Effect of Liquid Characteristics on the Wetting, Capillary Migration, and Retention Properties of Fibrous Polymer Networks

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ABSTRACT: Fluid penetration through porous networks consists of two different phenomena: (1) pore fluid displacement and (2) fluid flow through the pores. The first phenomenon depends on the pore size, the fluid–fluid interfacial tension, and the contact angle. The second phenomenon is pore-size- and viscosity-dependent. We adapted an experimental methodology often used for measurements of liquid permeability and hydraulic conductivity of soils and applied it to polymeric medical textiles. The methodology made use of a pressure/flow cell in which a sample was mounted. The flow rates were measured during sequences of increasing and decreasing pressures applied to the displacing nonwetting fluid (aqueous solution). The effects of the liquid parameters on penetration were investigated. Surface tension effects were studied with water and two solutions with surface tensions lower than that of pure water; the liquids with lower surface tensions had lower displacement pressures. To study viscosity effects, we used water and two solutions with viscosities higher than that of pure water. Increasing the viscosity not only caused the flow rate to decrease but also caused deformation, that is, enlargement, of the pores. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 97: 282–292, 2005

Key words: barrier; tension; viscosity

INTRODUCTION

Because penetration is difficult to measure, most research has been limited to the surface characteristics of the porous materials, that is, their wettability. However, not much has been done to determine how liquids behave inside the materials. This is because it is possible to observe the surface of the medium, but not the interior. Especially with very thin materials, such as fabrics, determining the material characteristics has been a major problem because the pore sizes are at a microscopic or even nanoscopic level. In this article, we present an experimental method that is similar to a methodology often used in soil physics to measure the liquid permeability and hydraulic conductivity. We adapted this method and used it for permeability measurements of polymeric medical textiles. However, even though the method was similar, the properties of the porous media were completely different. Soils are, under most circumstances, highly hydrophilic, whereas our fabric samples, consisting of polypropylene (PP) and polyester (PE), were hydrophobic. In a hydrophilic porous medium, the smallest pores are filled with water first as a result of the spontaneous attraction between the solid and liquid phases. On the other hand, in a hydrophobic porous medium, the biggest pores are filled first with water because they have the lowest displacement pressure. To understand liquid penetration into fabrics better, we must understand the penetration into individual pores. Because the pores were very small, they were considered capillaries. Penetration was studied by the consideration of two separate phenomena: (1) pore fluid displacement and (2) fluid flow through the pores. The first phenomenon depends on the pore size, liquid surface tension, and contact angle. When present in porous media, immiscible fluids, that is, fluids that do not mix with each other, are separated from each other by a curved interface, which is called a meniscus.[1] The pressure jump that exists across the interface is called the capillary pressure [P_c (Pa)]. The immiscible fluids that may be present simultaneously in the porous medium exert forces of adhesion or attraction of different intensities toward the pore surface, and this results in a competition by these fluids for the occupancy of the pore surface. The magnitude of these attraction forces is characterized by the contact angle. A contact angle lower than 90° indicates that the liquid wets the fabric surface; a contact angle greater than 90° indicates that the fabric surface is not wetted by the

TABLE I
Fabric Sample Properties

Sample		Thickness (mm)	Smallest pore size (μm)	Biggest pore size (μm)	Average pore size (μm)
A	100% PP	0.36 (0.012)	7.88 (1.38)	14.48 (0.36)	8.38 (1.11)
B	100% PP	0.36 (0.013)	11.62 (3.52)	25.00 (3.70)	13.80 (1.19)
C	80% PE and 20% wood pulp	0.29 (0.006)	15.55 (2.00)	36.66 (6.00)	30.00 (5.00)
D	100% PP	0.46 (0.013)	14.46 (1.16)	33.69 (0.62)	17.77 (0.77)
E	100% PP	0.36 (0.031)	12.87 (2.02)	36.77 (2.53)	19.22 (1.75)

Standard deviation values are in parentheses.

liquid. P_c is determined by the Young and Laplace equation:

$$P_c = \frac{2\gamma \cos \theta}{r} \tag{1}$$

where γ is the liquid surface tension (N/m), θ is the liquid/material contact angle, and r is the pore radius (m).

The second phenomenon, that is, flow through the pores, is pore-size- and viscosity-dependent. Poiseuille's equation is used to show the fluid-transmitting abilities of cylindrical pores:[2–4]

$$Q = \frac{\pi r^4}{8\eta} \frac{(P_0 - P_1)}{L} \tag{2}$$

where Q is the flow rate (m³/s), P_0 is the liquid pressure at the beginning of the capillary (Pa), P_1 is the liquid pressure at the end of the capillary (Pa), η is the liquid viscosity (Pa s), and L is the pore length (m).

Poiseuille's equation has been used to show the effect of the pore diameter on a transmitting fluid. The flow rate is proportional to r^4, whereas the cross-sectional area of a pore is proportional to r^2. Therefore, one large pore with the same cross-sectional area as that of several smaller pores will transmit considerably more fluid because of less viscous drag along the wall.[5]

The penetration pressure (also known as the displacement pressure or entry pressure) of protective materials, such as surgical garments, is an indicator of barrier performance. For a protective material, it is defined as the pressure value at which the first liquid drops are observed at the surface opposite from which the liquid is applied. The higher this penetration pressure is, the better the protection performance is. However, in certain cases, moisture and air permeability also need to be considered.[6]

Hollies et al.[7,8] studied the effects of yarn roughness and capillary-type penetration in yarns and fabrics on water-transport mechanisms. They showed that water movement in yarns is directly related to the advancing contact angle of water on the yarn and only indirectly related to the surface shape of the fiber material.

Fuzzy yarns, containing randomly arranged fibers, are usually nonwetting because of large water contact angles and the existence of discontinuous capillaries.[7] Both the amount of water carried by the fabric and the distance that it travels in a unit of time are influenced considerably by the randomness of the arrangement of fibers in the yarns.[8]

Minor and coworkers[9–11] observed that the fabric tightness and denseness have an influence on the capillary penetration. They observed that the fuzziness, or quantity and stiffness of the nap, is the most important fabric property influencing the penetration.

Most research to date has investigated the effect of fiber or fabric properties on penetration. Not much information is available on the effects of liquid properties such as the surface tension, viscosity, and contact angle. Miller and Schwartz[12] showed that the most important factor determining the liquid pressure required to enter a material is the wettability of the material. They did not, however, consider the exact pore size distribution and its effect on the penetration and flow rates. Instead, they estimated the pore size distribution with scanning electron microscopy (SEM) images. Our research addresses this void by evaluating the actual effect of the pore size distribution and liquid parameters on penetration (fluid displacement and subsequent fluid flow).

EXPERIMENTAL

Fabric materials

Five barrier fabrics (A–E) were tested. Fabrics A and B were provided by Kimberley Clark (Roswell, GA) and were made of 100% PP. They are commonly used for protective medical garments, such as surgical gowns and drapes. Fabric C was medical Sontara; it was provided by DuPont (Wilmington, DE) and was made of PE and wood pulp fibers. Fabrics D and E, also used for surgical gowns, drapes, and leggings, were provided by BBA Nonwovens, Inc. (Simpsonville, SC) and consisted of 100% PP. The values of the fabric sample properties are given in Table I. Each value is the average of five replicate measurements.
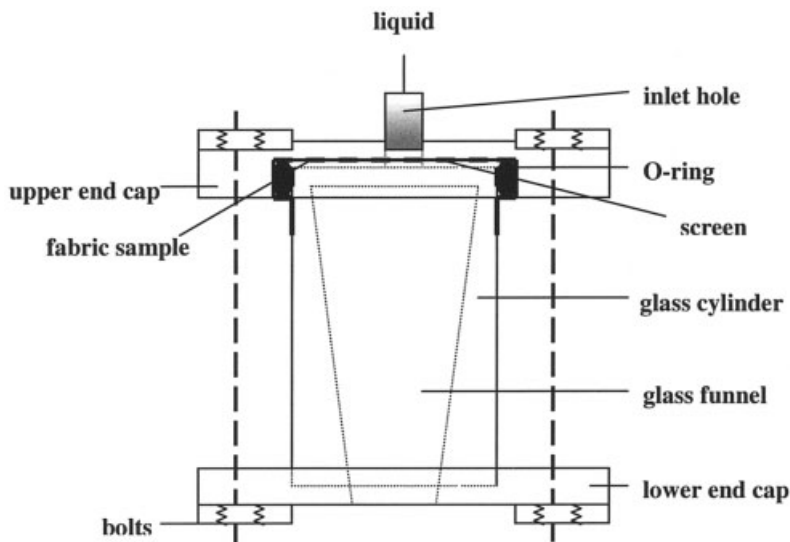
Figure 1 Pressure cell.

Material testing

The fabric thickness was determined with a digital micrometer (TMI, Ronkonkoma, NY) according to the guideline of ASTM Standard D 1777. The pore size distributions (Table I) were determined with a capillary flow porometer (Porous Materials, Inc., Ithaca, NY) and a scanning electron microscope. SEM images were obtained for all samples before and after each fluid penetration test so that we could visually inspect possible changes in the pore sizes due to testing. In comparison with the pore size distributions determined by the capillary porometer, the pore sizes measured from SEM images did not result in exact pore size distributions. They only provided an approximation of the average pore sizes.[5] Apparently, surface imaging is not an adequate way of describing the pore size distribution because it does not consider the bulk properties of the material. Nevertheless, approximations of the porosity were made with SEM images because the capillary flowmeter did not provide porosity values.

Experimental setup

The experimental setup that we designed consisted of a pressure/flow cell, which was used for measuring the displacement pressures and flow rates (Fig. 1). A schematic diagram (Fig. 2) depicts the configuration of the total experimental setup. The fabric sample was clamped between the upper end cap and a Viton O-ring (Parker Seal Group, Lexington, KY). The latter was supported by a glass cylinder, which was positioned between the upper and lower end caps. The cylinder's interior diameter was 80 mm, and its height was 100 mm. The caps were secured in place with threaded rods and nuts. The upper cap had an inlet hole for the invading liquid. To help minimize sagging of the sample, we supported the fabric sample with a rigid metal screen with openings large enough to allow air and water to pass freely through it. The displacing fluid was placed in a glass reservoir with a volume of 8 L (Fig. 2). The top of the reservoir was connected to an air pressure source to provide the
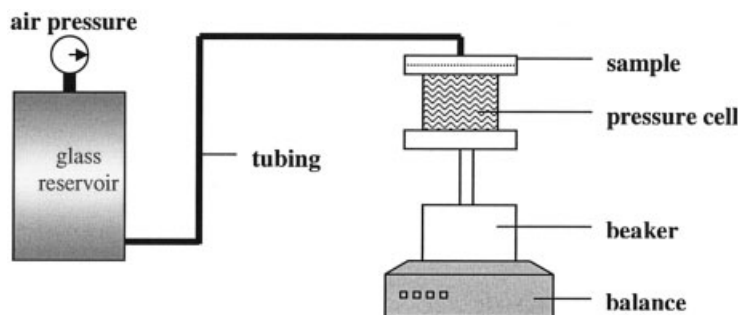

Figure 2 Test methodology.

required liquid pressure at the point of contact with the fabric sample.

When a sample was clamped between the screen and the O-ring, some damage could occur at the edges of the fabric, if not at the time of clamping, then during flow, especially under high pressures. To avoid anomalies in the flow results, we placed a glass funnel with a diameter smaller than the glass cylinder under the fabric to eliminate the collection of liquid from the edges of the fabric. The funnel diameter was 65 mm at the top. It was also important that the fabric's physical characteristics did not change over time. The size of the screen openings, therefore, had to be neither too small nor too large. Small openings could have resulted in a reduction in the available fabric surface area for flow. Large openings could have resulted in local sagging and a concomitant increase in the pore size. However, under high liquid pressures, some physical deformation could still occur.

Fluid penetration testing

The liquid pressure on top of the fabric at which the first liquid drops appeared at the bottom of the fabric sample was assumed to be the penetration pressure. At this point, the fabric was no longer able to provide further resistance, and the solution started to flow through. The liquid pressure was then increased in increments of 1 kPa (10 cm of H_2O) until a preset maximum pressure was reached. We refer to this first cycle as the initial displacement of air by water, or the initial air drainage phase. The changes in the liquid level in the supply bottle were accounted for in the calculation of the liquid pressure values on top of the fabric. Once the maximum preset pressure value was reached, the pressure was stepwise decreased in 1-kPa intervals to check for possible hysteresis in the flow-rate/pressure relations. This time, as the pressure decreased, the liquid started to drain from the pores, and the pores were filled with air again. This second cycle is called the main displacement of water by air, or the main air wetting curve. The pressure was reduced until the flow stopped. At each pressure, the flowing liquid was collected in a beaker positioned on a balance (Fig. 2), and the mass of the liquid per unit of time was recorded.

This article also addresses the effects of liquid parameters, that is, the surface tension, viscosity, and material/liquid contact angle, on the penetration and flow.

Surface tension tests

To determine the surface tension effect, we tested all fabric samples with deionized water and two solutions with surface tensions lower than that of water. Solution I had 4% (weight basis) surfactant (Tween 80,

TABLE II
Properties of Solutions

Solution		Surface tension (N/m)	Viscosity (cp)	Density (g/cm^3)
Water		71.6 (0.17)	1.00	0.99
I	4% Tween 80	39.67 (0.35)	1.20	1.00
II	1% Griffwet	33.3 (0.21)	1.30	0.99
III	1.5% PVA	67.29 (0.24)	2.46	0.99
IV	3% PVA	67.69 (0.31)	6.25	1.00

Standard deviations values are in parentheses.

Sigma–Aldrich, Milwaukee, WI). Solution II had 1% (weight basis) Griffwet NP-9 (GriffTex, Opelika, AL). Because the surfactants formed a gel in contact with water, they were stirred with a magnetic stirrer for 24 h to completely dissolve the gel. All surface tensions (Table II) were measured with a DuNoüy tensiometer (CSC Scientific Co., Inc., Fairfax, VA). Because our samples were all medical textiles, we wanted to measure their performance under realistic conditions. The surface tension of human blood is approximately 40 N/m.[13,14] We, therefore, prepared solution I to have a surface tension close to this value. The viscosities of all liquids were determined with a viscometer (Gilmont Instruments, Barrington, IL), which operated by measuring the rate of settling of a spherical ball. The liquid density was determined with a pycnometer. The solution properties are given in Table II.

Viscosity tests

To determine the viscosity effect on the flow rates, we used deionized water and two solutions with viscosities greater than that of water. Poly(vinyl alcohol) (PVA) (Sigma–Aldrich) was used to prepare the two solutions; solution III contained 1.5% PVA (weight basis), and solution IV contained 3% PVA (weight basis). PVA was hot-water-soluble; therefore, the water was first heated to 90°C before PVA was added, and the mixture was stirred for 24 h with a magnetic stirrer. The surface tensions of the solutions were also measured. The solution properties are shown in Table II. The data show that solutions III and IV had surface tension values similar to that of water.

Contact-angle tests

The contact angles of all sample/solution combinations were determined with a Cahn dynamic contact-angle analyzer (Cahn Instruments Corp., Madison, WI). These measurements were taken at the surface of the fabric; therefore, the macroscopic contact angle was determined. It did not necessarily correlate with the microscopic contact angle inside the material's pores. The contact angles of all solution/material combinations are given in Table III.

TABLE III
Fabric/Solution Contact Angles (°)

Sample	Water	Solution I	Solution II	Solution III	Solution IV
A	100 (10)	98 (15)	82 (5)	95 (1)	102 (11)
B	95 (3)	95 (9)	87 (1)	99 (1)	99 (1)
C	101 (8)	102 (14)	92 (2)	110 (15)	121 (3)
D	89 (1)	35 (3)	0 (0)	100 (2)	99 (5)
E	91 (5)	35 (7)	0 (0)	92 (7)	92 (1)

Standard deviation values are in parentheses.

RESULTS AND DISCUSSION

The penetration is discussed as two separate phenomena: (1) the pore fluid displacement and (2) the fluid flow through the pores.

Displacement pressure

Effect of the surface tension

According to eq. (1), if a fabric is tested with different solutions under the same conditions, the penetration pressures should be proportional to the surface tensions of the solutions and cos θ:

$$\frac{P_1}{P_2} = \frac{\gamma_1 \cos \theta_1}{\gamma_2 \cos \theta_2} \tag{3}$$

The effect of the contact angle is discussed in the following section, so for practical purposes let us assume that the contact angle is the same for all fabric/solution combinations. Comparing the penetration pressures for sample B, we find that the water penetration pressure is approximately 1.7 times the penetration pressure of solution I and 2.75 times that of solution II (Table IV). For the surface tensions, we obtained corresponding ratios of 1.8 and 2.15, respectively (Table II). The same comparisons were made for the other fabric samples, and similar ratios were obtained, except for sample A subjected to solution II (ratio = 5.1). We suspect that a possible attraction between the fabric surface and polymer solution could be the reason for the lower than expected penetration pressure value (also see the contact angle in Table III).

The experimental results were generally in agreement with eq. (1); solutions with lower surface tensions had lower penetration pressures. Water had the highest penetration pressure for all samples, and solution II had the lowest (Figs. 3 and 4).

Aliquots of the displacement liquids were obtained after the penetration tests, and the surface tension, density, and viscosity measurements were repeated. The penetration test had little or no effect on these parameters. Samples D and E exhibited hydrophilic behavior in contact with solutions I and II, even though they exhibited hydrophobic behavior with water. This could be due to either low liquid surface tension or an interaction between the surfactant and the polymer because the contact angles were lower than 90° (Table III).

Effect of the contact angle

The surface tension affects the pressure at which a pore will fill, but it does not give any information about the wettability of the solution. However, by considering θ, we can decide whether the solution will wet the fabric or not; that is, for $\theta > 90°$, the solution is nonwetting for that particular fabric, and for $\theta < 90°$, it will wet the fabric. For water (Table IV), all the samples had a displacement pressure greater than 0, and this means that all the samples were hydrophobic. However, for water (Table III), sample D had $\theta = 89°$ with water. This contradicts our displacement pressure value (Table IV). As mentioned earlier, the contact angles shown in Table III were taken at the fabric surface, and they were consequently macroscopic contact angles. What we really need to know is the angle in the pores, that is, the microscopic contact angle. However, it is impossible to measure the microscopic contact angle. The surface properties of the materials have a direct effect on the contact angles. Fuzzy yarns, containing randomly arranged fibers, are usually nonwetting because of high water contact angles.[7] Our fabric samples were formed by the random alignment of fibers because they were nonwoven. It is, therefore, reasonable to expect greater contact angles inside the fabric than at the surface of the fabric, and consequently greater contact-angle values than those

TABLE IV
Penetration Pressures of Solutions

Material	Water (kPa)	I (kPa)	II (kPa)	III (kPa)	IV (kPa)
A	12.7 (0.14)	6.49 (0.27)	2.50 (0.49)	13.93 (1.03)	13.43 (0.12)
B	7.18 (0.5)	4.24 (0.51)	2.61 (0.64)	8.26 (0.71)	8.2 (1.06)
C	2.90 (0.16)	1.58 (0.25)	1.32 (0.24)	2.98 (0.4)	3.59 (0.24)
D	3.11 (0.1)	0	0	2.8 (0.3)	4.78 (0.18)
E	2.95 (0.26)	0	0	1.5 (0.4)	3.73 (0.42)

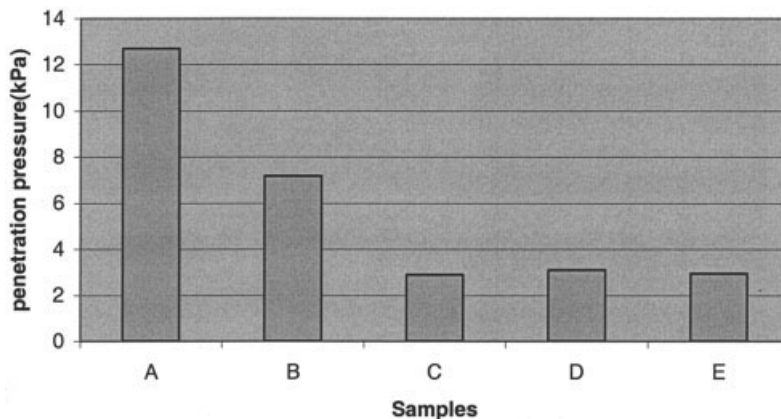Standard deviation values are in parentheses.

Figure 3 Penetration pressures of fabric samples with deionized water.

given in Table III. However, the differences were expected to be small because the polymer and the solution were the same. The difference was only due to the physical alignment of the fibers inside.

Solutions I and II exhibited nonwetting characteristics for samples A–C but not for samples D and E (Tables III and IV), which had measured contact angles of 0°. This maximum wettability behavior was probably due to an attraction between the polymer and the solution. Even though the contact angles (Table III) between samples A and B and solution II were lower than 90°, a force was still required to make the liquid penetrate the fabric (Table IV). This can be explained with the assumption of greater contact angles inside the pores because the surface contact angles did not differ too much from 90°.

Flow rates

Effect of the viscosity

By keeping the surface tension of water and solutions III and IV about the same, we were able to compare the effect of viscosity only. The solution properties are

given in Table II. The penetration pressures of the solutions were determined (Table IV), and in comparison with water, no significant viscosity effect was observed on penetration pressures.

According to Poiseuille's equation, flow rates through the same fabric are inversely proportional to the solution viscosity when the other conditions remain the same. Assuming two solutions, X and Y, with viscosities η_X and η_Y, respectively, we obtain for the same fabric from eq. (2)

$$\frac{Q_x}{Q_y} = \frac{\eta_y}{\eta_x} \tag{4}$$

Consequently, water was expected to have the highest flow rate, followed by the 1.5% PVA (III) solution and the 3% PVA (IV) solution at equal pressure differences. However, the expected order in the flow rates did not always occur (Figs. 5–9). In each figure, only the initial air drainage curves are shown. For sample A (Fig. 5), little difference existed between solution III and water, but the relative position of the curve for solution IV was as expected. For sample B (Fig. 6), the
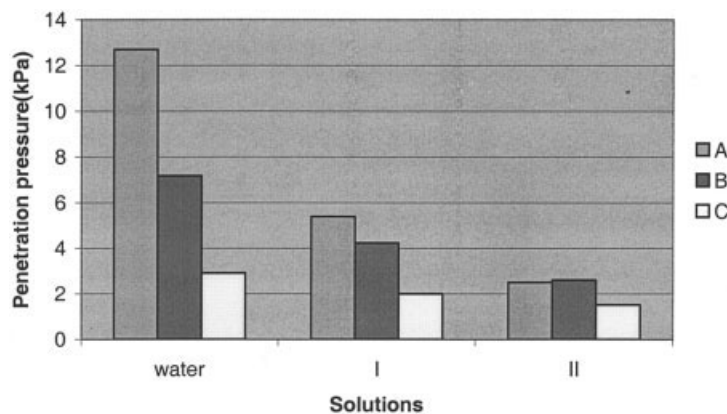

Figure 4 Penetration pressures of fabric samples A, B, and C with deionized water, solution I, and solution II.
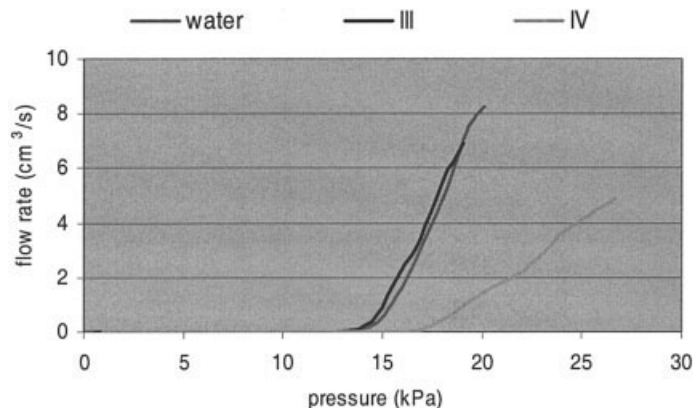
Figure 5 Flow properties of sample A.

relative positions for the curves of solutions III and IV were as expected, but for water the data showed the lowest rather than the highest flow rates. For sample C (Fig. 7), the expected order agrees with eq. (4) at higher pressures, but at lower pressures water and solution III exhibited similar flow rates. This discrepancy with eq. (4) was also observed for sample D (Fig. 8). The only sample that was completely consistent with eq. (4) was sample E (Fig. 9), not only orderwise but also ratiowise; solution IV had the lowest flow rates and water had the highest flow rates. The samples with the biggest pore sizes, samples C and E, exhibited flow rates that were in agreement with eq. (4).

Possible reasons for the differences between the experimental and theoretical results are

- The fabric samples were all very thin, and this may have obscured the effect of viscosity, as expected.
- As the viscosity increased, there was more viscous shear between the liquid and solid and more intermolecular drag between liquid molecules.

This may have caused greater pore size enlargements with solutions III and IV than with water during the initial contact. Fabrics A, B, and D had somewhat smaller pore sizes than fabrics C and E and showed greater deviations from the behavior expected from eq. (4).

In theory, porous media are assumed to be rigid. The fabric structure is not completely rigid, however, as the fibers can be moved by transporting fluids. Consequently, pore sizes may change; the higher the viscosity is, the greater the expected effect is. Although the flow rate should decrease with increasing viscosity at the same pressure gradient, it should increase with increasing pore size. If the pores are very small, the effect of increasing viscosity will be effective; if the pores are already relatively large, the effect of increasing pore size will be dominant on the flow rates.

Effect of the surface tension

In the previous section, we discussed the effect of viscosity on the flow rate, and we concluded that if
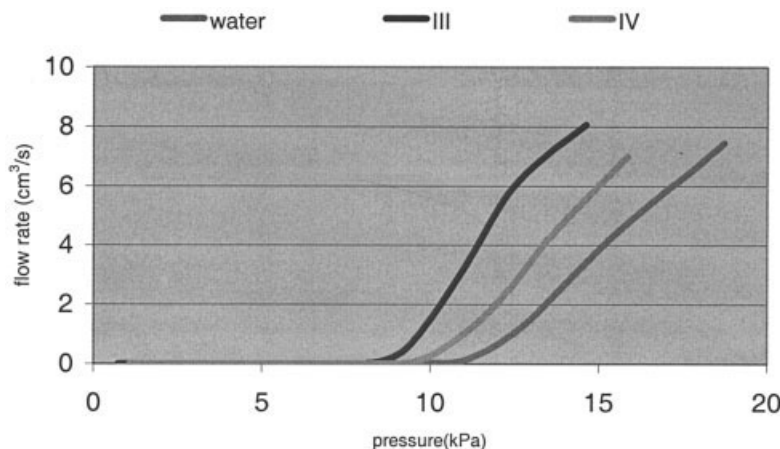

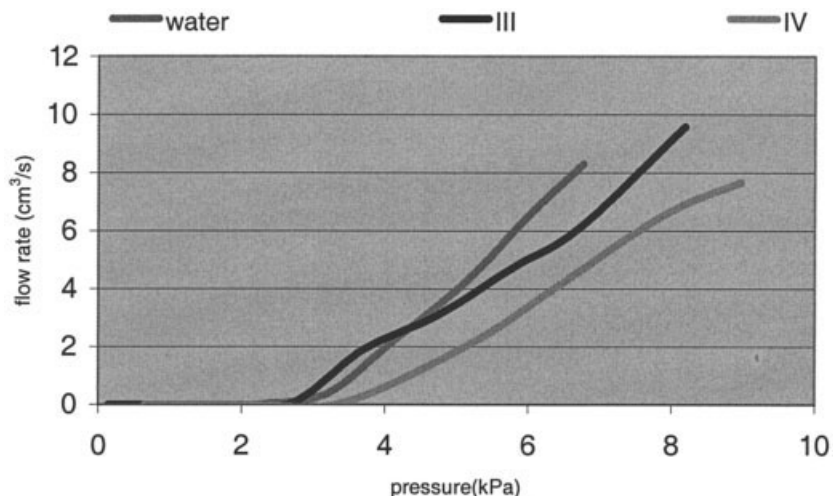**Figure 6** Flow properties of sample B.

Figure 7 Flow properties of sample C.

two solutions have the same surface tension, their flow rates are inversely proportional to their viscosity [eq. (4)]. However, when their surface tensions are different, this statement is not necessarily true. For example, the data presented in Figure 10 show that water had lower flow rates than solutions I and II, although they had similar viscosity values (Table II). Because of its lower surface tension, solution I had a lower displacement pressure (6 kPa) than water (12 kPa). Consequently, as the pressure increased and reached 12 kPa, solution I filled almost all the pores, but water had just started to fill the biggest pores. The same comparison can be made with solution II; it had the lowest surface tension, and the flow started even at lower pressures. The flow rates through samples A–C are shown in Figures 10–12, respectively. The order of flow rates was as expected for samples A–C. Water had the lowest flow rates, whereas solution II had the

highest flow rates. Samples D and E exhibited hydrophilic behavior with solutions I and II.

Hysteresis was observed between the flow rates with increasing solution pressure and decreasing solution pressure (Figs. 10–12). It was attributed to (1) the ink-bottle effect, (2) the increase in the pore sizes with the initial application of the liquid, (3) the contact-angle effect, and (4) the presence of trapped liquid in the pores. The capillary pores in fabrics are composed of sections with different diameters. Consequently, each of these sections has a different displacement pressure, with the smallest sections requiring the greatest displacement pressure. For an entire sequence of sections with different diameters to be filled, the liquid pressure must exceed the smallest section's displacement pressure. When the liquid pressure is being reduced, the smallest pores will lose their water first, and they will be followed by increasingly larger pores.
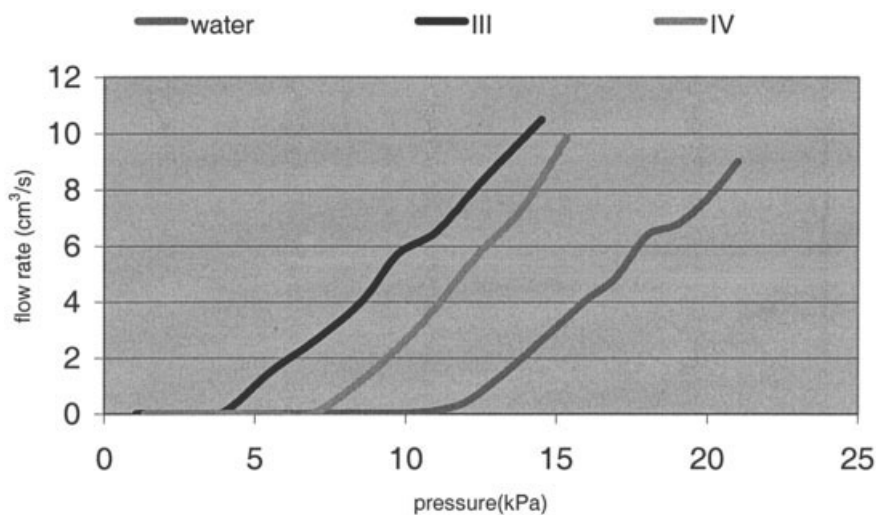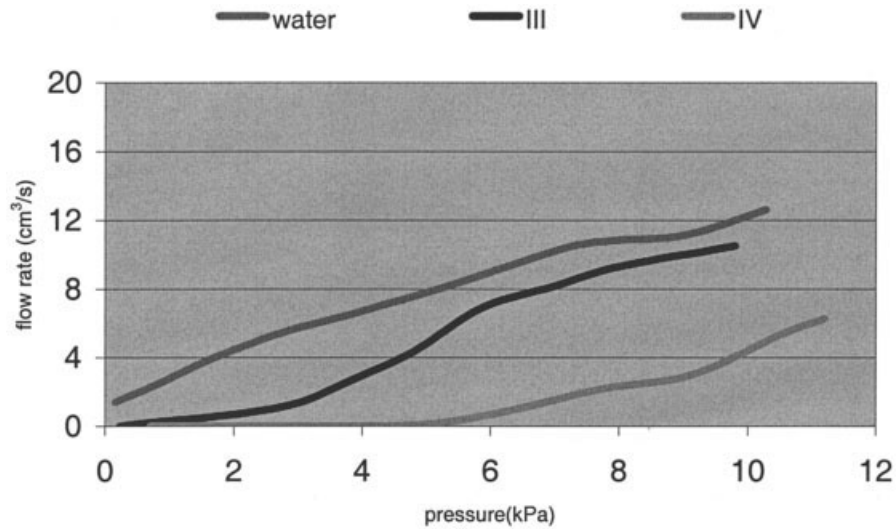

Figure 8 Flow properties of sample D.

Figure 9   Flow properties of sample E.

The drainage of water from a pore with different sections is, therefore, determined by the largest sections. As a result, pores with different sections will require greater pressures to fill with water than to drain and consequently exhibit hysteresis. This effect is called the ink-bottle effect.[15]

Although surface tension does not directly affect the flow rate, it determines at what pressure the flow will start. The viscosity effects described by Poiseuille's law are only applicable to situations in which the flow is not affected by the boundaries (inlet and outlet of capillaries).

CONCLUSIONS

The effects of the surface tension, viscosity, and liquid/material contact angle on the displacement pressure and flow through fabrics have been studied. Each of these parameters has been shown to be important:

- The liquid surface tension and liquid/material contact angle influence the pressure values at penetration. Liquids with lower surface tension and smaller contact angles fill the pores at lower pressure values. Lower surface tensions increase the wettability of the materials.
- Pores tend to be filled at lower pressures with lower surface tension liquids; consequently, greater flow rates occur at lower applied pressures than those for higher surface tension liquids.
- Liquids with higher viscosities have higher resistance to flow, and this results in lower flow rates.
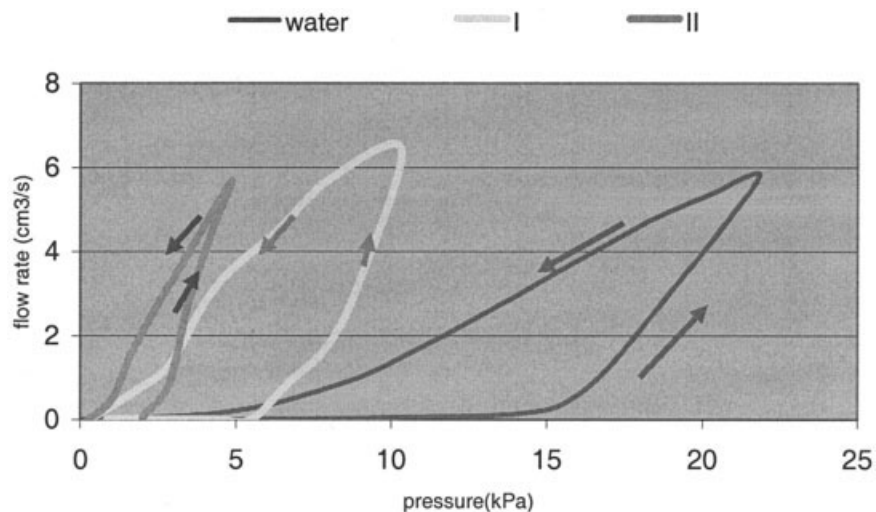

Figure 10   Flow rates of water, solution I, and solution II through fabric A.
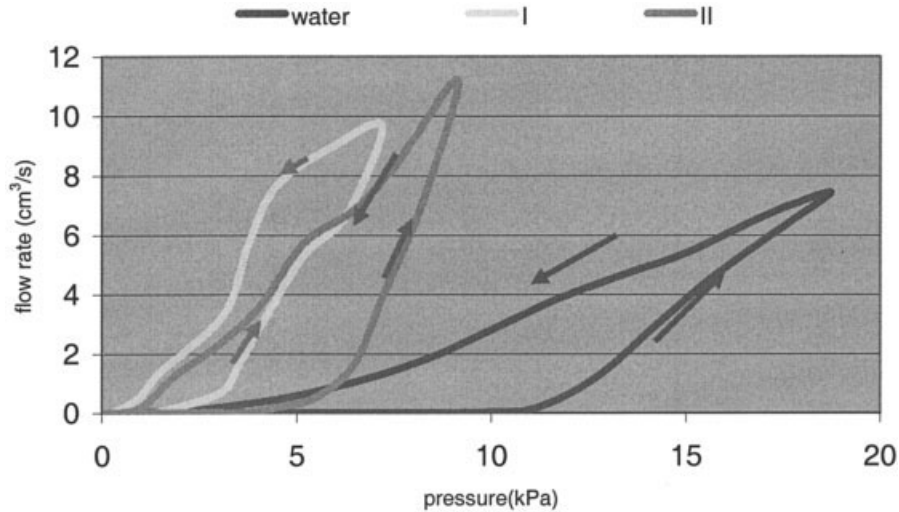
Figure 11 Flow rates of water, solution I, and solution II through fabric B.

- The liquid viscosity does not influence the penetration pressure.
- The liquid viscosity is important for determining the protection performance of fabrics. Even though high viscosities cause lower flow rates, they might cause enlargements in pore sizes. The enlarged pores will have lower penetration pressures and higher flow rates.
- The contact angles were measured at the fabric surface. However, the angles inside the capillaries might not be the same.

Our results indicate that the most important factors for the prediction of the penetration pressure are the surface tension and the largest pore size. However, the su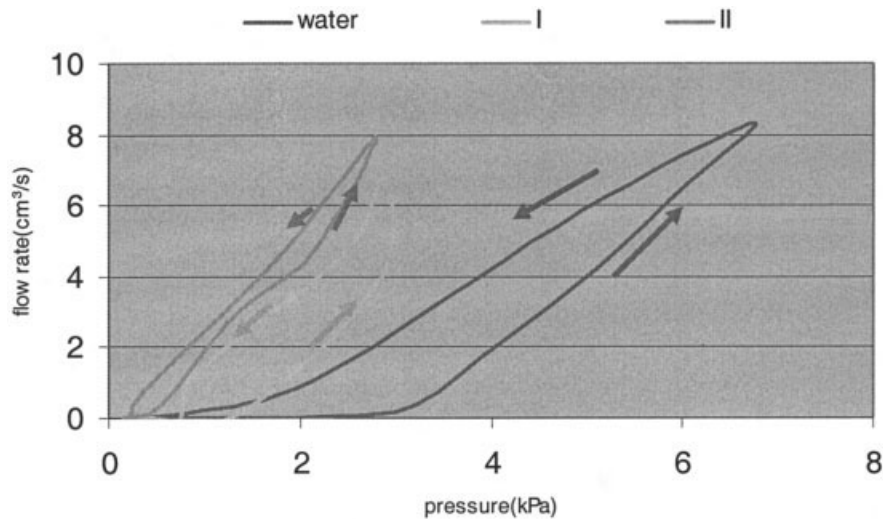rface tension itself does not tell the whole story about the wetting properties of the liquid. Although the contact angle is less significant, it indicates whether the liquid is wetting or nonwetting for the fabric. We also evaluated the protective performance of medical fabrics. Even though all samples were strong enough for most operational purposes, in some operations, such as open heart surgery and bypass surgery, the pressure of blood that spurts from a vessel can reach up to 413 kPa.[16] In such cases, we need protection barriers that can handle higher pressures. One way of accomplishing this is to reduce the pore sizes; however, we should also keep the comfort level of the protective garment in mind. As mostly the chest and arm areas are under exposure, outfits with smaller pores at these regions could be a way of providing the desired protection.

Figure 12 Flow rates of water, solution I, and solution II through fabric C.

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