures the filter surface is in contact with the surface of the liquid. Capillary forces pull liquid upward into the filter medium. The liquid penetration is related to the contact angle of the liquid. Many authors have shown the usefulness of the Washburn’s equation for the characterization of porous media, powdered solids, woven and, non-woven fabrics, porous foods, filter media of sand particles for wastewater treatment and fibrous media [82-94]. The modified Washburn’s approach for the characterization of wettability of fibrous filter media is explained below.

4.5.2. Modified Washburn’s equation

By assuming steady-state laminar flow of a Newtonian fluid in a single capillary tube and neglecting inertial effects, the Poiseuille’s law gives the rate of liquid
penetration in the capillary. The Poiseullie’s law for flow of liquid through a porous medium can be written as

\[
\frac{dV}{dt} = \frac{\pi r_{ef}^4 \Delta P}{8\eta h} \quad (4.1)
\]

where, \( h \) is height of penetrating liquid, \( \eta \) is viscosity of liquid, \( r_{ef} \) is effective capillary radius and \( \Delta P \) is pressure drop.

The pressure drop in a capillary is the difference between the capillary pressure \((P_c)\) and hydrostatic pressure \((P_h)\).

\[
P_c = \frac{2\gamma}{r} \cos \theta_A \approx \frac{2\gamma}{r_{ef}} \cos \theta_A \quad (4.2)
\]

Where, \( r = r_{ef} \)

\[
P_c = \rho gh \quad (4.3)
\]

Where, \( \theta_A \) is the advancing contact angle of the rising liquid in pores and \( \rho \) is the density of the liquid. As liquid is moving in upward direction due to capillary pressure, the hydrostatic pressure can be neglected.

The height of liquid and volume of liquid is related as

\[
dV = \pi r_{ef}^2 dh \quad (4.4)
\]

Substituting equation 4.2 and 4.4 in to equation 4.1, the equation becomes,

\[
\frac{h}{\eta} \frac{dh}{dt} = \frac{r_{ef} \gamma \cos \theta_A}{4\eta} \quad (4.5)
\]
Integrating the above equation gives the Washburn’s equation which relates the height of penetrated liquid, $h$, at time $t$, in the filter medium to the advancing contact angle, $\theta$, of the liquid rising in the pores.

$$h^2 = \frac{r_{\text{eff}} \cos \theta \gamma t}{2\eta}$$  \hspace{1cm} (4.6)

where, $\eta$ is the viscosity of the penetrating liquid, and $\gamma$ is the surface tension of the penetrating liquid.

Real fibrous filters are made up of non-uniform pore structures and therefore, the pores have a distribution of radii that effect the capillary rise in a complicated way due to the interconnectivity, shapes, and sizes of the pores. In Washburn’s equation, $r_{\text{eff}}$ is an effective capillary radius defined as the pore radius of a medium made of uniform capillaries that has the same performance as observed of the real medium. The effective radius is difficult to determine and leads to significant error in the penetrating rates of liquid [88], however it can be lumped with other constant values in the equation during the analysis.

The height of liquid penetration into the filter medium is related to the mass of penetrating liquid given by

$$w = \varepsilon \rho A h$$  \hspace{1cm} (4.7)

where, $\varepsilon$ is the porosity of the filter medium, $A$ is the cross-sectional area of the filter medium, and $\rho$ is the density of the liquid.

Combining equations 4.6 and 4.7 we get,
\[ w^2 = \frac{c \rho^2 \gamma \cos \theta_A t}{\eta} \]  \hspace{1cm} (4.8)

where the lumped constant,

\[ c = \frac{\tau_{eff} \epsilon^2 A^2}{2} \]  \hspace{1cm} (4.9)

can be determined when one of the liquids totally wets the surface (i.e., zero contact angle). From the slope of \( w^2 \) versus \( t \) and knowing the liquid properties, the value of \( c \) can be computed.

Once the lumped constant is found for a total wetting liquid, it can be used for other liquids from their measured slopes, \( S = \frac{dw^2}{dt} \), of the curve \([24, 87]\). The slopes are related to the advancing contact angle by

\[ S = \frac{c \rho^2 \gamma \cos \theta_A}{\eta} \]  \hspace{1cm} (4.10)

Equation (4.10) is called the modified Washburn’s equation.

The lipophilic to hydrophilic Ratio \((L/H)\) is defined by

\[ \frac{L}{H} = \frac{\cos \theta_o}{\cos \theta_w} \]  \hspace{1cm} (4.11)

and represents how the medium performs when in contact with a non-polar liquid (e.g. oil) compared to when it is in contact with a polar liquid (e.g. water). The angles, \( \theta_o \) and \( \theta_w \), are the contact angles of oil and water in the filter medium as determined by Washburn’s equation.
Combining equations (4.10) and (4.11) we get,

\[
\frac{L}{H} = \frac{S_o \eta \rho_{o} \gamma_{o}}{S_w \eta \rho_{w} \gamma_{w}}
\]

where, subscripts \(o\) and \(w\) represents the oil and water phase respectively. The lumped constants \(c_w\) and \(c_o\) are equal for the same filter medium and cancel out of Eq. (4.12) and in this analysis, the constant, \(c\), does not need to be calculated. Thus, the \(L/H\) value reflects the wettability of the filter medium. Small values of \(L/H\) indicate the filter medium is hydrophilic and large values indicate the filter medium is lipophilic.

4.5.3. Washburn’s wettability characterization technique

4.3.1. Experimental set-up

In this experiment, a filter medium is suspended using a thin wire attached to a cantilever beam fixed to a scissor stand, the height of which can be adjusted. The schematic of experimental set-up is shown in Figure 4.1.
Figure 4.1. Schematic of experimental set-up for wettability characterization of filter media.

A transparent glass beaker with test liquid is placed on an electronic balance. The liquid temperature is maintained around 23\textdegree{}-25\textdegree{}C. The balance is placed on vibration free, leveled surface. The scissor stand is placed on a rigid support to limit vibrations and achieve controlled motion of the stand in only vertical direction. A stop watch is placed in front of the electronic balance, both of which are in view of a video camera to record time and mass. The experimental set-up is shown in Figure 4.2.
4.3.2. Characterization procedure

The filter medium is lowered very slowly until it breaks the air-liquid interface. When the surface of the filter medium touches to surface of the liquid its position is fixed for the rest of the experiment. The liquid mass in the filter sample due to capillary action is determined from the reduction in liquid mass in the beaker measured by the scale over time. The weight on microbalance with its corresponding time is recorded by the video camera. The experiment is stopped when liquid reaches the top of filter medium or the balance shows a steady value. The duration of experiment varies for each filter media based on its wettability. In a case, when significant amount of liquid is taken by the filter media, then the surface of the liquid can be stretched with the solid surface due to surface tension effects and liquid uptake is reduced which leads to error in measurements. In such
case, surface area of the liquid can be increased to minimize the stretching of liquid at the solid surface.

The test liquids used in this study are Viscor oil 1487 (Rock Valley Oil & Chemical Company) and deionized water. The Viscor oil is a calibration fluid and has similar properties to that of diesel fuel. Physical properties of deionized water and the Viscor oil are presented in Table 4.1.

Table 4.1. Physical properties of reference liquids at 20 °C

<table>
<thead>
<tr>
<th>Liquid</th>
<th>Density (g/cm³)</th>
<th>Viscosity (Ns/m²)</th>
<th>Surface tension (dynes/cm)</th>
<th>Boiling point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deionized water</td>
<td>1.000</td>
<td>0.00102</td>
<td>72.0</td>
<td>100</td>
</tr>
<tr>
<td>Viscor 1487</td>
<td>0.832</td>
<td>0.00207</td>
<td>28.5</td>
<td>160</td>
</tr>
</tbody>
</table>

4.3.3. Factors affecting on wettability

In the modified Washburn’s equation, L/H depends on density, viscosity, surface tension and rate of liquid uptake of both test liquids used. It also depends on porosity, cross-sectional area and effective capillary radius. The wettability of a filter medium characterized by Washburn’s equation is influenced by many factors including porosity and packing density, pore size distribution, surface roughness, fiber size, fiber material, liquid properties and surfactants [84, 93, 97-99]. The pore size distribution of the filter
medium has significant influence on wettability. In addition, during the experimental procedure, the initial contact of the solid surface to the liquid surface affects the liquid penetration due to surface tension of the liquid. Evaporation will change the weight of liquid on the balance but can be neglected for experiments lasting less than a few minutes [89, 95].

4.5.4. Filter media characterization

4.4.1. Porosity

Porosity of the filter, $\varepsilon$, is the ratio of the volume occupied by the pores to the total volume of the filter medium. The porosity varies from zero to one. A good filter medium has a higher porosity that results in a lower pressure drop. The bulk porosity of all the media are measured by using a custom made pycnometer. Figure 4.3 shows the pycnometer and its components. The pycnometer has two chambers connected with a ball valve. Initially the ball valve is closed. The filter sample is loaded in the top chamber and bottom chamber is pressurized to 15 psig. The top chamber is designed to fit thick filter samples and a number of spacers having different thicknesses can be used to fill the remaining gap. The valve between the two chambers is then opened to equalize the pressure in both the chambers. Boyle’s law states that pressure is inversely proportional to volume. Void volume is determined by the difference in pressure in the sample chamber before and after opening the valve. Three different sets of pressure readings are taken for each sample. In first set of readings, three readings are taken with just the spacer in the top chamber. In second set of readings, three readings are recorded with a
calibration cube with known dimensions in the top chamber and in third set of readings are recorded with filter sample placed in the top chamber. The readings in triplicate are averaged. The average values are used to determine the porosity using Boyle’s law.

4.4.2. Air permeability

Permeability is a measure of the ease of a fluid that passes through the filter medium. A number of techniques can be used to measure the permeability. Frazier® Differential Pressure Air Permeability Measuring Instrument (Frazier Precision Instrument Company, Inc.) is used for permeability measurement. Filter media with low fluid permeability typically indicate high pressure drop. Generally, permeability is measured by passing a fluid through a medium at a known pressure drop and observing the flow rate. For fibrous filters, the fluid permeability is given by Darcy’s Law.

\[
\frac{Q}{A} = \frac{Gk \Delta P}{\mu L}
\]  

(4.13)
where \( Q \) = volumetric flow rate

\( A \) = cross section area of the exit surface

\( G \) = shape factor

\( k \) = permeability

\( \Delta P \) = pressure drop

\( L \) = media thickness

In this work, the Frazier® Differential Pressure Air Permeability Measuring Instrument, shown in Figures 4.4 and 4.5, is used to measure air permeability of all filter samples. Air permeability of the filter media has a unit of \( \text{m}^2 \). The filter medium is placed in the sample holder. The filter medium must fit snugly in the sample holder to avoid any air flow along the sides of the sample. Air flow along the sides of the medium will produce erroneous results because the air is not flowing through the medium itself. Different orifice openings are tested and a specific orifice opening is selected to obtain the pressure drop at 0.5 psig as the test is designed to produce accurate result at 0.5 psig pressure drop. Three sets of readings are taken for each sample. The Frazier® Differential Pressure Air Permeability Measuring Instrument is designed to measure air permeability of thin samples where the shape factor is 1. For other samples, the shape factor is read from a correlation and is incorporated in the permeability calculation.
Figure 4.4. Frazier® Differential Pressure Air Permeability Measuring Instrument.

Figure 4.5. Components of Frazier® Differential Pressure Air Permeability Measuring Instrument (a) Sample holder (b) Manometer to read pressure drop (c) Orifice (d) Pump and pump control (e) Different orifice openings.
4.4.3. Strength and hardness

Strength or hardness of the media indicates its structural strength. The filter media need to have structural integrity to minimize the fiber shedding as well as to withstand at high flow rates. Strength is an indirect measure of amount of binder in the medium. A filter medium without a binder is too soft and structurally weak. Hardness of the sample is measured using a durometer (Rex Gauge, Type 0 and Model 1600) as shown in Figure 4.6. The durometer has a scale of 1 to 100 with 100 being the hardest.

![Figure 4.6. Lab scale Durometer.](image)

The filter medium is placed on a hard surface. The pointer at the end of the Durometer is pressed against the surface of the filter medium and the measurement is taken directly from the scale.
4.5.5. Characterization results

The liquid penetration experiments were performed and the mass \( (w) \) of liquid taken by the filter media with time was measured for each medium and for each test liquid. The \( w^2 \) was plotted against the time to obtain the overall wetting kinetics of each medium. All of the filters were characterized in triplicate and plotted as an average of three filters with error bars indicating one standard deviation. The slopes of the wetting curves \( (S_w \) and \( S_o) \) were obtained from initial wetting kinetics data and the L/H values for the filter media were calculated using the modified Washburn’s equation (Equation 4.10).

Reed and Wilson [99] show that the Washburn’s equation is best fitted to the initial kinetic data of the rate of liquid penetration. This is also shown by Yang and Chang [87]. By definition, the L/H value is the ratio of cosines of contact angles of the oil and water phases respectively as shown in Equation (4.11) and assuming contact angle is approximately equal to the advancing contact angle. A value of L/H=1, represents a medium having the same wettability for the water and oil phases. If L/H value is greater than unity, then the medium is hydrophobic and if L/H is less than unity, then the medium is considered hydrophilic. When comparing filter media prepared with hydrophilic and hydrophobic fibers, those with higher L/H value are more preferentially wetted by oil than are media with lower L/H values.

4.5.1. Mixed hydrophilic/hydrophobic fiber media

The mixed glass and polypropylene fiber media were prepared in different compositions. The filter samples are described in Table 2 for their mass, height, fiber
composition, binder content and notations. In the notations, M represents mixed filter media, G represents glass, PP represents polypropylene and ratios of respective fiber materials in percentages are mentioned in bracket.

Table 4.2. Mass, Height and notations of different filter media. The ± range is one standard deviation of the value for the three samples.

<table>
<thead>
<tr>
<th>Filter Sample</th>
<th>Mass of glass fibers (gm)</th>
<th>Mass of polymer fibers (gm)</th>
<th>Mass of binder* (gm)</th>
<th>Total mass of filter (gm)</th>
<th>Thickness of sample (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>0.5</td>
<td>0.00</td>
<td>0.155</td>
<td>0.655 (±0.006)</td>
<td>1.038 (±0.004)</td>
</tr>
<tr>
<td>MGPP(80:20)</td>
<td>0.4</td>
<td>0.1</td>
<td>0.095</td>
<td>0.612 (±0.007)</td>
<td>1.092 (±0.023)</td>
</tr>
<tr>
<td>MGPP(60:40)</td>
<td>0.3</td>
<td>0.2</td>
<td>0.143</td>
<td>0.643 (±0.025)</td>
<td>1.140 (±0.007)</td>
</tr>
<tr>
<td>MGPP(40:60)</td>
<td>0.2</td>
<td>0.3</td>
<td>0.112</td>
<td>0.595 (±0.031)</td>
<td>1.146 (±0.015)</td>
</tr>
<tr>
<td>MGPP(20:80)</td>
<td>0.1</td>
<td>0.4</td>
<td>0.111</td>
<td>0.611 (±0.071)</td>
<td>1.204 (±0.019)</td>
</tr>
</tbody>
</table>

*Calculated by subtracting fiber mass from total mass of filter media

The wetting kinetics for mixed glass and polypropylene media obtained using water and Viscor oil as test liquids and are shown in Figure 4.7 and Figure 4.8 respectively. The water uptake rate for glass fiber media is highest and it decreases with increasing amounts of polypropylene fibers in filter media. Oil uptake rate is lowest for glass only fiber media and increases with the amount of polymeric fibers. The surface energy of glass surface is higher and surface energy of the polypropylene fiber surface is
lower. The water spreads on high energy surface and it shows high contact angle on low energy surface. Combining the Young’s equation (equation 2.1) and equation 4.8, we get

$$w^2 = \frac{c^2 (\gamma_S - \gamma_SL) \tau}{\eta}$$

(4.14)

As glass is high energy surface, the water uptake rate is highest and oil uptake rate is lowest.

The initial wetting kinetics for layered glass and polypropylene media with water and Viscor oil as test liquids are shown in Figure 4.9 and Figure 4.10 respectively.

Figure 4.7. Wetting kinetics of mixed glass/polypropylene media with water as test liquid.
Figure 4.8. Wetting kinetics of mixed glass/polypropylene media with Viscor oil as test liquid.

Figure 4.9. Initial wetting kinetics of mixed glass/polypropylene media with water as test liquid.
Figure 4.10. Initial wetting kinetics of mixed glass/polypropylene media with Viscor oil as test liquid.

The values of initial slopes and calculated L/H for each filter media are shown in Table 4.3. The initial slopes are obtained from linear least square fit to initial wetting kinetic data with a regression coefficient, $R^2$, more than 0.95. The values of initial slopes and $R^2$ for each media are shown in Figure 4.9 and Figure 4.10 for the illustration purpose. The variation in L/H with different fraction of polypropylene fibers in the filter media is shown in Figure 4.11. The L/H of filter media increases with increase in amount of polypropylene fibers in the filter media. The media becomes hydrophobic with increasing amounts of polypropylene fibers. The porosity and air permeability as function of L/H are shown in Figure 4.12 and Figure 4.13 respectively. The porosity and permeability appear to be not to have any effect on L/H, since these quantities are primarily functions of geometry and not surface wettability. The filter samples were
prepared with same amount of fibers and similar vacuum molding process and conditions.

Table 4.3. Initial slopes and L/H values of mixed glass and polypropylene media

<table>
<thead>
<tr>
<th>Filter media</th>
<th>Water</th>
<th>Viscor Oil</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( S_w )</td>
<td>( R^2 )</td>
<td>( S_o )</td>
</tr>
<tr>
<td>Glass</td>
<td>6.792 (±0.789)</td>
<td>0.961</td>
<td>0.429 (±0.040)</td>
</tr>
<tr>
<td>MGPP(80:20)</td>
<td>1.486 (±0.111)</td>
<td>0.984</td>
<td>0.462 (±0.024)</td>
</tr>
<tr>
<td>MGPP(60:40)</td>
<td>1.162 (±0.056)</td>
<td>0.993</td>
<td>0.486 (±0.040)</td>
</tr>
<tr>
<td>MGPP(40:60)</td>
<td>0.441 (±0.052)</td>
<td>0.959</td>
<td>0.849 (±0.060)</td>
</tr>
<tr>
<td>MGPP(20:80)</td>
<td>0.115 (±0.014)</td>
<td>0.959</td>
<td>0.915 (±0.059)</td>
</tr>
</tbody>
</table>

Figure 4.11. Variation L/H with variation in fraction of polypropylene fibers in mixed media
Figure 4.12. Porosity Vs L/H of mixed glass polypropylene media

Figure 4.13. Air Permeability Vs L/H of mixed glass polypropylene media

4.5.2. Layered hydrophilic/hydrophobic fiber media

The layered media were prepared in different compositions of glass-polypropylene fibers and glass-polyester fibers. The mass, height and notations of each filter media are shown in Table 4.4. In notations, L represents layered and PET represents polyester and rest are consistent with mixed filter media.
Table 4.4. Mass, height and notations of different layered media

<table>
<thead>
<tr>
<th>Filter Sample</th>
<th>Mass of glass fibers (gm)</th>
<th>Mass of polymer fibers (gm)</th>
<th>Mass of binder* (gm)</th>
<th>Total mass of filter (gm)</th>
<th>Thickness of sample (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>0.5</td>
<td>0.0</td>
<td>0.289</td>
<td>0.789 (±0.006)</td>
<td>1.049 (±0.004)</td>
</tr>
<tr>
<td>LGPP(80:20)</td>
<td>0.4</td>
<td>0.1</td>
<td>0.235</td>
<td>0.735 (±0.067)</td>
<td>1.092 (±0.023)</td>
</tr>
<tr>
<td>LGPP(60:40)</td>
<td>0.3</td>
<td>0.2</td>
<td>0.136</td>
<td>0.636 (±0.048)</td>
<td>1.140 (±0.007)</td>
</tr>
<tr>
<td>LGPP(40:60)</td>
<td>0.2</td>
<td>0.3</td>
<td>0.120</td>
<td>0.620 (±0.026)</td>
<td>1.146 (±0.015)</td>
</tr>
<tr>
<td>LGPP(20:80)</td>
<td>0.1</td>
<td>0.4</td>
<td>0.187</td>
<td>0.687 (±0.094)</td>
<td>1.204 (±0.019)</td>
</tr>
<tr>
<td>LGPET(80:20)</td>
<td>0.4</td>
<td>0.1</td>
<td>0.128</td>
<td>0.628 (±0.016)</td>
<td>1.138 (±0.041)</td>
</tr>
<tr>
<td>LGPET(60:40)</td>
<td>0.3</td>
<td>0.2</td>
<td>0.105</td>
<td>0.605 (±0.003)</td>
<td>1.069 (±0.007)</td>
</tr>
<tr>
<td>LGPET(40:60)</td>
<td>0.2</td>
<td>0.3</td>
<td>0.122</td>
<td>0.622 (±0.004)</td>
<td>1.123 (±0.009)</td>
</tr>
<tr>
<td>LGPET(20:80)</td>
<td>0.1</td>
<td>0.4</td>
<td>0.127</td>
<td>0.627 (±0.002)</td>
<td>1.036 (±0.037)</td>
</tr>
</tbody>
</table>

*Calculated by subtracting fiber mass from total mass of filter media.

The wetting kinetics for layered glass and polypropylene media with water and Viscor oil as test liquids are shown in Figure 4.14 and Figure 4.15 respectively and the wetting kinetics for layered glass and polyester media are shown in Figure 4.18 and Figure 4.19 respectively. The initial wetting kinetics for layered glass and polypropylene media with water and Viscor oil as test liquids are shown in Figure 4.16 and Figure 4.17 respectively and the initial wetting kinetics of layered glass and polyester media with water and Viscor oil as test liquids are shown in Figure 4.20 and Figure 4.21 respectively.
The water uptake rate for glass fiber media is the highest and it decreases with increasing amounts of polymeric fibers in filter media. The uptake rate of Viscor oil increases with the amount of polymeric fibers and it is the lowest for only glass fiber media. Some plots do not show steady values of \( w^2 \), it means that the filter did not reached steady state and the filter media was not saturated with the test liquid.

Figure 4.14. Wetting kinetics of layered glass/polypropylene media with water as test liquid.
Figure 4.15. Wetting kinetics of layered glass/polypropylene media with Viscor oil as test liquid.

Figure 4.16. Initial wetting kinetics of layered glass/polypropylene media with water as test liquid.
Figure 4.17. Initial wetting kinetics of layered glass/polypropylene media with Viscor oil as test liquid.

Figure 4.18. Wetting kinetics of layered glass/polyester media with water as test liquid.
Figure 4.19. Wetting kinetics of layered glass/polyester media with Viscor oil as test liquid.

Figure 4.20. Initial wetting kinetics of layered glass/polyester media with water as test liquid.
The initial slopes for water and Viscor oil and L/H values of each layered filter media are shown in Table 4.5. The table also includes the regression coefficients obtained from linear least square fit of initial wetting kinetics of each layered filter media. The variation in L/H values with variation fraction of polymer fibers are shown in Figure 4.22. The filter media with a range of wetting properties can be prepared by using different compositions of hydrophilic and hydrophobic fibers arranged in layered structures. The porosity and permeability as functions of L/H of the layered media are shown in Figures 4.23 and Figure 4.24. Not unexpected, the data show L/H does not affect the porosity and air permeability, since these quantities are primarily functions of geometry and not surface wettability. Figure 4.23 shows the Glass-PP and the Glass-PET
media have approximately the same porosities. However, Figure 4.24 shows the Glass-PET media have a permeability that is about 2 to 3 times that of Glass-PP media. The cause for this requires further investigation. One possible cause, to be investigated in future work, is whether the binders adhere differently to the PP and PET fibers resulting in larger pores for the Glass-PET media.

Table 4.5. Initial slopes and L/H values of different layered media

<table>
<thead>
<tr>
<th>Filter media</th>
<th>Water</th>
<th>Viscor Oil</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$S_w$</td>
<td>$R^2$</td>
<td>$S_o$</td>
</tr>
<tr>
<td>Glass</td>
<td>7.594 (±0.766)</td>
<td>0.980</td>
<td>0.895 (±0.021)</td>
</tr>
<tr>
<td>LGPP(80:20)</td>
<td>2.575 (±0.211)</td>
<td>0.980</td>
<td>1.048 (±0.072)</td>
</tr>
<tr>
<td>LGPP(60:40)</td>
<td>1.239 (±0.049)</td>
<td>0.994</td>
<td>1.224 (±0.043)</td>
</tr>
<tr>
<td>LGPP(40:60)</td>
<td>0.483 (±0.02)</td>
<td>0.988</td>
<td>1.776 (±0.075)</td>
</tr>
<tr>
<td>LGPP(20:80)</td>
<td>0.221 (±0.014)</td>
<td>0.966</td>
<td>1.217 (±0.084)</td>
</tr>
<tr>
<td>LGPET(80:20)</td>
<td>0.496 (±0.028)</td>
<td>0.987</td>
<td>0.286 (±0.011)</td>
</tr>
<tr>
<td>LGPET(60:40)</td>
<td>0.284 (±0.024)</td>
<td>0.972</td>
<td>0.312 (±0.014)</td>
</tr>
<tr>
<td>LGPET(40:60)</td>
<td>0.106 (±0.003)</td>
<td>0.995</td>
<td>0.359 (±0.006)</td>
</tr>
<tr>
<td>LGPET(20:80)</td>
<td>0.068 (±0.005)</td>
<td>0.973</td>
<td>0.418 (±0.003)</td>
</tr>
</tbody>
</table>
Figure 4.22. Variation in L/H with variation in composition of polymer fibers in media.

Figure 4.23. Porosity Vs L/H of different layered filter media.
4.5.3. Radially layered hydrophilic/hydrophobic fiber media

The radially layered filter media were prepared from composite sheets of glass-polypropylene and glass-polyester fibers in different compositions. The average mass of the filter media is 0.6 (±0.02) and it is relatively similar for all the media. The notation RL stands for radially layered media, G stands for glass, PP stands for polypropylene and PET stands for polyester. The wetting kinetics for radially layered glass-polypropylene media with water and Viscor oil as test liquid are shown in Figure 4.25 and Figure 4.26 respectively and the wetting kinetics for radially layered glass-polyester media are shown in Figure 4.29 and Figure 4.30 respectively. The initial wetting kinetics for radially layered glass-polypropylene media with water and Viscor oil as test liquids are shown in Figure 4.27 and Figure 4.28 respectively and the initial wetting kinetics of layered glass-polyester media with water and Viscor oil as test liquids are shown in Figure 4.31 and Figure 4.32. As compare previously described mixed and layered structures, the rate of
liquid uptake is higher for radially layered media. It could be because of the pores formed in the media are separate hydrophilic and hydrophobic capillaries. The flow of water in hydrophilic capillaries and flow of oil in hydrophobic capillaries dominates the wetting dynamics.

Figure 4.25. Wetting kinetics of radially layered glass/polypropylene media with water as test liquid.

Figure 4.26. Wetting kinetics of radially layered glass/polypropylene media with Viscor oil as test liquid.
Figure 4.27. Initial wetting kinetics of radially layered glass/polypropylene media with water as test liquid.

Figure 4.28. Initial wetting kinetics of radially layered glass/polypropylene media with Viscor oil as test liquid.
Figure 4.29. Wetting kinetics of radially layered glass/polyester media with water as test liquid.

Figure 4.30. Wetting kinetics of radially layered glass/polyester media with Viscor oil as test liquid.
Figure 4.31. Initial wetting kinetics of radially layered glass/polyester media with water as test liquid.

Figure 4.32. Initial wetting kinetics of radially layered glass/polyester media with Viscor oil as test liquid.
The initial slopes for water and Viscor oil and L/H values of each radially layered filter media are shown in Table 4.6. The regression coefficients from linear least square fit of initial wetting kinetic data of each radially layered filter media are more than 0.95 and shown in Table 4.6. The variation in L/H values with variation in fraction of polymer fibers are shown in Figure 4.33.

Table 4.6. Initial slopes and L/H values of different radially layered media.

<table>
<thead>
<tr>
<th>Filter media</th>
<th>Water $S_w$ (±)</th>
<th>$R^2$</th>
<th>Viscor Oil $S_o$ (±)</th>
<th>$R^2$</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>12.675</td>
<td>0.982</td>
<td>3.345 (±0.289)</td>
<td>0.971</td>
<td>1.95</td>
</tr>
<tr>
<td>RLGPP(80:20)</td>
<td>11.792 (±1.03)</td>
<td>0.985</td>
<td>4.133 (±0.226)</td>
<td>0.988</td>
<td>2.59</td>
</tr>
<tr>
<td>RLGPP(60:40)</td>
<td>7.300 (±0.1078)</td>
<td>0.958</td>
<td>3.968 (±0.346)</td>
<td>0.971</td>
<td>4.01</td>
</tr>
<tr>
<td>RLGPP(40:60)</td>
<td>2.816 (±0.14)</td>
<td>0.979</td>
<td>6.329 (±0.407)</td>
<td>0.984</td>
<td>16.58</td>
</tr>
<tr>
<td>RLGPET(80:20)</td>
<td>11.900 (±1.473)</td>
<td>0.956</td>
<td>3.914 (±0.169)</td>
<td>0.995</td>
<td>2.43</td>
</tr>
<tr>
<td>RLGPET(60:40)</td>
<td>10.852 (±0.868)</td>
<td>0.981</td>
<td>4.025 (±0.358)</td>
<td>0.977</td>
<td>2.74</td>
</tr>
<tr>
<td>RLGPET(40:60)</td>
<td>3.850 (±0.539)</td>
<td>0.944</td>
<td>4.020 (±0.474)</td>
<td>0.960</td>
<td>7.70</td>
</tr>
</tbody>
</table>
The porosity and permeability as functions of L/H of the radially layered media are shown in Figures 4.34 and Figure 4.35. The data in Figure 4.34 shows that the porosity has no effect on L/H of all the radially layered media. However, as compared to mixed and layered structures of glass and polymer fibers, porosity of radially layered media is slightly higher. The Figure 4.35 shows that the permeability of radially layered filter media with polypropylene and polyester fibers increases with increasing L/H. In preparation on composite layered sheets, the fibers lay in horizontal planes perpendicular to the flow direction. However, in the radially layered filter media the fibers are oriented in planes parallel to the fluid flow which causes less resistance to flow as compared to horizontal fibers.
4.5.4. Hydrophobic electrospun fibers mixed filter media

The polypropylene fibers were prepared using electrospinning process as described in section 3.3. The polypropylene electrospun fibers were prepared in different
fiber sizes (i.e. average diameters of 300nm, 600nm, 900nm) with variation in polypropylene solution concentration. These polypropylene electrospun fibers were added to glass fiber media in different area ratios (0.5, 1 and 2). The purpose of this study is to determine the effect of hydrophobic electrospun fibers on wettability and filter performance. The effect of fiber size and amount of hydrophobic electrospun fibers on wettability is also studied.

The total mass, mass of electrospun fibers added to the glass fibers and nomenclature of different PP electrospun fiber added filter media are shown in Table 4.7. In the notation, AR = 0.5 (300nm) means PP electrospun fibers of 300nm were added to glass fiber media with area ratio 0.5. The similar notation are used for the filter media with variation in fiber size and area ratio.
The wetting kinetics of PP electrospun fiber mixed media with water and Viscor oil as test liquids are shown in Figure 4.36 and Figure 4.37 respectively. The initial wetting kinetics of PP electrospun fiber mixed media with water and Viscor oil as test liquids are shown in Figure 4.38 and Figure 4.39. The wetting kinetic data show that liquid-uptake rates for glass fiber media are highest for water and Viscor oil. The results of layered glass fiber media are used for comparison with the electrospun fiber added media. The media with small amounts of electrospun fibers i.e. filter media with area
ratio 0.5; have higher water uptake rates as compare to filter media with electrospun fibers of higher area ratio. There is not a significant variation in Viscor oil uptake except for glass and media with 600nm PP electrospun fibers added in area ratio of 2.

Figure 4.36. Wetting kinetics of PP electrospun fiber added glass fiber media with water as test liquid.
Figure 4.37. Wetting kinetics of PP electrospun fiber added glass fiber media with Viscor oil as test liquid.

Figure 4.38. Initial wetting kinetics of PP electrospun fiber added glass fiber media with water as test liquid.
Figure 4.39. Initial wetting kinetics of PP electrospun fiber added glass fiber media with Viscor oil as test liquid.

The initial slopes and L/H values of all the PP electrospun fiber added media are shown in Table 4.8 along with regression coefficients from least square fit of initial kinetic data of each filter medium. The effect of area ratio and fiber size added to the glass fiber media on L/H is shown in Figure 4.40 and the effect of the amount of PP electrospun fibers added is shown in Figure 4.41. It is observed that L/H value of glass fiber media increases as the amount of PP electrospun fibers in the media increases. The effect of electrospun fiber size on L/H is clearly observed on filter media with 900nm fibers and different area ratios. The variation in L/H for PP electrospun fiber added media is from 0.89 to 2.32, which is a very small variation as compared to other geometries with glass and polymer microfibers.
The effect of porosity and permeability is shown in Figure 4.42 and Figure 4.43. The data show that porosity of the filter media slightly decreased as amount of electrospun fibers increased. The electrospun fibers can decrease the size of pores formed by microfibers without changing the overall geometry of the filter media. Thus, within a similar sample volume, more solid fibers are present which reduces the porosity. The permeability of media with PP electrospun fiber added to glass fibers increased as compare to only glass fiber media. The cause behind this needs further investigation. There is no significant effect of amount of PP electrospun fibers is observed on permeability.

Table 4.8. Initial slopes and L/H values for PP electrospun fiber mixed media.

<table>
<thead>
<tr>
<th>Filter media</th>
<th>Water</th>
<th>Viscor Oil</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$S_w$</td>
<td>$R^2$</td>
<td>$S_o$</td>
</tr>
<tr>
<td>Glass</td>
<td>7.023 (±0.698)</td>
<td>0.952</td>
<td>0.854 (±0.045)</td>
</tr>
<tr>
<td>AR=0.5(300nm)</td>
<td>3.385 (±0.282)</td>
<td>0.995</td>
<td>0.481 (±0.011)</td>
</tr>
<tr>
<td>AR=0.5(600nm)</td>
<td>4.120 (±0.606)</td>
<td>0.981</td>
<td>0.553 (±0.059)</td>
</tr>
<tr>
<td>AR=0.5(900nm)</td>
<td>3.607 (±0.631)</td>
<td>0.978</td>
<td>0.500 (±0.326)</td>
</tr>
<tr>
<td>AR=1(300nm)</td>
<td>3.026 (±0.298)</td>
<td>0.986</td>
<td>0.383 (±0.017)</td>
</tr>
<tr>
<td>AR=1(600nm)</td>
<td>3.278 (±0.575)</td>
<td>0.980</td>
<td>0.468 (±0.040)</td>
</tr>
<tr>
<td>AR=1(900nm)</td>
<td>2.189 (±0.187)</td>
<td>0.986</td>
<td>0.547 (±0.031)</td>
</tr>
<tr>
<td>AR=2(300nm)</td>
<td>2.850 (±0.105)</td>
<td>0.965</td>
<td>0.479 (±0.011)</td>
</tr>
<tr>
<td>AR=2(600nm)</td>
<td>2.550 (±0.073)</td>
<td>0.955</td>
<td>0.651 (±0.062)</td>
</tr>
<tr>
<td>AR=2(900nm)</td>
<td>1.634 (±0.054)</td>
<td>0.978</td>
<td>0.515 (±0.053)</td>
</tr>
</tbody>
</table>
Figure 4.40. Effect of area ratio and fiber size of PP electrospun fibers added on L/H.

Figure 4.41. Variation in L/H with variation in amount of electrospun fibers added to glass media.
Figure 4.42. Effect of amount of electrospun fibers added to glass fiber media on porosity.

Figure 4.43. Effect of amount of PP nanofibers added to glass fiber media on air permeability.
4.5.5. Mixed media with silane coated fibers

The wettability of filter media is influenced by filter geometry, fiber size, porosity and composition of filter media. The filter media prepared with same fiber size with variation in composition can give better understanding of effect of composition of hydrophilic and hydrophobic fibers on wettability of filter media. The FTS silane coated glass surface had a contact angle of water above 90° and was selected to coat the glass fibers. The coating procedure is explained in section 3.4. The filter media were prepared by mixing coated and uncoated fibers in different compositions. The glass fiber sheets coated with FTS silane show variation in wettability depending on the concentration of the silane solution and the coating time. Table 4.9 shows the behavior of water drops and Viscor oil on a FTS coated glass fiber sheet with different concentrations of FTS silane solutions and coating time.
Table 4.9. Behavior of water and Viscor oil drops on FTS coated glass sheet with different concentration and coating time.

<table>
<thead>
<tr>
<th>Description</th>
<th>Behavior of water (clear or red with food coloring) and Viscor oil drop (yellow) on FTS coated glass fiber sheet</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>Concentration of FTS = 200 µl Coating time = 30 min. Water drops bead up on one side of the sheet and spread on other side.</td>
</tr>
<tr>
<td>(b)</td>
<td>Concentration of FTS = 200 µl Coating time = 1 hr. Water drops bead up on both sides of glass fiber sheet.</td>
</tr>
<tr>
<td>(c)</td>
<td>Concentration of FTS = 200 µl Coating time = 1 hr. Viscor oil drops bead up on one side and spread on other side of glass fiber sheet.</td>
</tr>
</tbody>
</table>
The glass sheet coated with 200 µl concentration FTS silane solution and 1 hr coating time was selected for the preparation of filter media. The coated sheet shows oleophilic behavior on one side and oleophobic on other side (Figure (c) in Table 4.9) and the sheet is hydrophobic on both sides (Figure (b) in Table 4.9). This shows that the silane coating is not the same on both sides. The filter media with different compositions of coated and uncoated fibers were prepared. The filter media were characterized for wettability using Washburn’s method. The media with lower compositions (i.e., uncoated: coated (80:20) and (60:40)) showed some liquid uptake but media with higher composition (uncoated: coated (40:60) and (20:80) ) and also only FTS coated fiber media showed no liquid uptake for both water as well as Viscor oil. In the capillary rise experiments, hydrophobic fibers can restrict the flow of liquid in the upward direction.
To apply Washburn’s method, the media should be wetted by both liquids to a certain extent. In the case here, Washburn’s method is not applicable for the hydrophobic and oleophobic filter media.

4.5.6. Summary

- Filter media were prepared in mixed, layered and radially layered geometries with varying the composition of hydrophilic and hydrophobic fibers. The compositions of fibers were varied from only glass fiber media and amount of glass fibers 80%, 60%, 40%, 20%. The filter media showed variation in L/H with variation in filter geometry.

- For mixed filter media L/H varies from 0.47 to 58. For layered filter media it varies from 3 to 40 for Glass-PP media and 4 to 45 for Glass-PET media. The radially layered media were prepared with composite sheets and had variation in L/H from 2 to 16 for Glass-PP media and 2 to 7 for Glass-PET media.

- The porosity and permeability of filter media with different composition and geometries did not vary significantly with L/H. This shows that surface wettability of fibers only effected L/H and hence L/H can be used to characterize the wettability of the media.

- Filter media prepared by mixing hydrophobic polypropylene submicron electrospun fibers do not show as much variation in L/H as compare to polypropylene microfiber mixed media. It was observed that, as amounts of electrospun polypropylene fibers increases, L/H increases. The effect of fiber size
also showed influence on L/H when electrospun fibers were added to glass fiber media with higher area ratio.

- The filter media were prepared by mixing FTS silane coated glass fibers with uncoated glass fibers. The purpose of this study was to understand the effect of composition of mixed fibers on wettability. It was observed that with variation in concentration of silane solution and coating time one can vary the wettability of fibers. The filter media prepared with coated and uncoated fibers are hydrophobic and also oleophobic. Therefore, Washburn’s method was not used to characterize this type media.
CHAPTER V

LIQUID-LIQUID COLAESCEENCE

This chapter describes the design of liquid-liquid coalescence experimental set-up. It also describes the sampling method and surface and interfacial tension characterization of Viscor oil.

5.1. Experimental set-up

The coalescence experiments were performed using a fine water-in-oil emulsion, in which deionized water was dispersed in Viscor 1487. The schematic of the experimental set-up is shown in Figure 5.1.

The Viscor oil is pumped using a peristaltic pump (Master flex, model L/S EW-07543-60) at flow rate of 210 ml/min. At the mixing pipe water droplets were introduced into the oil stream using a syringe pump (WPI, Model sp101i) at a flow rate of 80 µl/min. The mixing pipe is a Plexiglas tube with a 1mm inside diameter, with a hypodermic needle inserted at the middle of the 1mm cylindrical channel through which the oil flows. The water droplets form at the tip of the needle and are stripped off by the oil flow to generate the fine emulsion. The concentration of water and size of water drops can be
controlled by the flow rate of water from the syringe pump. The flow rates of oil and water were the same for all of the layered media experiments.

Figure 5.1. Liquid-liquid coalescence set-up schematic.

The filter is placed in a Plexiglas filter media holder designed with a cylindrical opening of 2.25 cm diameter. The filter media here is actually a fibrous coalescer, however, the terms coalescing filter media, coalescing filter or filter are used in this text interchangeably. The filter media holder is shown in Figure 5.2. The emulsion flows through a filter sample and into a gravity settling tank and reservoir. The upstream sampling point is positioned between the mixing pipe and the filter. The downstream sampling point is positioned after the gravity settling tank. The pressure drop is measured across the filter on a 10 min interval. The tests were performed for 110 min to 130 min
and steady state was typically achieved between 70 min to 90 min depending upon the filter media. The size distribution of water droplets at the upstream and downstream were measured using an Accusizer (780 PALS- Particle Sizing Systems, sensor range 0.5 μm to 500 μm) at intervals of 10 to 25 minutes. The Accusizer is shown in Figure 5.3. The Accusizer use a single particle optical sensing (SPOS) method which is based on the principle of laser light scattering or obscuration (blockage). Particles in gas or liquid suspension flow through a small "photozone" -- a narrow, slab-like region of uniform illumination, produced by light from a laser diode or incandescent bulb. The particle suspension is sufficiently dilute when the particles pass, one at a time, through the illuminated region, avoiding coincidences. The passage of a particle through the sensing zone causes a detected pulse, the magnitude of which depends on the mean diameter of the particle. The method assumes all particles are of spherical shape and have similar optical properties.
Figure 5.2. Filter holder (a) Assembled view (b) opened view.

Figure 5.3. Accusizer (a) Electronic box (b) Stage with sensor (c) Dilution chamber.
5.2. Experimental procedure

The Figure 5.4 shows the lab-scale experimental set-up for liquid-liquid coalescence.

1. Check the oil level in tank.
2. Check water in syringe pump and connection to mixing chamber.
3. Check and set the type of syringe, volume and flow rate.
4. Turn of the Accusizer and Jorin ViPA instrument with computers.
5. Clean the filter holder, place filter sample, assemble the filter holder.
6. Check pressure transducer connections.
7. Check downstream is connected to settling tank and downstream valves are open.
8. Check if all pipe connections and unions are properly tightened.
9. Keep the upstream sampling valve open and upstream valve before filter holder closed.
10. Start peristaltic pump and syringe pump.
11. Allow previous oil and emulsion to drain out through sampling valve.
12. Open the valve before filter holder and close the sampling valve.
13. Experiment is now started.
14. Record the pressure drop.
15. Take samples at the upstream and downstream at required interval of time.
16. Open the valve after settling tank for measurements using Jorin ViPA. Adjust the flow rate.
17. Turn of the peristaltic pump, syringe pump and pressure transducers.
18. Dissemble the filter holder, remove the filter medium and clean the filter holder.
19. Close all reservoirs of oil to avoid any dust into the oil.

20. Clean the area around the apparatus.

Figure 5.4. Liquid-liquid coalescence experimental set-up.

5.3. Surface and interfacial tension measurements

The Viscor oil is continuously characterized for its surface and interfacial tensions. The surface tension of Viscor oil was 26 dynes/cm to 28.5 dynes/cm and interfacial tension was 16 dynes/cm to 18 dynes/cm. The surface tension of the liquid was
measured using Tensiometer (Dunoy Ring method). The interfacial tension was measured using Pendant drop method with Drop shape analyzer (Kruss Instruments, Germany). The Figure 5.5 shows the Drop Shape Analyzer (DSA). The contact angle measurements also done using DSA.

Figure 5.5. Drop Shape Analyzer (DSA).
CHAPTER VI

EXPERIMENTAL RESULTS

The objective of this work is to study the effect of wettability on filter performance for liquid-liquid coalescence. The filter media with different wetting properties were developed using hydrophilic and hydrophobic fibers in different geometries and varying compositions. The developed filter media were characterized for their wettability using modified Washburn’s equation. The developed filter media were evaluated for their performance using liquid-liquid coalescence experiments. This chapter presents the experimental results of the filter media tested for liquid-liquid coalescence. The effect of wettability on separation efficiency, pressure drop and overall performance of filter media was studied.

6.1. Performance evaluation of filter media

All the developed filter media were tested in liquid-liquid coalescence set-up as described in chapter V. The upstream and downstream particle size distributions were measured with the Accusizer on samples taken from the flow streams. The combined performance is evaluated using separation efficiency ($E$) and pressure drop ($\Delta P$) across the filter medium (6). The separation efficiency is calculated as...
\[ E = \frac{C_{IN} - C_{OUT}}{C_{IN}} \]  \hspace{1cm} (6.1)

where, \( C_{IN} \) and \( C_{OUT} \) are the inlet and outlet mass concentrations of water drops respectively. The inlet and outlet mass concentrations are calculated from the upstream and downstream particle size distribution by the formula

\[ C_i = \sum N_i \frac{\pi}{6} d_i^3 \rho_{\text{water}} \]  \hspace{1cm} (6.2)

where, \( N_i \) is number of drops, \( d_i \) is diameter of droplet and \( \rho_{\text{water}} \) is the density of water.

The overall performance of filter media is measured as a figure of merit, also known as quality factor, as defined by Brown [40], which accounts for both the separation efficiency and pressure drop. The quality factor (QF) is calculated as

\[ QF = \frac{-\ln(1 - E)}{\Delta P} \]  \hspace{1cm} (6.3)

Typical steady state droplet size distributions for upstream and downstream for the glass filter media are shown in Figure 6.1.
Figure 6.1. Representative droplet size distribution of upstream, downstream of glass filter media and with no filter media.

The upstream size distribution was fairly consistent for all the experiments. The distribution curve labeled as ‘No filter’ represents the distribution obtained at the downstream without any filter placed in the holder but with the supporting wire mesh in its position. The same wire mesh was used to support the filter samples during all of the experiments. The variation in ‘Upstream’ and ‘No filter’ distribution curves shows that some of the droplets are separated by the settling tank. Thus, ‘No filter’ experiment gives the efficiency of settling tank as 32% (±3%) obtained over multiple samplings. The coalesced droplets settle at in the settling tank and suspended droplets are further carried in the downstream. The downstream sampling point is after the first settling tank. The separation efficiency of filter and settling tank is coupled. Therefore, the separation
efficiency obtained with particular filter media is the separation efficiency of the liquid-liquid coalescence system. The filters were tested in triplicate and the results are plotted as average of three filters with one standard deviation indicated by the error bars.

6.2. Mixed hydrophilic/hydrophobic fiber media

Mixed fiber media with glass-polypropylene fibers developed in different compositions were tested in liquid-liquid coalescence set-up. The steady state pressure drop as a function of L/H is plotted in Figure 6.2. The pressure drop of MGPP(80:20) filter media is increased as compare to only glass fiber media and pressure drop of other mixed media is decreased with increase in L/H. In general, pressure drop is reduced as composition of polypropylene fibers increased in mixed filter media.

![Figure 6.2. Effect of L/H on pressure drop of mixed glass and polypropylene filter media.](image-url)
The effect of L/H on separation efficiency with mixed glass and polypropylene filter media is shown in Figure 6.3. The separation efficiency of MGPP(80:20) media is 94% and MGPP(60:40) media is 91% shows improvement over only glass fiber media which has efficiency of 86%. The separation efficiency of other filter media decreased as amount of polypropylene fibers in filter media increased. In general, separation efficiency increased initially above the efficiency of only glass fiber media and decreased as L/H increased. The best separation efficiency is shown by mixed filter media of L/H = 2.3 but it also has higher pressure drop as compare to only glass fiber media. The data show that the best separation efficiency with mixed glass-polypropylene media can be achieved at L/H between 2 to 4.

![Figure 6.3. Effect of L/H on separation efficiency of mixed glass and polypropylene filter media.](image)
The combined measure of efficiency and pressure drop is given by quality factor. The effect of L/H on quality factor is shown in Figure 6.4. The quality factors of all the mixed filter media improved as compare to only glass fiber media. The highest quality factor is shown by MGPP(60:40) media. The higher quality factor of MGPP(20:80) media is primarily due to significant reduction in pressure drop.

![Figure 6.4. Effect of L/H on quality factor of mixed glass and polypropylene filter media.](image)

6.3. Layered hydrophilic/hydrophobic fiber media

The steady state pressure drop of all the layered glass-polypropylene and glass-polyester filter media is plotted as a function of L/H as shown in Figure 6.5. The pressure drop of all the LGPP media and LGPET media decreased with increase in L/H with exception of LGPET(20:80) media with L/H =45. The reason for this increase in pressure drop need to be further investigated. The LGPET (80:20) media shows higher pressure...
drop as compare to only glass fiber media. The data also show that pressure drops of layered glass-polypropylene media are lower as compare to glass-polyester layered media with similar compositions.

Figure 6.5. Effect of L/H on pressure drop of mixed glass and polypropylene filter media.

The effect of L/H on separation efficiency of layered glass-polypropylene and glass-polyester filter media is shown in Figure 6.6. The separation efficiency initially increased, followed by gradual decrease with increase in L/H. The separation efficiency with LGPET (80:20) i.e., 93% and LGPP (80:20) i.e., 91% shows improvement over glass only fiber media. The separation efficiency of all layered glass-polyester media is higher as compare to layered glass-polypropylene media with similar compositions. In general, layered filter media having L/H between 3 to 10 gives better separation efficiency.

The effect of L/H on quality factor of layered filter media is shown in Figure 6.7. The quality factor of all the layered filter media are improved as compare to glass only filter media. The exception to this is LGPET(20:80) media with L/H=45. The reason for
this low quality factor is the high pressure drop as described above. The highest quality factor is obtained with LGPP(80:20) and LGPP (60:40) media. The increase in quality factor is primarily due to reduction in pressure drop. It is also observed that quality factors of layered filter media are higher than mixed filter media.

Figure 6.6. Effect of L/H on efficiency of different layered filter media

Figure 6.7. Effect of L/H on quality factor of different layered filter media
6.4. Radially layered hydrophilic/hydrophobic fiber media

The steady state pressure drop of radially layered glass-polypropylene and glass-polyester media are shown in Figure 6.8. The pressure drop of radially layered filter media reduced as L/H increased. The pressure drop of glass fiber media reduced as compare to layered and mixed filter geometry. By comparison with Figure 6.2 and Figure 6.5, it is also observed that radially layered filter media with different compositions show reduced pressure drop as compare to other filter geometries with similar compositions. In general, the radially layered geometry gives reduced pressure drop.

![Figure 6.8. Effect of L/H on pressure drop of different radially layered filter media.](image)

The effect of L/H on separation efficiency of radially layered glass-polypropylene and glass-polyester filter media is shown in Figure 6.9. There is no significant increase or decrease in efficiency with radially layered media except the lowered efficiency with
RLGPP (60:40) media which has L/H=17 and 73% efficiency. The L/H of radially layered media developed are in the range of 2 to 8 and most of the media fall in the range of 2 to 3. Not unexpected, media with similar wettability shows similar separation efficiencies. However, radially layered media has showed significant reduction in pressure drop compare to layered and mixed media geometries.

The quality factor of radially layered media as a function of L/H is shown in Figure 6.11. By comparison with Figure 4.4 and Figure 4.7, in general, the quality factor of radially layered media is improved as compare to layered and mixed filter geometries.

![Figure 6.10. Effect of L/H on efficiency of radially layered filter media.](image-url)
Figure 6.11. Effect of L/H on quality factor of radially layered filter media

6.5. Hydrophobic electrospun fiber mixed glass fiber media

The pressure plots in Figure 6.12 show a significant increase in pressure drop for media with electrospun polypropylene fiber mixed filter media compared with glass fiber only media. The electrospun polypropylene fibers are smaller in diameter and also have some beads. Both small fibers and beads reduce the pore size of glass fiber media. This can also be observed from slight reduction in porosity with increase in amount of electrospun fibers in media. The smaller fibers can capture smaller droplets and were expected to increase efficiency. The hydrophobic nature of smaller fibers tend to hold water in the medium and further reduces the pore size of the filter media and increase the pressure drop. However, the significant increase in pressure drop because of addition of electrospun polypropylene fibers should be explored further in future work.
The effect of size of electrospun PP fibers and area ratio on efficiency is shown in Figure 6.13. The separation efficiency of all the filter media with electrospun fibers is increased significantly. The higher separation is observed with area ratio of 1 as compared to area ratio of 0.5 and area ratio of 2. The highest separation efficiency of 98% is obtained with the media having 300nm electrospun fibers and area ratio of 1. Here, wettability of electrospun fibers, fiber size and amount of fibers added to the glass fiber media has influence in increase in separation efficiency as well as pressure drop. The increase in pressure drop reduced the quality factor significantly compared to only glass fiber media, which is shown in Figure 6.14.
Figure 6.13. Effect of size and area ratio of electrospun fibers on separation efficiency.

Figure 6.14. Effect of electrospun fiber size and area ratio (AR) of electrospun fibers on quality factor.
6.6. Mixed media with silane coated fibers

The mixed filter media with silane coated fibers with uncoated glass fibers were prepared in different compositions. It is expected that wettability of the filter varies with the composition of FTS coated fibers in a mixed media. However, as explained in chapter IV, section 4.6, the media show certain hydrophobicity and oleophobicity where modified Washburn’s equation is not applicable. The mixed media with coated fibers were evaluated for their performance in liquid-liquid coalescence and compared with only glass fiber media.

The steady state pressure drop of mixed FTS coated fiber media is plotted as a function of composition of coated fibers as shown in Figure 6.16.

![Graph showing pressure drop of mixed silane coated fiber media.](image)

Figure 6.16. Pressure drop of mixed silane coated fiber media.
The pressure drop is decreased with increased composition of FTS coated fibers in the filter media. All the filter media with FTS coated fibers show reduced pressure drop as compare to only glass fiber media.

The steady state separation efficiency of mixed silane coated media is plotted against composition of coated fibers and shown in Figure 6.17. The separation efficiency is increased initially with increased composition of coated fibers above glass fiber media and decreased when composition of coated fibers is above 80%. The highest separation efficiency obtained with GFTS (60:40) media i.e., 91%.

Figure 6.17 Separation efficiency of mixed silane coated fiber media.

The quality factors of mixed silane coated media are shown in Figure 6.18. The quality factors of all the mixed silane coated filter media are improved as compare to
glass only fiber media. The improvement in quality factor is mainly due to the decrease in the pressure drop. The highest quality factor was achieved with GFTS (60:40). This media showed improved separation efficiency i.e., 91% and also reduced pressure drop as compare to glass only filter media.

Figure 6.18. Quality factor of mixed silane coated filter media.

6.7. Effect of interfacial tension

The liquid fuels contain different types of surfactants as performance and quality enhancers. In the filtration industry, use of a model surfactant for filter testing is manufacturer specific and information is generally proprietary. The SAE J1488 standard for Emulsified water/fuel test procedure, recommends use of Ethyl D113 (Ethyl
Corporation) as a model surfactant. This surfactant is used to simulate additives normally used in diesel fuel such as cetane enhancer. The Ethyl D11 is a commercially available additive and has limited shelf life. According to revised SAE J1488 standard, Glycerol Mono-Oleate is a recommended surfactant for test procedures of fuel/water separation. This surfactant is used to simulate Biodiesels in diesel fuels and can provide test fuels interfacial in the range of 15-19 mN/m.

6.1.1. Variation in interfacial tension of Viscor using model surfactant

The Glycerol Oleate (GO) additive, which is compatible and used with Viscor 1487, were obtained from Rock Valley Chemical Company, IL. The Glycerol Oleate is a thick liquid and has a dark smell of vegetable oils. The surfactant was used to study the variation in interfacial tension of Viscor oil. The GO was added in Viscor oil in different percentages and interfacial tension was measured using pendent drop method (Drop shape Analyzer, Kruss Instruments, GMBH). The Figure 6.19 shows the effect of GO surfactant on Interfacial tension. The samples were prepared in 100ml of fresh Viscor oil with desired amount of GO. The samples were agitated using hand held sonicator for 15 sec. The surfactant was completely dissolved in Viscor oil without any variation in color. The samples were tested for interfacial tension within 5 min. time of agitation. The each sample were characterized for the interfacial tension with deionized water (Surface tension= 72 ±1.27 mN/m) for six times. The average value of six measurements are reported with error bar indicating one standard deviation. The interfacial tension of
Viscor oil is decreased from 19.5 mN/m to 6 mN/m with addition of about 5% of Glycerol Oleate.

![Figure 6.19. Variation of interfacial tension with concentration of Glycerol Oleate in Viscor 1487.](image)

6.1.2. Experiments with reduced interfacial tension Viscor oil

The experiments were prepared with 4 L of Viscor oil batch with 0.4% of Glycerol Oleate for the experiment. The Viscor oil was stirred using a stirrer for 2 min. and whole batch was circulated two times using the peristaltic pump. The interfacial tension of Viscor oil after addition of surfactant was about 11 mN/m. The Viscor oil in this experiments was used and recycled multiple times and this causes variation in interfacial tension. The interfacial tension values reported in Figure 6.19 are for the never used Viscor oil. Three experiments were repeated with same batch. The interfacial tension of Viscor oil increased up to 13 mN/m after the experiments. The representative
upstream droplet size distribution after addition of GO is shown in Figure 6.20 and Figure 6.21. The downstream distribution with glass fiber media is shown in Figure 6.22 and Figure 6.23. The figures are the actual results given by Accusizer. The upstream distributions were not steady but varied with time. The oil flow rate, water flow rate, and other experimental conditions were similar for experiments with and without surfactant. The interfacial tensions of the Viscor oil were also observed to change after the Viscor oil passed through filter and thus makes it difficult to re-use the Viscor oil in the subsequent experiments. Because of this variability the experiments were not performed till steady state. The experiments were for about 20 min. The experimental results with glass and MGPP(80:20) media were shown in Table 6.1.

Figure 6.20. Upstream Size distribution after addition of Glycerol Oleate with glass fiber media.
Figure 6.21. Upstream Size distribution after addition of Glycerol Oleate with glass fiber media.

Figure 6.22. Downstream Size distribution after addition of Glycerol Oleate with glass fiber media.
Figure 6.23. Downstream Size distribution after addition of Glycerol Oleate with glass fiber media.

Table 6.1. Efficiency, pressure drop and interfacial tension of experiments with Glycerol Oleate (0.4%)

<table>
<thead>
<tr>
<th>Filter media</th>
<th>Efficiency (%)</th>
<th>Pressure drop (KPa)</th>
<th>IFT (mN/m) (± 0.3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass - 1</td>
<td>73.48</td>
<td>13.16</td>
<td>11.03</td>
</tr>
<tr>
<td>Glass - 2</td>
<td>73.91</td>
<td>12.69</td>
<td>13.26</td>
</tr>
<tr>
<td>Glass - 3</td>
<td>78.12</td>
<td>13.24</td>
<td>13.12</td>
</tr>
<tr>
<td>MGPP (80:20)</td>
<td>80.03</td>
<td>11.99</td>
<td>11.5</td>
</tr>
<tr>
<td>MGPP(80:20)</td>
<td>78.82</td>
<td>13.10</td>
<td>13.01</td>
</tr>
</tbody>
</table>

It was observed that, number of smaller drops at the downstream were increased for experiments with surfactant. The high shear fields in the filter media and presence of excess surfactant could have effect on generation of smaller drops at the
downstream. In coalescence when water drops coalesce to form bigger drops, the surface area of drops reduced and concentration of surfactant present at the interface increases. There is a possibility of droplet breakage due to shear fields in the filter media. The excess surfactant can stabilize the smaller drops and restricts coalescence. In addition, surface energy of fibers can vary due to adsorption of surfactant and can restricts the coalescence process.

The study can be extended with separate batches of Viscor oil for each experiment with different interfacial tension. The other liquid fuels such as kerosene or diesel fuel can be used for separate experiment.

6.8. Summary of results

- Mixed filter media – In general, the pressure drop of mixed filter media is reduced with increase in L/H. The efficiency of MGPP(80:20) media is highest among all mixed glass-polypropylene media.
- Layered filter media – In layered media also, pressure drop reduced with increase in L/H. The highest separation efficiency was obtained with LGPP(80:20) and LGPET(80:20) media.
- Radially layered media – The efficiency of radially layered media slightly improved as compare to glass fiber media. The pressure drop decreased significantly as compare to mixed and layered filter media.
- In most filter media the quality factor of mixed, layered and radially layered filter media improved as compare to mixed glass fiber media. The increase in quality factor is primarily the result of reduction in pressure drop.
• For filter media prepared with electrospun polypropylene fiber mixed media, separation efficiency significantly increased as compare to only glass fiber media. However, pressure drop also significantly increased. The media with area ratio 1 and 300nm fibers showed highest separation efficiency of 98% but the quality factor was much less than only glass fiber media.

• The fiber size has significant influence on filter performance. The reduction in pressure drop could be a result of larger fiber size. However, mixed media FTS silane coated fibers, having similar fiber size, had reduced pressure drop due to an increase in composition of hydrophobic fibers in filter media. The fibers used in mixed, layered or radially layered filter media were of similar size. Therefore, it can be deduced that, higher L/H reduces pressure drop. The quality factor of MGFTS (60:40) media was the highest.
CHAPTER VII

CONCLUSIONS

The objectives of the work and achievements are summarized below

- The objective of the work was to design and develop different geometries of filter media using hydrophilic and hydrophobic fibers. Following geometries of filter media were developed.

  - Mixed hydrophilic/hydrophobic fiber media, in which fibers of hydrophilic and hydrophobic fibers are randomly mixed. The vacuum molding process was modified to achieve uniform mixing of fibers with different surface properties.

  - Layered hydrophilic/hydrophobic fiber media in which layers of hydrophilic and hydrophobic fibers were arranged alternatively with flow perpendicular to each layer. This type of media were prepared using the vacuum molding process.

  - Radially layered hydrophilic/hydrophobic filter media in which hydrophilic and hydrophobic layers were arranged alternatively with flow parallel to each layer. The lab-scale sheet former was used to develop the composite layers of the media.
- The objective of the work was to develop filter media with different compositions of hydrophilic and hydrophobic fibers. The filter media were prepared in different compositions of hydrophilic and hydrophobic fibers in each different type of geometry.
  - Media made of different types of microfibers were prepared using commercially available microfibers. These media were constructed as layers of the different fibers or by blending the individual fibers together.
  - The filter media with different size electrospun polypropylene fibers with different fiber area ratios were prepared. The electrospun fibers of size 300nm, 600nm, 900nm were added to glass fiber media in different area ratios of 0.5, 1 and 2.
  - The mixed filter media with FTS silane coated fibers and uncoated fibers were prepared in different compositions. The purpose of this study is to understand the effect of varying composition on wettability and effect of wettability of filter performance with all other parameters constant.
  - All the filter media were evaluated for their filter performance in liquid-liquid coalescence experiments. The effect of wettability on filter performance was studied based on results obtained from wettability characterization and liquid-liquid coalescence experiments.
- The next objective of the work was to apply appropriate characterization techniques for fibrous filter media.
- The modified Washburn’s equation was successfully applied for the wettability characterization. The experimental apparatus was fabricated for the wettability characterization. The wettability of the filter media was expressed as the lipophilic to hydrophilic (L/H) ratio.

- The filter media were characterized for its porosity, permeability and strength. The effect of porosity, permeability, composition of fibers, amount of fibers and geometry of filter media on wettability of filter media were studied, which is fourth objective of this work.

- Another objective of the work was to study the effect of interfacial tension on filter performance. The model surfactant Glycerol Oleate were selected and effect of surfactant on interfacial tension of Viscor oil was studied. The glass and mixed polypropylene media were tested at lowered interfacial tension in liquid-liquid coalescence experiment for 20 min. The study can be further extended to study the effect of interfacial tension of Viscor oil on filter performance by testing separate batches of Viscor oil with different interfacial tension.

- The set-up of theory to account for the pressure drop and saturation as a function of filter bed length and as a function time is shown in Appendix A. The constitutive relation for capillary pressure and of appropriate numerical scheme can be used to solve the developed equations.

In this work three hypothesis were evaluated and following conclusion can be drawn,

The hypothesis of the work are
1. By constructing filter media of mixed hydrophilic and hydrophobic fibers one can control the wettability of the filter medium and hence can control the filter performance for liquid-liquid coalescence.

2. Wettability of the filter media can be varied by constructing filter media in different geometries of layers of wetting and non-wetting fibers.

3. Wettability of the filter media can be controlled by choosing the right composition of hydrophilic and hydrophobic fiber materials in media. The media with required wettability can be designed for particular application.

The quality factor data as a function of L/H of all the filter media with different geometries and varying composition is shown in Figure 7.1. This shows that filter media with L/H range of 2 to 10 has highest quality factors. Thus, by mixing hydrophilic and hydrophobic fibers it is possible to control the wettability and performance of filter media for liquid-liquid coalescence.

The filter media were prepared in different geometries of layers of wetting and non-wetting fibers and L/H data show that the geometry of filter media has influence on variation in wettability. The radially layered media has lower L/H values as compare to other type of filter geometries. The geometry of the filter media also shows significant influence on reducing the pressure drop of filter media and hence showing the higher quality factors.
The wettability characterization results show that as composition of hydrophobic fibers increases the L/H value increases. By varying the composition of hydrophilic and hydrophobic fibers in filter media wettability of the filter can be controlled. The wettability of the filter media is influenced by porosity, permeability, fiber size and geometry. These latter factors are primarily the functions of geometry and not the surface properties and can be controlled. For any two immiscible liquids, the wettability of filter media can be characterized using modified Washburn’s equation. The filter media with right wettability can be designed for a particular application by controlling the composition of fibers of different surface properties.

Figure 7.1. Quality factor Vs L/H for different filter geometries.
Conclusions of the work

1. The filter media with different wetting properties can be designed by choosing a right composition of hydrophilic/hydrophobic fibers in a desired geometry.

2. The modified Washburn’s equation can be used to characterize the wettability of the filter media.

3. The optimum wettability range can be between L/H value of 2 to 10. However, wettability is dependent on test liquids under consideration and vary with flow of those liquids through fibrous media.

4. In general, pressure drop of filter media is reduced with increased L/H. It means, hydrophobic fibers can aid in reduction of pressure drop.

5. The filter media MGPP (80:20) – 94%, LGPP(80:20)- 91%, LGPET(80:20) – 93%, MGFTS (60:40) – 91% shows higher separation efficiency.

6. The filter media prepared with electrospun polypropylene fibers shows significant improvement in separation efficiency, however, pressure drop also increased significantly. The more smaller size fibers i.e., 100nm in small amounts may give higher filter performance.
CHAPTER VIII

FUTURE WORK

The recommendations for future work are explained below.

1. The effect of wettability on filter performance was studied with low interfacial tension Viscor oil. The study can be further extended to study the effect of wettability on filter performance with variation in interfacial tension fuels. This study can be performed with diesel fuel or kerosene and reducing the interfacial tension of liquids by addition of surfactant.

2. The biodiesels are emerging as alternative to fuels or blends in ULSD. This leads to additional filtration requirements for diesel engines. The work can be extended to study the effect of biodiesels blends on filter performance.

3. The droplet size of water has significant influence on filter performance. In future work, performance of filter media can be evaluated with submicron size droplets. The emulsions with smaller droplets sizes can be achieved by using appropriate centrifugal pump.

4. The mixed hydrophilic/hydrophobic filter media study can be further extended to learn the effect of bed length of different geometries on filter performance.
5. The effect of interfacial tension on wettability of filter media can be studied in the future work. The L/H for different filter media can be obtained for liquids with different interfacial tensions.

6. The current set of equation provides foundation for modeling the two-phase flow through fibrous media considering the concepts of relative wettability of the filter media. The work can be further extended by incorporating a relationship for capillary pressure and saturation.
BIBLIOGRAPHY


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APPENDICES
APPENDIX A

MODELLING OF TWO-PHASE FLOW OF THROUGH FILTER MEDIA –
CORRELATION FOR PRESSURE DROP AND SATURATION THROUGH FILTER
DEPTH CONSIDERING RELATIVE PERMEABILITY OF WETTING AND NON-
WETTING PHASES IN POROUS MEDIA

The objective of this work is to model two-phase flow of water and oil through porous media and develop a correlation for saturation and pressure drop as a function of filter bed length with considering the effect of wettability.

A.1. Multiphase flow through porous media

The oil phase (continuous) carries water droplets into filter media. The droplets are captured on to the fibers; coalesce to form bigger drops and drain from the filter media. The multiphase flow through porous media can be described by multiphase continuum theory. The multiphase flow theory consider that local measurement of dispersed phase is representative of averaged effect of processes occurring at the micro scale. Volume averaging theory is used to develop multiphase continuum equations. [101]The jump balance for two-dimensional surface is used by subtracting the balances for adjoining phases from the balance on material volume at the surface.
The volume averaged for any property \( \phi \) for the \( \alpha \) phase can be written as follows

\[
\frac{\partial}{\partial t} \left( \rho_\alpha \phi_\alpha \right) + \nabla \cdot \left( \rho_\alpha \phi_\alpha \mathbf{v}_\alpha \right) + \nabla \cdot \left( \mathbf{t}_\alpha \right) - \nabla \cdot \left( \rho_\alpha \mathbf{f}_\alpha \right) + \left( E^\alpha + I^\alpha + S^\alpha + G^\alpha \right) = 0
\]  

(1)

The excess terms are constrained as

\[
\sum_{\alpha} \left( E^\alpha + I^\alpha + S^\alpha + G^\alpha \right) = 0
\]  

(2)

The jump balance approach for property \( \phi \) for singular surfaces can be written as follows.

\[
\left[ \rho_1 \phi_1 (\mathbf{w} - \mathbf{v}_1) - \mathbf{i}_1 \right] \cdot \mathbf{n}_{12} = \left[ \rho_2 \phi_2 (\mathbf{w} - \mathbf{v}_2) - \mathbf{i}_2 \right] \cdot \mathbf{n}_{12}
\]  

(3)

Where the velocity of phase 1 and 2 are \( \mathbf{v}_1 \) and \( \mathbf{v}_2 \) respectively and \( \mathbf{w} \) is the velocity of the interface. \( \rho_1 \) and \( \rho_2 \) are the density of phase 1 and 2 respectively.

The detail description of volume averaging theory is given by Vasudevan [105], Andan [106]. The coalescence filtration process can be modeled by dividing it into four stages (1) start-up stage – where saturation is very less and porosity of the filter media is constant (2) loading stage – where saturation is slightly increased but not too high (3) unsteady state – where saturation is high enough to cause drainage but it changes with time (4) steady state – where inlet and outlet rate of droplets is equal and process operates at steady state. The solutions for the start-up and loading stage is given by Vasudevan [105] and solution for the steady state is given by Chase [107].

In this work, attempt is made to develop a model for pressure drop and saturation through the depth of medium which varies with time. The rate of droplet capture also
varies with time. The conservation of species balances for oil and water species are the start-up equations used to set-up equations to obtain rate of droplet capture on the fibers. The mass balance on oil and water phases and momentum balances on oil and water phases are used to obtain saturation and pressure drop through a depth of a medium. The constitutive relations and drag force correlations are necessary to solve the mass and momentum balances for the flow through porous media. The general equations, simplified governing equations and solutions are described here.

A.2. Relative permeability, wettability and saturation of porous media

The water droplets moves through the medium on the fibers due to a drag force by bulk phase. The two-phase flow through porous media analogous to Darcy’s law for two phases can be written as follows [107],

Oil Phase

$$\varepsilon^o v^o = -\frac{k^o}{\mu^o} \nabla P$$  \hspace{1cm} (4)

Water Phase

$$\varepsilon^w v^w = -\frac{k^w}{\mu^w} \nabla P$$  \hspace{1cm} (5)

Where, \(\mu, \varepsilon, v\) and \(k\) are the viscosity, porosity, velocity and permeability of phase flow through porous media. The superscripts \(o\) and \(w\) represents the oil and water phase. \(\nabla P\) is the pressure drop. The permeability of each phase is referred as ‘effective
permeability’ or phase permeability of the porous medium with particular liquid. The ‘relative permeability’ of porous medium with each phase is defined as [108],

\[ k_r^w = \frac{k^w}{k} \]  
\[ k_r^o = \frac{k^o}{k} \]

The relationships for saturation and relative permeability of water and oil phase were given by [109-110]. The relationship assumes water as wetting fluid and oil as non-wetting fluid.

\[ k_r^w = S^3 \]  
\[ k_r^o = (1-S)^3 \]

A.3. General conservation equations

The general conservation equations for each phase are as follows,

Oil phase water species balance

\[ \frac{\partial}{\partial t} (\rho^o \rho_w) + \nabla \cdot (\rho^o \rho_w \mathbf{v}_w) + \nabla \cdot \varepsilon^o \mathbf{j}_w^o - \varepsilon^o \rho_w^o \left( f^o + g^o \right) + (E_{w}^o + l_{w}^o + \xi_{w}^o + G_{w}^o) = 0 \]  
(10)

Oil phase mass balance

\[ \frac{\partial}{\partial t} (\rho^o \rho^o) + \nabla \cdot (\rho^o \rho^o \mathbf{v}) + (E^o_m + G^o_m) = 0 \]  
(11)
Oil phase momentum balance
\[
\frac{\partial}{\partial t} (\varepsilon^O \rho^O \mathbf{v}^O) + \nabla \cdot (\varepsilon^O \rho^O \mathbf{v}^O \mathbf{v}^O) + \varepsilon^O \nabla P + \nabla \cdot \varepsilon^O \mathbf{f}_w^O + E^O_{z} - \varepsilon^O \rho^O g + I_M^O + S_M^O = 0 \quad (12)
\]

Water phase mass balance
\[
\frac{\partial}{\partial t} (\varepsilon^W \rho^W \mathbf{v}^W) + \nabla \cdot (\varepsilon^W \rho^W \mathbf{v}^W \mathbf{v}^W) + (E_m^W + G_m^W) = 0 \quad (13)
\]

Water phase momentum balance
\[
\frac{\partial}{\partial t} (\varepsilon^W \rho^W \mathbf{v}^W) + \nabla \cdot (\varepsilon^W \rho^W \mathbf{v}^W \mathbf{v}^W) + \varepsilon^W \nabla P + \nabla \cdot \varepsilon^W \mathbf{f}_w^W + E^W_{z} - \varepsilon^W \rho^W g + I_M^W + S_M^W = 0 \quad (14)
\]

A.4. Assumptions

The following assumptions simplify the multiphase continuum equations

- The process is isothermal.
- The system is at quasi-steady state.
- The flow is one dimensional (only in the z direction).
- The media is incompressible. Solid phase is stationary.
- The filter coefficient is a constant.
- The water concentration in the oil phase is low, hence the bulk density of the incoming emulsion is a constant and approximately equal to the density of oil at that temperature.
- The fibers do not move within the matrix (held stationary by the binder).
- The porosity of a new unused media is uniform and constant.

Alternatively, the fiber phase volume fraction is uniform and constant.
The permeability of a new unused media is uniform and isotropic.

There is no re-entrainment of water.

No chemical reaction.

Inertial (convection) terms are neglected.

No property transfer due to slip. No phase change.

Viscous forces are neglected.

Viscosity of air and liquid are constant. No temperature effects.

No surfactants presents and Interfacial tension of liquid is not low.

The simplified general equations after applying above assumptions are as follows,

Oil phase water species balance

\[
\frac{\partial}{\partial t} \left( \epsilon^o \rho_w^o \right) + \nabla \cdot \left( \epsilon^o \rho_w^o \mathbf{v}_w^o \right) + \left( I_w^o \right) = 0
\]  

(15)

Oil phase mass balance

\[
\frac{\partial}{\partial t} \left( \epsilon^o \rho^o \right) + \nabla \cdot \left( \epsilon^o \rho^o \mathbf{v}^o \right) + \left( E_m^o \right) = 0
\]  

(16)

Oil phase momentum balance

\[
\epsilon^o \nabla P + E_{\epsilon}^o = 0
\]  

(17)

Water phase mass balance

\[
\frac{\partial}{\partial t} \left( \epsilon^w \rho_w^w \right) + \nabla \cdot \left( \epsilon^w \rho_w^w \mathbf{v}_w^w \right) + \left( E_m^w \right) = 0
\]  

(18)
Water phase momentum balance

\[ \varepsilon^w \nabla P + F^w_z = 0 \]  \hspace{1cm} (19)

Constraints from volume averaging theory

Species and mass continuity

\[ \sum I^O_w = E^O_m \quad \text{Therefore}, \quad I^O_w = E^O_m = -E^w_m \]  \hspace{1cm} (20)

Constitute relation from filtration theory

\[ I^G_O = \alpha \rho^O_w \varepsilon^O \Sigma^O_z \]  \hspace{1cm} (21)

Where, \( \alpha \) is filter coefficient which is constant.

Hassanizadeh and Gray [101] introduce the constitutive relation, which can be written for oil and water phase as follows

\[ F^O = R^O \cdot (\nu^O - \nu^S) \]  \hspace{1cm} (22)

\[ F^W = R^W \cdot (\nu^W - \nu^S) \]  \hspace{1cm} (23)

Where, the stationary solid phase velocity, \( \nu^S \) is zero. \( R \) is a second order tensor resistance function that is characteristic of the material properties and the flow conditions.

Porosity of the filter medium, \( \varepsilon \), is ratio of volume of voids to the volume of filter and saturation, \( S \), is the ratio of volume of water to the volume of voids. The saturation,


\( S \), is related to the porosity of filter medium, \( \varepsilon \) and the water and oil phase volume fractions, \( \varepsilon^W \) and \( \varepsilon^O \), by,

\[
S = \frac{\varepsilon^W}{\varepsilon}
\]

\[
\varepsilon^W = S\varepsilon
\]

\( \varepsilon^O = (1-S)\varepsilon \)


The mass fraction of water in oil phase is the ratios of mass concentrations:

\[
w^O_0 = \frac{\rho^O_w}{\rho^O} \]

Define, \( w = \left. \frac{\omega^O_w}{\omega^O} \right|_{t_0} \), The quantity varies between 0 and 1.

\( \omega^O_w \)


A.5. Governing equations and boundary conditions

Substituting above equations into equations (15) to (19), we get,

Oil phase water species balance

\[
\frac{\partial}{\partial t} ((1-S)\omega) + \frac{\partial}{\partial z} ((1-S)\bar{v}_z^O \omega) + \alpha(1-S)\omega \bar{v}_z^O = 0
\]

(27)

Oil phase mass balance

\[
\frac{\partial}{\partial t} (1-S) + \frac{\partial}{\partial z} (1-S)\bar{v}_z^O + \alpha(1-S)\omega \bar{v}_z^O = 0
\]

(28)

Oil phase momentum balance

\[
\varepsilon(1-S)\frac{\partial P}{\partial z} + F^O_z = 0
\]

(29)
Water phase mass balance

\[
\frac{\partial}{\partial t} (S) + \frac{\partial}{\partial z} (S\bar{v}_z) - \alpha (1-S) \omega_o \left| \omega_o \frac{\rho_o}{\rho_w} \bar{v}_z \right| = 0
\]  

(30)

Water phase momentum balance

\[
(\varepsilon S) \frac{\partial \bar{p}}{\partial z} + F_z^w = 0
\]

(31)

Initial and boundary conditions

Saturation before start-up is zero

\[
S^o_{t=0} = 0
\]  

(32)

\[
\omega^o_{t=0} = 1
\]  

(33)

\[
P^o_{Z=Z} = P_{aim}
\]  

(34)

A.6. Solutions

By solving the oil phase mass balance and applying jump balance at the inlet and outlet surface of filter media. The jump balance balances the mass of water in oil at inlet boundary and mass of water at the exit boundary and results in following expression,

\[
(1-S)\bar{v}_z^o \bigg|_{t=0} = \frac{m}{A \varepsilon \rho^o}
\]  

(35)
By using equations 5, 7 and 9, the Darcy’s law for oil and by using equations 6, 7 and 9 the Darcy’s law for water can be written as,

**Oil Phase**

\[ \varepsilon^O v^O = -\frac{k k^O}{\mu^O} \nabla P \Rightarrow \varepsilon^O v^O = -k(1-S)^2 \frac{dP}{\mu^O dz} \]  \hspace{1cm} (36)

**Water Phase**

\[ \varepsilon^W v^W = -\frac{k k^W}{\mu^W} \nabla P \Rightarrow \varepsilon^W v^W = -kS^2 \frac{dP}{\mu^W dz} \]  \hspace{1cm} (37)

Now, solving oil phase momentum balance by using constitutive relation (equation 22) and Darcy’s law for oil phase (equation 36), we get

\[ \frac{dP}{dz} = -\varepsilon^O v^O = -\frac{k(1-S)^2 dP}{k(1-S)^2 dz} \]  \hspace{1cm} (38)

Solving water phase momentum balance by using constitutive relation (equation 23) and Darcy’s law (equation 37), we get

\[ \frac{dP}{dz} = -\varepsilon^W v^W = -\frac{kS^2 dP}{kS^2 dz} \]  \hspace{1cm} (39)

Dullien [108] described, the pressures in any two phases at any point in the porous media are assumed to be related to each other via the capillary pressure, \( P_c \). Hence, two pressure gradients are related to each other by the gradient of capillary pressure [108].
\[ \frac{dP_w}{dz} = \frac{dP_c}{dz} + \frac{dP_o}{dz} \]  (40)

To further solve these equations, a constitutive relationship between capillary pressure and saturation is necessary. At present, by assuming capillary pressure is very negligible the equations (38), (39), (40) and (35) can be solved to get velocity of water phase through a porous media,

\[ v_w^z = -\frac{S^2}{(1-S)^2} \frac{\mu^o}{\mu_w} v_o^z \]  (41)

\[ v_w^z = -\frac{S^2}{(1-S)^3} \frac{\mu^o}{\mu_w} \frac{m}{A\varphi^o} \]  (42)

Substituting velocities of oil and water phase in water species balance and water phase mass balance, we get the equations relating saturation and rate of droplets capture as a function of filter bed length and time. These expression accounts for the wettability (in terms of relative permeability) of each phase in porous media.

Oil phase water species balance

\[ \frac{\partial}{\partial t} (1-S)\omega + \left( \frac{m}{A\varphi^o} \right) \frac{\partial}{\partial z} (\omega) + \alpha\omega \frac{m}{A\varphi^o} = 0 \]  (43)

Water phase mass balance

\[ \frac{\partial}{\partial t} \left( \frac{S^3}{(1-S)^3} \right) + \left( \frac{\mu^o}{\mu_w A\varphi^o} \right) \frac{\partial}{\partial z} \left( \frac{S^3}{(1-S)^3} \right) - \alpha\omega_w \frac{m}{\rho_w A\varepsilon} = 0 \]  (44)
These equations (43) and (44) are non-linear partial differential equations and can be solved with appropriate numerical scheme and computer programming. The solutions to these equations will give saturation profile through a depth of filter media with variation in time and the rate of droplet capture through filter bed length and as function of time. In future work, the constitutive relation for capillary pressure - saturation ($P_C - S$) should be used to learn the effect of wettability on filter performance.
APPENDIX B

NOMENCLATURE

\[ A \] Cross sectional area of the filter, \( m^2 \)

\[ E^o \] Interface transfer excess term for the oil phase, \( \text{kg/m}^3\text{s} \)

\[ E^w \] Interface transfer excess term for the water phase, \( \text{kg/m}^3\text{s} \)

\[ I^o_w \] Interface transfer excess term, \( \text{kg/m}^3\text{s} \)

\[ \cdot \] Oil phase mass flow rate, \( \text{m}^3/\text{s} \)

\[ S \] Saturation of oil media (volume of water per volume of voids)

\[ v^o_z \] Velocity of oil in the z direction, \( \text{m/s} \)

\[ \bar{v}^w_z \] Velocity of water phase in z direction \( \text{m/s} \)

\[ z \] Distance into the filter medium from the inlet surface, \( \text{m} \)

\[ \alpha \] Filter coefficient, \( \text{l/m} \)

\[ \varepsilon^o \] Porosity (void volume fraction) of the oil phase
\( \varepsilon^w \) Water phase volume fraction

\( \varepsilon \) Porosity of filter medium

\( \rho_o^o \) Mass concentration of oil in the oil phase, kg/m³

\( \rho_w^o \) Mass concentration of the water species in the oil phase, kg/m³

\( \rho^o \) Mass density of the oil phase, kg/m³

\( \rho^w \) Water phase density on the fibers, kg/m³

\( \mu^o \) Oil phase viscosity, kg/ms

\( \mu^w \) Water phase viscosity kg/ms

\( k \) Permeability of the filter media (m²)

\( k^o \) Oil phase effective permeability of filter media (m²)

\( k^w \) Water phase effective permeability of filter media (m²)

\( k_r^o \) Oil phase relative permeability of filter media (m²)

\( k_r^w \) Water phase relative permeability of filter media (m²)
APPENDIX C

EXPERIMENTAL RESULTS USING JORIN ViPA INSTRUMENTS

The Jorin ViPA (Visual Process Analyzer) is a particle size analysis system. The ViPA uses a video microscope to capture images of the discrete objects, or ‘species’ (i.e. solid particles, liquid droplets, gas bubbles), present in a given multiphase (i.e., solid/liquid or liquid/liquid) process stream. The instrument is designed to operate continuously, on-line and at process temperature and pressure. Image analysis techniques are then applied to differentiate between the different object populations present and the measurable characteristics of these populations - such as size, size distribution and relative concentrations (in Vppm – Visual parts per million) are reported by the instrument.

The instrument was used for the upstream and downstream sampling of liquid - liquid coalescence experiment. In this instrument, the liquid-liquid emulsion was passed through a backlit flow cell. The high speed digital video camera rapidly captures images of fluid. The software then analyze the particles and droplets for their size, shape factor, optical density, concentration etc. The instrument provides information about seventeen different parameters for each particle measured. The important user defined settings necessary for the software are given below. These parameters varies based on the multiphase system under consideration and different types of particles to be measured.
The strength of this instrument is, it can distinguish between solid particles, liquid droplets and air bubbles.

Important settings in the JORIN ViPA

1. Flow rate: 30-50 ml/min

2. Particle Classes

   Table C.3. Particle classes and their settings

<table>
<thead>
<tr>
<th>Parameter/Particle Class</th>
<th>Water</th>
<th>Air</th>
<th>Solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size (microns)</td>
<td>1.5 - 1000</td>
<td>1.5 - 1000</td>
<td></td>
</tr>
<tr>
<td>Shape Factor</td>
<td>0.8 – 1.0</td>
<td>0.8 -1.0</td>
<td>0.0-0.6</td>
</tr>
<tr>
<td>Optical Density</td>
<td>0.0 - 0.45</td>
<td>0.45- 2</td>
<td></td>
</tr>
</tbody>
</table>

3. Mode of operation: Continuous mode

Liquid-liquid experiments and data analysis

- The sampling was done in continuous mode and typical plot of mean drop size and concentration in ppm over time is shown in Figure C.1 and Figure C.2 respectively. The typical upstream and downstream cumulative concentration distributions are shown in Figure C.3 and Figure C.4 respectively. The x-axis in this plots shown proportion in percentage and y-axis is concentration in ppm for that particular cycle.

- The upstream and downstream mass concentrations was calculated as the average of three cycles (1 cycle was 1 sampling) and efficiency is calculated using average of 3 experiments of each filters. The upstream
was measured over first 5 cycles and then downstream was measured continuously for rest of experiment.

- In entire experiment, JORIN ViPA performed about 70-80 cycles (samplings). In continuous mode this depend on how long experiment is performed and also on the settings in the software for minimum number of particles required for each sampling. In each cycle instruments takes about 550 frames (images) and based on these images it provides measurements of species (given particles classes in Table C.1) present in stream.

- The JORIN ViPA instrument was used for upstream and downstream sampling during the experiments with different mixed glass/polypropylene filter media. The comparison of efficiency values obtained using JORIN ViPA and Accusizer is shown in Figure C.5.

- The results obtained from the both instruments are comparable. However, the efficiency results obtained from Jorin ViPa and Accusizer shows that the efficiency values from Jorin ViPA are slightly higher as compare to Accusizer. The following are the possible reasons for the difference between the measurements of the two instruments.

1. The Accusizer measures particles in the range of 0.5 µm to 500 µm. The Jorin ViPA measures particles in the range of 1.5 µm to 1000 µm. The droplets below the 1.5 µm range were not considered in the calculations and this can account in the difference in the efficiency values.
2. The downstream sampling points were placed after the gravity settling tank. The sample for Accusizer, which uses batch sampling method, was taken from the stream exiting the settling tank. However, downstream sampling using Jorin ViPA is done in continuous mode and stream passing through flow cell of Jorin ViPA passed through a larger diameter (3/4”) joint, gate valve and connecting pipe. There is possibility of settling of some of the larger drops before reaching the flow cell. The further modification in the sampling technique may give more comparable results.

Figure C.1. Mean size of water and solids measured with time. First 5 cycles represents upstream sampling and downstream was measured for rest of the experiment. Each data point represents mean size obtained for 1 cycle (i.e. 1 sampling).
Figure C.2. Concentration (ppm) of water and solids measured with time. First 5 cycles represents upstream sampling and downstream was measured for rest of the experiment. Each data point represents concentration obtained for 1 cycle (i.e. 1 sampling). The concentration of solids and air can also be plotted here but settings are made only for water.
Figure C.3. Cumulative concentration distributions at upstream. Ch=1, Ch=2 and Ch=3 represents the channels of water, solids and air particles respectively. The concentration (Conc (ppM) = 50.39) is the Vppm of water at the upstream.
Figure C.4. Cumulative concentration distributions at downstream. Ch=1, Ch=2 and Ch=3 represents the channels of water, solids and air particles respectively. The concentration (Conc (ppM) = 3.62 is the Vppm of water at the upstream.
Figure C.5. Comparison of Accusizer and JORIN ViPA instruments with filter efficiency of different mixed filter media.
The objective of this work is to learn about the relationship between the L/H value of filter media of particular geometry and material with the composition of polymer fibers in a filter media. The polynomial regression model of L/H and fraction of polymers fibers in the filter media were developed using a method of least squares. The MiniTab 15 software was used to develop the models. The regression analysis can also be developed using excel data analysis tool pack.

The separate regression models for the mixed glass/polypropylene media, layered glass/polypropylene media and layered glass/polyester media were developed. The developed models for different media geometries are discussed below.

Mixed glass/polypropylene media

The data used for regression analysis is shown in Table D.1. The fraction of PP fibers as independent variable and L/H as response variable.
Table D.1. Fraction of PP fibers and L/H values of mixed media

<table>
<thead>
<tr>
<th>Fraction of Polypropylene fiber</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.47</td>
</tr>
<tr>
<td>0.2</td>
<td>2.29</td>
</tr>
<tr>
<td>0.4</td>
<td>3.09</td>
</tr>
<tr>
<td>0.6</td>
<td>14.20</td>
</tr>
<tr>
<td>0.8</td>
<td>58.77</td>
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</table>

The regression model developed is shown in equation D.1 and has a $R^2 = 0.90$.

The regression model fit with experimental data is shown in Figure D.1 and residual plots are shown in Figure D.2. The P-value = 0.05 shows that model is statistically useful.

$$L/H = 171.1x^2 - 72.62x + 3.749$$

(D.1)

Where, $x$ is the fraction of polypropylene fibers in the filter media. The standard error for the coefficients of the model are, 51.945 for $x^2$, 43.336 for $x$ and 7.317 for constant.

The model results are shown below.

**Polynomial Regression Analysis: L/H versus Fraction of PP fiber**

The regression equation is

$L/H = 3.749 - 72.62$ Fraction of PP fiber $+ 171.1$ Fraction of PP fiber$^2$

$S = 7.77441 \quad R$-Sq $= 95.0\% \quad R$-Sq(adj) $= 90.0\%$

Analysis of Variance

| Source | DF | SS  | MS  | F   | P   |
Regression  2  2307.16  1153.58  19.09  0.050  
Error        2   120.88    60.44  
Total        4  2428.05  

Sequential Analysis of Variance  

<table>
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<th>F</th>
<th>P</th>
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<td>1651.48</td>
<td>6.38</td>
<td>0.086</td>
</tr>
<tr>
<td>Quadratic</td>
<td>1</td>
<td>655.68</td>
<td>10.85</td>
<td>0.081</td>
</tr>
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</table>

**Fitted Line: L/H versus Fraction of PP fiber**

**Residual Plots for L/H**

![Residual Plots for L/H](image)

Figure D.1. Fitted line plot for L/H Vs fraction of PP fibers for mixed fiber media
Layered glass/polypropylene media

The data used for regression analysis is shown in Table D.2. The fraction of PP fibers as independent variable and L/H as response variable.

Table D.2. Fraction of PP fibers and L/H values of layered media

<table>
<thead>
<tr>
<th>Fraction of Polypropylene fiber</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.86</td>
</tr>
<tr>
<td>0.2</td>
<td>3.00</td>
</tr>
<tr>
<td>0.4</td>
<td>7.29</td>
</tr>
</tbody>
</table>
The regression model shown in equation D.2 shows good fit with experimental data with $R^2 = 0.959$ as shown in Figure D.3. The residual plots are shown in Figure D.4. The P-value = 0.021 which less than 0.05, shows that model is statistically useful.

$$\frac{L}{H} = 68.32x^2 - 2.84x + 0.516$$

(D.2)

The standard error for the coefficients of the model are, 23.600 for $x^2$, 19.689 for $x$ and 3.324 for constant.

The model results are shown below.

**Polynomial Regression Analysis: L/H versus Fraction of PP fiber**

The regression equation is

$L/H = 0.516 - 2.84 \text{ Fraction of PP fiber} + 68.32 \text{ Fraction of PP fiber}^2$

$S = 3.53214 \quad R^2 = 97.9\% \quad R^2(\text{adj}) = 95.9\%$

### Analysis of Variance

<table>
<thead>
<tr>
<th>Source</th>
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<th>MS</th>
<th>F</th>
<th>P</th>
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</thead>
<tbody>
<tr>
<td>Regression</td>
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<td>1178.68</td>
<td>589.342</td>
<td>47.24</td>
<td>0.021</td>
</tr>
<tr>
<td>Error</td>
<td>2</td>
<td>24.95</td>
<td>12.476</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>4</td>
<td>1203.64</td>
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### Sequential Analysis of Variance

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<tbody>
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<td>1074.12</td>
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<td>0.101</td>
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</table>

**Fitted Line: L/H versus Fraction of PP fiber**

**Residual Plots for L/H**
Figure D.3. Fitted line plot for L/H Vs fraction of PP fibers for layered fiber media

Figure D.4. Residual plots for L/H and fraction of PP fibers for layered fiber media
Layered glass/polyester media

The data used for regression analysis is shown in Table D.3. The fraction of PET fibers as independent variable and L/H as response variable.

Table D.3. Fraction of PET fibers and L/H values of layered media

<table>
<thead>
<tr>
<th>Fraction of PET fiber</th>
<th>L/H</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.86</td>
</tr>
<tr>
<td>0.2</td>
<td>4.26</td>
</tr>
<tr>
<td>0.4</td>
<td>8.11</td>
</tr>
<tr>
<td>0.6</td>
<td>24.93</td>
</tr>
<tr>
<td>0.8</td>
<td>45.39</td>
</tr>
</tbody>
</table>

The regression model shown in equation D.3 shows very good fit with experimental data with $R^2 = 0.989$ as shown in Figure D.5. The residual plots are shown in Figure D.6. The P-value = 0.006 which less than 0.05, shows that model is statistically useful.

$$\frac{L}{H} = 84.09x^2 - 12.41x + 1.491$$  \hspace{1cm} (D.3)

The standard error for the coefficients of the model are, 13.227 for $x^2$, 11.035 for $x$ and 1.863 for constant.

The model results are shown below.

**Polynomial Regression Analysis: L/H versus Fraction of PET fiber**

The regression equation is
L/H = 1.491 - 12.41 Fraction of PET fiber + 84.09 Fraction of PET fiber**2

S = 1.97964  R-Sq = 99.4%  R-Sq(adj) = 98.9%

Analysis of Variance

<table>
<thead>
<tr>
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<tr>
<td>Regression</td>
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<td>1362.46</td>
<td>681.229</td>
<td>173.83</td>
<td>0.006</td>
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<tr>
<td>Error</td>
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<td>7.84</td>
<td>3.919</td>
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<td></td>
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<tr>
<td>Total</td>
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<td>1370.30</td>
<td></td>
<td></td>
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</table>

Sequential Analysis of Variance

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</tr>
</thead>
<tbody>
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<td>Linear</td>
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<td>1204.07</td>
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<td>0.019</td>
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<tr>
<td>Quadratic</td>
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<td>158.39</td>
<td>40.42</td>
<td>0.024</td>
</tr>
</tbody>
</table>

Fitted Line: L/H versus Fraction of PET fiber

Residual Plots for L/H

Figure D.5. Fitted line plot for L/H Vs fraction of PET fibers for layered fiber media
Figure D.6. Residual plots for L/H and fraction of PET fibers for layered fiber media