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# Production of Polyethersulfone Hollow Fiber Ultrafiltration Membranes. II. Effects of Fiber Extrusion Pressure (EP) and PVP Concentration in the Spinning Solution

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## ABSTRACT

The dimensional and UF performance characteristics of hollow fiber membranes produced by the solution spinning technique using three polymer solutions (C3, C4, and C5) were studied experimentally. The polymer (polyethersulfone, PES)/solvent (1-methyl-2-pyrrolidone, NMP)/additive (polyvinyl pyrrolidone, PVP) concentration (wt%) used were 20/65/15, 20/60/20, and 25/63/12, respectively, for C3, C4, and C5 solutions, and their corresponding viscosities were 9222, 22,809, and 29,286 cP. The extrusion pressures (EP) used in fiber production were 5 to 15, 20 to 40, and 20 to 60 psig, respectively, for C3, C4, and C5 solutions; the internal coagulant water flow rate (WFR) used were 7.5 and 10 mL/min for C3 fibers, and 5 mL/min for C4 and C5 fibers; and the length of air gap (LAG) was held constant at 80 cm in the production of all the fibers. An increase in EP always tended to increase OD, while ID decreased, increased, or remained constant depending on the WFR used. An increase in PVP concentration in the fiber spinning solution contributed to greater fiber swelling effects. Nascent fiber velocity (NFV) tended to increase with an increase in EP, but it decreased considerably with an increase in PVP concentration in the fiber spinning solution and the consequent increase in solution viscosity. Both fiber dimensions and skin layer morphology were found to be governed by the combined effects of desolvation, fiber swelling, and fiber stretching during fiber production.

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## INTRODUCTION

This paper is in continuation of the earlier one (1) on the effects of water (internal coagulant) flow rate (WFR) and length of air gap (LAG) on the dimensional and UF performance characteristics of polyethersulfone (PES, Victrex 4800P) hollow fiber membranes produced by the solution spinning technique using two polyethersulfone (PES)–1-methyl-2-pyrrolidone (NMP)–polyvinyl pyrrolidone (PVP) polymer solutions. Following the solution structure–desolvation rate approach to the development of RO/UF membranes (2), the above fiber characteristics were discussed from the points of view of the physicochemical events of desolvation, fiber swelling, and fiber stretching taking place during fiber production. For a given polymer solution (i.e., constant solution structure), the experimental results obtained under different conditions of fiber production were found to be consistent with the qualitative cause–effect tendencies as shown in Tables 1 and 2, where an upward arrow ( ↑ ) indicates a tendency

TABLE 1
Effects of Desolvation, Fiber Swelling, and Fiber Stretching on Fiber Dimensions

| | OD | ID | Wall thickness |
|---|---|---|---|
| Desolvation | ↓ | ↓ | ↓ |
| Fiber swelling | ↑ | ↓ | ↑ |
| Fiber stretching | ↑ | ↑ | ↓ |

TABLE 2
Effects of Desolvation, Fiber Swelling, and Fiber Stretching on Skin Layer Morphology

| Skin layer morphology | Desolvation rate: WFR ↑ | Desolvation rate: WFR ↑↑ | Fiber swelling | Fiber stretching |
|---|---|---|---|---|
| Skin layer thickness | ↓ | ↑ | ↑ | ↓ |
| Average pore size | ↓ | ↑ | ↑, ↓, – | ↑ |
| Pore density | ↑ | ↓ | ↓ | ↓ |
| Pore size distribution | Narrower | Wider | Wider | Wider |

to increase, a downward arrow ( ↓ ) indicates a tendency to decrease, and a dash (−) indicates a tendency to remain unchanged.

In the work reported in this paper, the focus of attention is on the effects of fiber extrusion pressure (EP) and PVP concentration in the fiber spinning solution. Three additional fiber spinning solutions were used for fiber production. The materials, apparatus, and experimental details used for fiber production and testing were the same as those described earlier (1, 3). The experimental data on fiber characteristics are reported and discussed on the basis of the effects of desolvation, fiber swelling, and fiber stretching on fiber dimensions and skin layer morphology as stated above.

## RESULTS AND DISCUSSION

### Effects of Fiber Extrusion Pressure (EP) and PVP Concentration in the Spinning Solution

#### *Fiber Production Variables*

Table 3 gives the compositions of the five (C1 to C5) spinning solutions used in this work. Of these, solutions C1 to C4 had the same PES concentration (20 wt%) but different PVP and NMP concentrations. The experimental data on the characteristics of fibers obtained from C1 and C2 solutions have already been reported and discussed (1); a few of the above data are recalled in this paper for comparison and further discussion. From the point of view of polymer solution structure, in C1 to C4 solutions the PVP concentration increased from 5 to 20 wt% and the NMP concentration



TABLE 3
Fiber Spinning Solutions[a]

| Solution | wt% | | | NMP/PES | PVP/NMP | PVP/PES | PVP/(PES + PVP) | Viscosity (cP) |
|---|---|---|---|---|---|---|---|---|
| | PES | NMP | PVP | | | | | |
| C1 | 20 | 75 | 5 | 3.75 | 0.0667 | 0.25 | 0.20 | 2,112 |
| C2 | 20 | 70 | 10 | 3.50 | 0.1429 | 0.50 | 0.33 | 3,924 |
| C3 | 20 | 65 | 15 | 3.25 | 0.2308 | 0.75 | 0.43 | 9,222 |
| C4 | 20 | 60 | 20 | 3.00 | 0.3333 | 1.00 | 0.50 | 22,809 |
| C5 | 25 | 63 | 12 | 2.52 | 0.1905 | 0.48 | 0.32 | 29,286 |

[a] PES: Polyethersulfone (Victrex 4800 P) supplied by ICI. NMP: 1-Methyl 2-pyrrolidone supplied by Merck. PVP: Polyvinyl pyrrolidone, molecular weight 10,000, supplied by Sigma Chemical.

decreased from 75 to 60 wt%. Consequently, the NMP/PES ratio progressively decreased, and the PVP/NMP and PVP/PES ratios correspondingly increased in order from C1 to C4. On the basis of such composition changes, considering PVP as a nonsolvent for PES polymer, one would expect (2) that, in the absence of other effects, the supermolecular polymer aggregates in the above solutions will also be successively bigger in the sequence of C1 to C4, and the corresponding aggregate pores in the skin layer of the resulting fibers would also be successively bigger in the same sequence. On the other hand, since the PVP/(PES + PVP) ratio also increased successively from C1 to C4 solutions, on the basis of PES–PVP interactions discussed earlier (1, 4), one may conclude that the concentration of bound PVP in C1 to C4 solutions will also be successively higher, giving rise to more numerous smaller skin layer pores. Such a PES–PVP interaction effect may be expected to be the highest for C4 solution and progressively lower for C3, C2, and C1 solutions. At the same time, the concentration of the water leachable unbound PVP also will be highest for C4 solution and progressively lower for C3, C2, and C1 solutions.

Further, because of the higher PVP concentrations, the viscosities of C3 and C4 solutions were even higher (Table 3) than those of C1 and C2 solutions. The EPs used for the production of C3 fibers were in the 5 to 15 psig range and those used for the production of C4 fibers were in the 20 to 40 psig range. WFRs of 7.5 and 10 mL/min were used in the production of C3 fibers, and a WFR of 5 mL/min was used in the production of C4 fibers. The LAG was kept constant at 80 cm in the production of both C3 and C4 fibers.

In C5 spinning solution (Table 3), a higher viscosity was achieved by increasing the PES concentration to 25 wt%; to avoid too high a viscosity, PVP concentration was reduced to 12 wt%. Compared to C3 and C4 solutions, PVP/NMP, PVP/PES, and PVP/(PES + PVP) ratios were lower. In the production of C5 fibers, the EPs used were in the 20 to 60 psig range, and the WFR and LAG were held constant at 5 mL/min and 80 cm, respectively.

## Data on C3 and C4 Fibers

### *Fiber Dimensions*

Under the experimental conditions of EP, WFR, and LAG used with respect to C3 fibers, the data on the outside diameter (OD) were in the 1.10 to 1.27 mm range, those on the inside diameter (ID) were in the 0.65 to 0.69 mm range, and those on the fiber wall thickness were in the 0.21 to 0.29 mm range; with respect to C4 fibers the corresponding ranges were 1.52 to 1.86 mm for OD, 0.90 to 1.02 mm for ID, and 0.31 to 0.42 mm for wall thickness. These data show that the general effect of higher EP and higher PVP concentration in the spinning solution is to increase OD, ID, and wall thickness for the resulting fibers.

Figure 1 shows the effects of EP and WFR on fiber OD, ID, wall thickness, and OD/ID ratio for C3 and C4 fibers. The data show that, with respect to C3 fibers at constant WFR, when the extrusion pressure increased, OD tended to increase, ID tended to decrease or remain constant, and consequently, both fiber wall thickness and the OD/ID ratio increased; and an increase in WFR at constant EP tended to increase both OD and ID but to reduce wall thickness and the OD/ID ratio marginally. These results are similar to those reported earlier for C1 and C2 fibers (1). With respect to C4 fibers, where the range of EP used was higher, OD, ID, wall thickness, and OD/ID increased with an increase in EP at the WFR



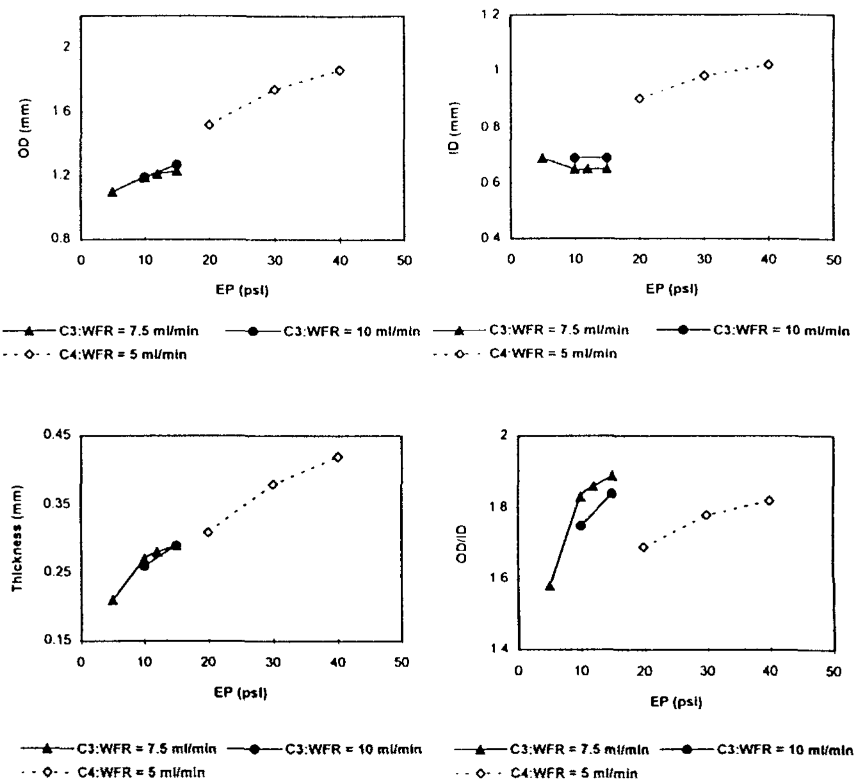


FIG. 1 Effects of EP and WFR on C3 and C4 fiber dimensions.

of 5 mL/min. These results indicate that an increase in EP always tended to increase OD and wall thickness, while ID may increase, decrease, or remain constant depending on the magnitude of WFR used for the C3 and C4 fibers. Fiber swelling (which tends to increase OD and wall thickness, and to decrease ID) and lateral fiber stretching (which tends to increase OD and ID, and to decrease wall thickness) can account for the observed effects of EP and WFR on OD, ID, and wall thickness for C3 and C4 fibers.

Figure 2 reviews the above experimental data on OD and wall thickness as functions of WFV and WFP. It is useful to consider these correlations along with the corresponding numerical data given in Table 4.

Fiber formation is always associated with desolvation which is governed primarily by WFV and WFP. Table 4 shows that in the EP range of 10



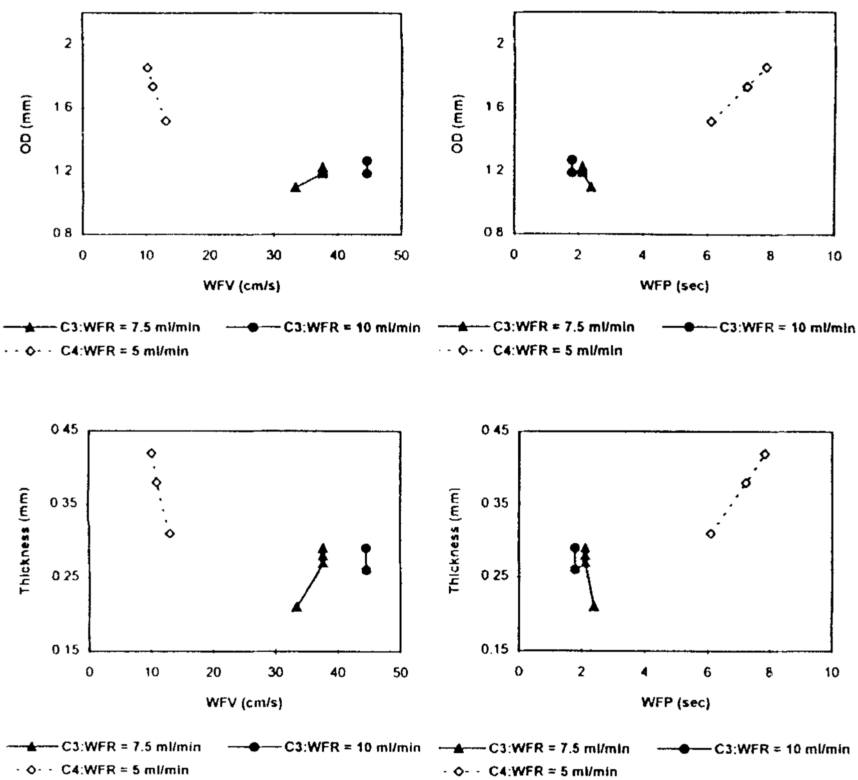


FIG. 2 Effects of WFR, WFV, and WFP on C3 and C4 fiber dimensions.

TABLE 4
Comparison of Spinning Solution, Fiber Production Conditions, and the Resulting Fiber Dimensions

| Solution | PVP (wt%) | EP (psig) | WFR (mL/min) | OD (mm) | ID (mm) | Wall thickness (mm) | WFV (cm/s) | WFP (s) | NFV (cm/s) |
|---|---|---|---|---|---|---|---|---|---|
| C3 | 15 | 5 | 7.5 | 1.10 | 0.69 | 0.21 | 33.4 | 2.39 | 14.4 |
| | | 10 | 7.5 | 1.19 | 0.65 | 0.27 | 37.7 | 2.12 | 17.9 |
| | | 12 | 7.5 | 1.21 | 0.65 | 0.28 | 37.7 | 2.12 | 18.6 |
| | | 15 | 7.5 | 1.23 | 0.65 | 0.29 | 37.7 | 2.12 | 19.2 |
| C3 | 15 | 10 | 10 | 1.19 | 0.69 | 0.26 | 44.6 | 1.79 | 18.1 |
| | | 15 | 10 | 1.27 | 0.69 | 0.29 | 44.6 | 1.79 | 20.8 |
| C4 | 20 | 20 | 5 | 1.52 | 0.90 | 0.31 | 13.1 | 6.11 | 3.94 |
| | | 30 | 5 | 1.74 | 0.98 | 0.38 | 11.0 | 7.24 | 5.24 |
| | | 40 | 5 | 1.86 | 1.02 | 0.42 | 10.2 | 7.84 | 5.93 |
| C1 | 5 | 5 | 7.5 | 1.03 | 0.65 | 0.19 | 37.7 | 2.12 | 22.6 |
| C3 | 15 | 5 | 7.5 | 1.10 | 0.69 | 0.21 | 33.4 | 2.39 | 14.4 |
| C2 | 10 | 15 | 7.5 | 1.25 | 0.87 | 0.19 | 21.0 | 3.80 | 15.3 |
| C3 | 15 | 15 | 7.5 | 1.23 | 0.65 | 0.29 | 37.7 | 2.12 | 19.2 |
| C5 | 12 | 20 | 5 | 1.46 | 0.81 | 0.33 | 16.2 | 4.95 | 2.44 |
| | | 30 | 5 | 1.48 | 0.83 | 0.33 | 15.4 | 5.19 | 2.97 |
| | | 40 | 5 | 1.52 | 0.86 | 0.33 | 14.3 | 5.58 | 4.69 |
| | | 50 | 5 | 1.58 | 0.92 | 0.33 | 12.5 | 6.38 | 4.68 |
| | | 60 | 5 | 1.62 | 0.96 | 0.33 | 11.5 | 6.95 | 5.08 |



to 15 psig, WFV remained constant at 37.7 cm/s and WFP also remained constant at 2.12 s—which means that the desolvation conditions during fiber formation remained constant for the C3 fibers produced. Consequently, the experimental data given in Table 4 show the effects of EP and WFR on OD and wall thickness at constant desolvation conditions. At the above values of WFV and WFP, with respect to C3 fibers, an increase in EP tended to increase OD and wall thickness at both the WFR conditions studied; these results can be accounted for on the basis of the fiber swelling effect. Furthermore, at constant EP with an increase in WFR, OD either remained constant or increased while wall thickness either decreased or remained essentially constant; these results can be accounted for on the basis of the combined effects of fiber swelling and fiber stretching. Thus, for C3 fibers, in addition to desolvation, fiber swelling and fiber stretching have significant effects during fiber formation and on the resulting fiber dimensions.

A comparison of the OD, ID, and wall thickness data for the two pairs of fibers, namely C1-7.5-80-5 and C3-7.5-80-5 as well as C2-7.5-80-15 and C3-7.5-80-15, shows the combined effects of an increase in PVP concentration in the fiber spinning solution and an increase in EP. Referring to Table 4, an increase in PVP concentration from 5 to 15 wt% tended to increase OD, ID, and wall thickness at the EP of 5 psig, as a result of which WFV decreased and WFP increased. These results indicate that the effects of fiber swelling and fiber stretching are more dominant than the effect of desolvation under the above experimental conditions. On the other hand, an increase in PVP concentration from 10 to 15 wt% tended to decrease OD and ID, with a considerable increase in wall thickness at the EP of 15 psig. These results indicate that both desolvation and fiber swelling have significant effects on the resulting fiber dimensions at the higher EP.

With respect to C4 fibers, Fig. 2 shows that OD and wall thickness decreased with an increase in WFV, but they increased with an increase in WFP. Referring to Table 4, the data show that WFV decreased and WFP increased with an increase in EP. Consequently, the increases in OD and wall thickness with a decrease in WFV and an increase in WFP are essentially the effect of an increase in EP, or the fiber swelling effect, which is particularly significant for C4 fibers in view of their higher bound PVP content.

### *Nascent Fiber Velocity*

The NFV values for C3 fibers were in the 14.4 to 20.8 cm/s range and those for C4 fibers were in the 3.9 to 5.9 cm/s range. Figure 3 shows that NFV tended to increase with an increase in EP and an increase in WFR, but decreased considerably with an increase in PVP concentration in the fiber spinning solution and its consequent high viscosity. Figure 3 also shows that, with respect to C3 fibers, at constant WFV or constant WFP, NFV tended to increase with an increase in EP. With respect to C4 fibers, NFV tended to decrease with an increase in WFV and to increase with an increase in WFP. Since increase in EP resulted in a decrease in WFV and a increase in WFP, the above correlations of NFV with WFV and WFP are only expressions of the effect of increase in EP.

Figure 4 illustrates the effect of NFV on OD, ID, wall thickness, and the OD/ID ratio. With respect to C3 fibers, OD, wall thickness, and the OD/ID ratio increased and ID tended to decrease or remain constant with an increase in NFV. With respect to C4 fibers, OD, ID, wall thickness, and the OD/ID ratio increased with an increase in NFV. These correlations indicate the dominant effects of fiber swelling and fiber stretching (the EP

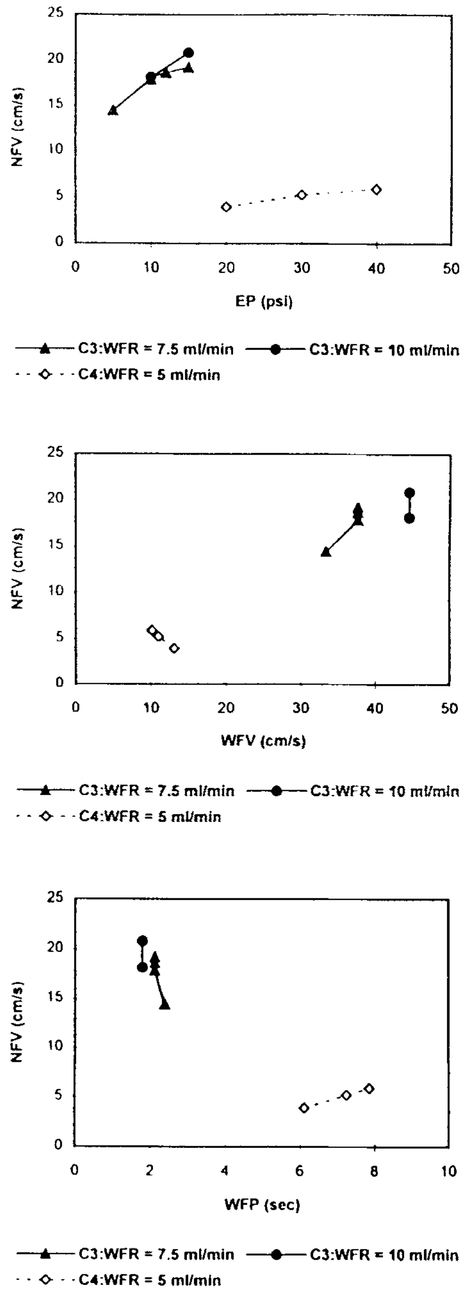


FIG. 3 Effects of EP, WFV, and WFP on NFV for C3 and C4 fibers.

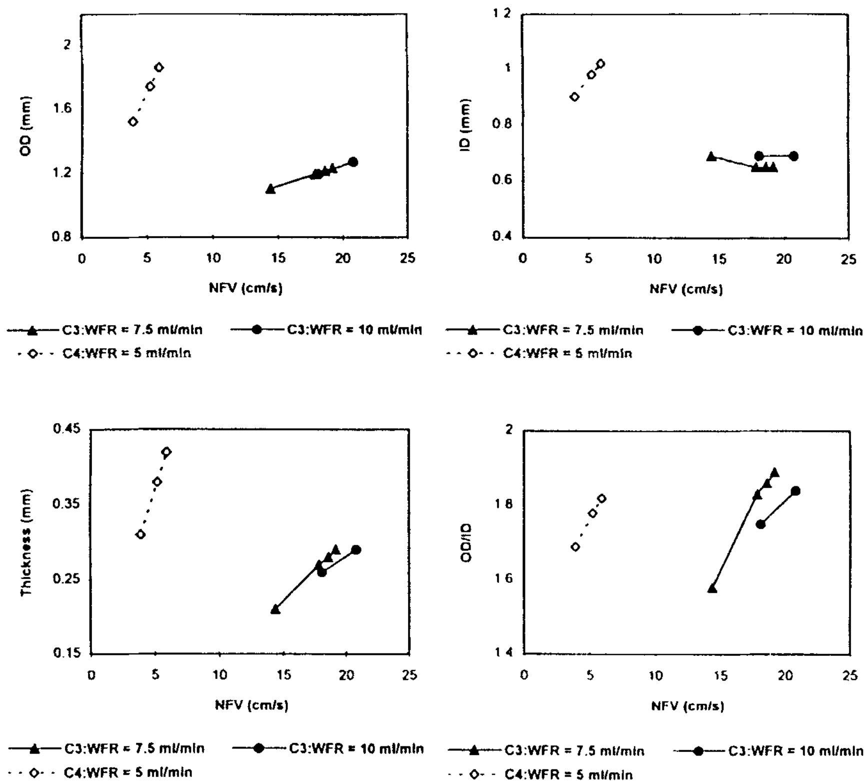


FIG. 4 Effects of NFV and WFR on C3 and C4 fiber dimensions.



effect) on C3 and C4 fiber dimensions. It may be noted that the correlations shown in Fig. 4 are in contrast with those shown earlier (1) for C1 and C2 fibers where the dominant effect was desolvation.

## Spinnerette Design and Fiber Dimensions

Figure 5 illustrates the effect of one aspect of spinnerette design ($OD_{FIX}$ and $ID_{FIX}$ shown in Fig. 2 in Ref. 1) on the resulting fiber dimensions. Since the values of $OD_{FIX}$ and $ID_{FIX}$ are constants fixed by the spinnerette design, the correlation of $OD/OD_{FIX}$ and $ID/ID_{FIX}$ as functions of NFV, EP, WFV, and WFP are similar to the corresponding correlations given in Figs. 1, 2, and 4, which have already been discussed. The objective of Fig. 5 is to show again that in all cases studied, the values of $OD/OD_{FIX}$

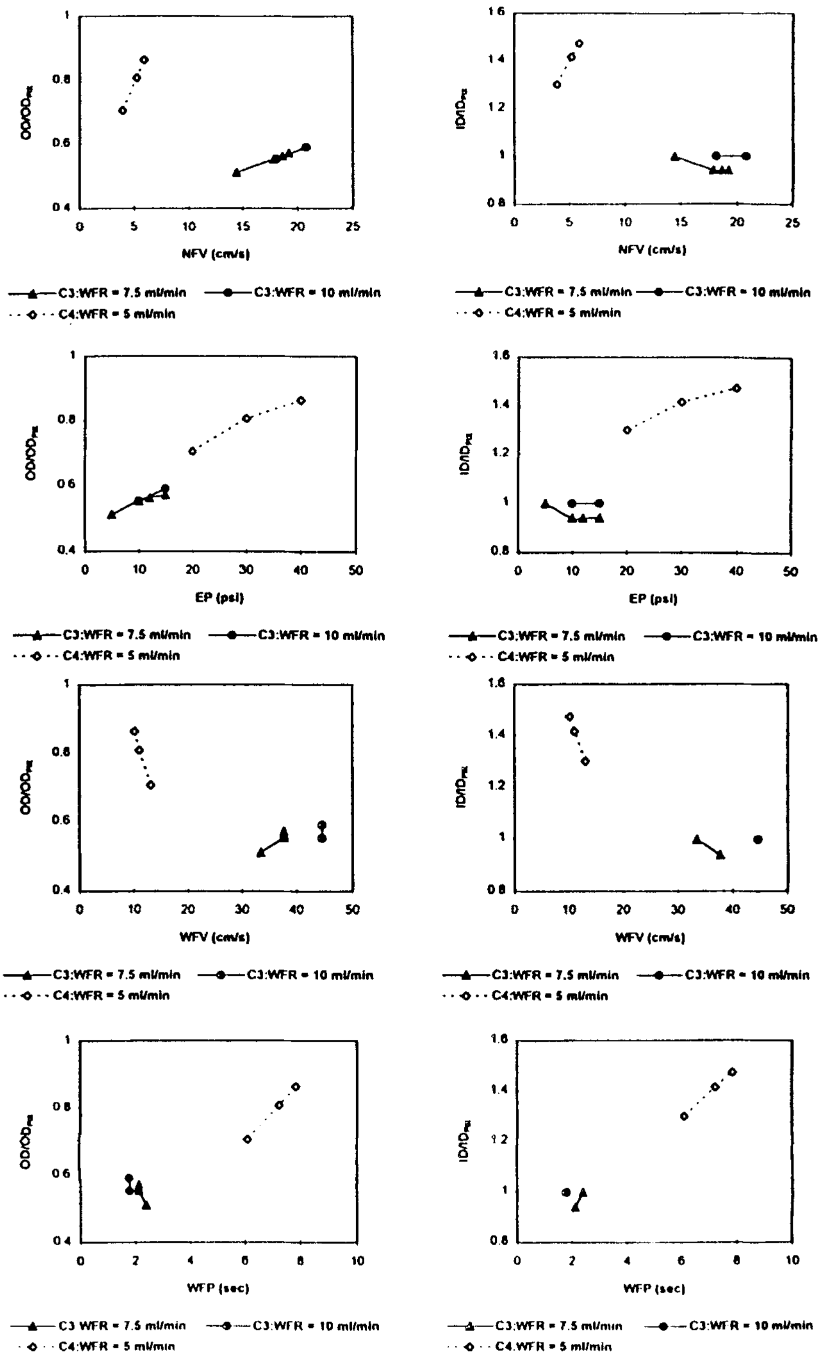


FIG. 5 Effects of NFV, EP, WFV, and WFP on $OD/OD_{FIX}$ and $ID/ID_{FIX}$ for C3 and C4 fibers.

are always less than unity, while those of $ID/ID_{FIX}$ can be higher than, equal to, or less than unity, depending on the fiber production conditions.

### *Fiber Performance in UF Experiments*

Figure 6 shows that with respect to C3 fibers, at a WFR of 7.5 mL/min, an increase in fiber EP decreased PEG separation, steeply in the EP range of 5 to 10 psig and less steeply in the EP range of 10 to 15 psig, and the corresponding PR tended to increase in the EP range of 5 to 15 psig. These data indicate that an increase in fiber EP tended to increase the average size of skin layer pores, which confirms the effects of fiber swelling and fiber stretching on the size of skin layer pores (1).

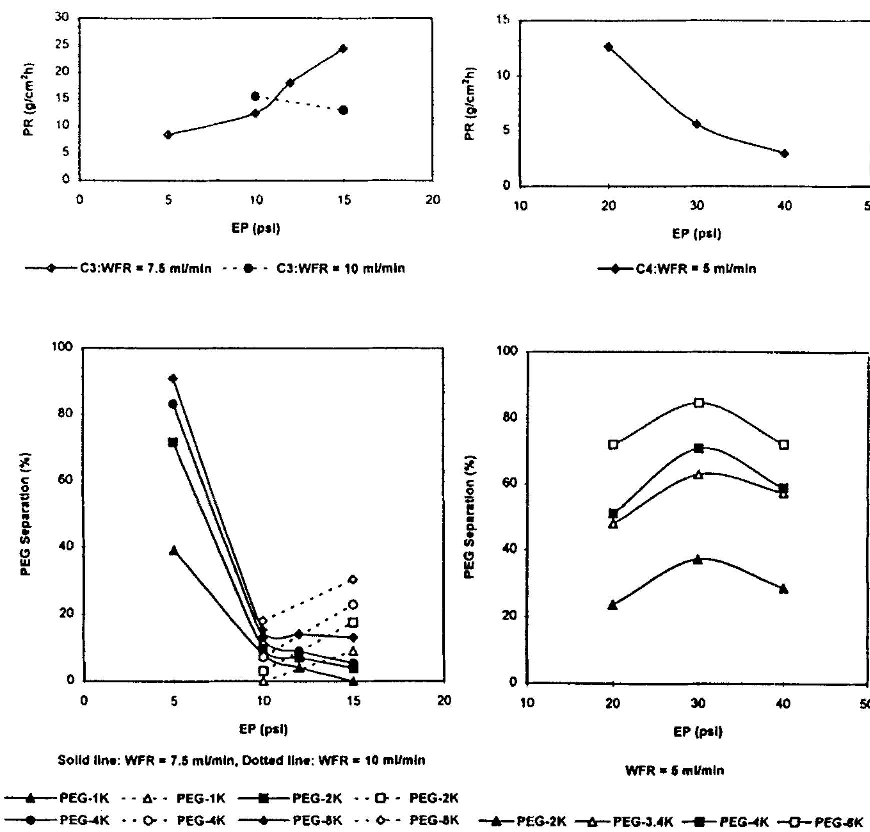


FIG. 6 Effects of EP and WFR on UF performance of C3 and C4 fibers.

The relatively smaller size of skin layer pores obtained at a fiber EP of 5 psig is attributable to the prevailing desolvation conditions of WFV and WFP. Table 4 shows that in the fiber EP range of 10 to 15 psig, the WFV and WFP remained constant. Under the latter fiber production conditions, the average size of skin layer pores was such that PEG separation in UF decreased still further, but less steeply. These data indicate that in the EP range of 5 to 15 psig, the fiber swelling effect became more dominant, and in the EP range of 10 to 15 psig, such fiber swelling not only increased the size of the prevailing skin layer pores, but also tended to open up some new smaller size pores on the skin layer of the fiber, which made the magnitude of the decrease in PEG separations in UF less steep. The latter possibility is confirmed by the PEG separation data of the fibers produced at EPs of 10 and 15 psig with a WFR of 10 mL/min. PEG separations were relatively lower for the fibers produced at an EP of 10 psig with a WFR of 10 mL/min (compared to those of fibers produced at an EP of 10 psig with a WFR of 7.5 mL/min), but the PEG separations increased for the fibers produced at an EP of 15 psig with a WFR of 10 mL/min; the corresponding PR decreased with an increase in PEG separations.

The significant conclusion arising from the data on C3 fibers in Fig. 6 is that fiber swelling not only increase the size of the prevailing skin layer pores, but it also opens up new smaller size pores; consequently, the average size of the resulting skin layer pores can increase, decrease, or remain unchanged.

The foregoing conclusion is supported by the data on C4 fibers given in Fig. 6 (right), which shows that PEG separations passed through a maximum with an increase in EP in the 20 to 40 psig range, and the maximum in solute separation (smallest average pore size on the skin layer) was obtained with fibers made at an EP of 30 psig; PR decreased for C4 fibers in the EP range of 20 to 40 psig. The above results indicate the simultaneous effects of pore generation and pore depletion due to the fiber swelling effect, which is significantly more for C4 than for C3 fibers because of the higher concentration of bound PVP in the fiber spinning solution.

The fiber performance data given in Fig. 6 are reviewed in Figs. 7 and 8 in terms of the effects of WFV and WFP during fiber production on the UF performance of the resulting fibers.

The effects of WFV and WFP during fiber production on the skin layer morphology of the resulting fibers may be considered to correspond to those of the solvent evaporation rate and the solvent evaporation period, respectively, in the process of formation of the original flat sheet cellulose acetate reverse osmosis membranes by the phase inversion process. As already pointed out in the literature (2), both solvent evaporation rate and

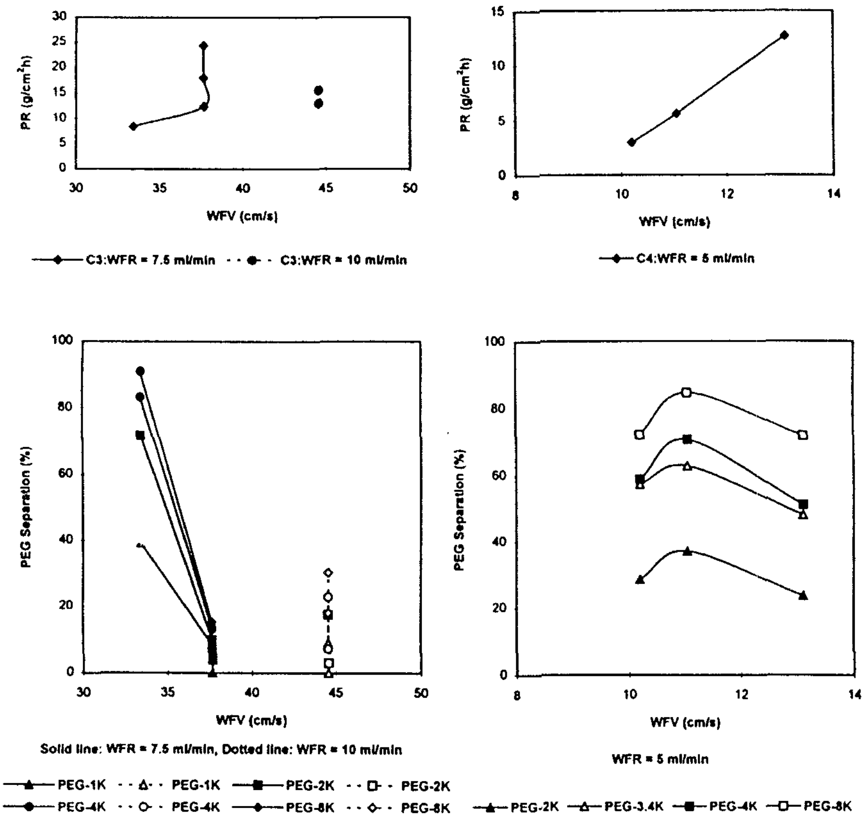


FIG. 7 Effects of WFV and WFR on UF performance of C3 and C4 fibers.

solvent evaporation period during film formation have significant effects on the creation of both polymer network pores (smaller size pores) and polymer aggregate pores (bigger size pores) and their respective distributions on the skin layer of the resulting membranes. There is an optimum condition of solvent evaporation rate and solvent evaporation period for maximum productivity of the resulting membranes. Accordingly, the effects of WFV and WFP during fiber production on the skin layer morphology of the resulting fiber may be expected to be similar.

Referring to the performance data of C3 fibers illustrated in Figs. 7 (left) and 8 (left), with an EP of 5 psig and with a WFR of 7.5 mL/min, the WFV and WFP values were 33.4 cm/s and 2.39 s, respectively, which resulted in C3 fibers giving solute separations of 39 and 91%, respectively,

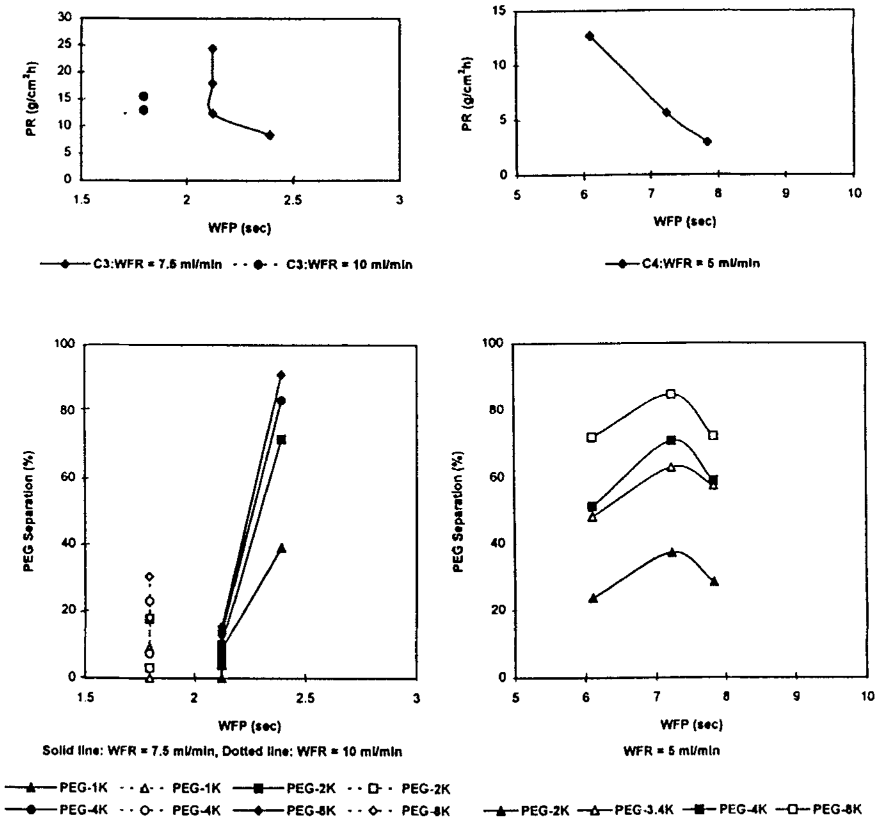


FIG. 8 Effects of WFP and WFR on UF performance of C3 and C4 fibers.

for PEG-1K and PEG-8K. When EP was increased to 10 psig, the WFV and WFP values for the resulting fibers changed to 37.7 cm/s and 2.12 s, respectively. Even though the latter changes were more conducive to the formation of smaller size skin layer pores, they yielded fibers which gave only 8 and 15%, respectively, for PEG-1K and PEG-8K solute separations, indicating that the average skin layer pore size for the latter fibers was larger. This means that with an increase in EP to 10 psig, the fiber stretching effect became more dominant than the desolvation and fiber swelling effects on skin layer morphology. In the EP range of 10 to 15 psig, the WFV and WFP values remained constant, indicating constant desolvation conditions. Under the latter conditions, PEG separations continued to decrease and PR continued to increase, indicating that in the EP range of

10 to 15 psig the fiber stretching effect was the predominant one governing skin layer morphology.

In the EP range of 10 to 15 psig, together with a WFR of 10 mL/min, WFV and WFP changed to 44.6 cm/s and 1.79 s, respectively. Even though the latter conditions were more conducive to the formation of smaller size skin layer pores, the UF data for the resulting fibers showed that the PEG separations were even lower for the fibers produced at an EP of 10 psig; however, the PEG separations tended to increase for fibers produced at 15 psig together with a WFR of 10 mL/min. These results indicate that at the latter conditions of fiber production, desolvation, fiber swelling, and fiber stretching have comparable effects on the resulting skin layer morphology.

The UF performance data of C4 fibers illustrated in Figs. 7 (right) and 8 (right) show that WFV values were considerably lower and WFP values were considerably higher for C4 fibers compared to the corresponding values for C3 fibers (Table 4) discussed above. These variations are the results of higher PVP concentration, and the consequent higher viscosity, of the C4 fiber spinning solution. Further, there was an optimum combination of WFV and WFP which gave rise to the smallest size of skin layer pores (i.e., maximum solute separations) for C4 fibers. This result, which reflects the desolvation effect on skin layer morphology, is complementary to the fiber swelling effect which was particularly significant in the production of C4 fibers.

Figure 9 illustrates the effect of NFV on the UF performance of C3 and C4 fibers. Table 4 shows that NFV increased with an increase in EP for both C3 and C4 fibers, and that there is a direct correlation between NFV and EP. Naturally, therefore, the correlations shown in Fig. 9 are similar to those shown in Fig. 6 which shows the effect of EP on fiber performance. Thus the effect of NFV on UF performance of C3 and C4 fibers can be understood on the same basis as those of EP on such performances discussed earlier.

## Data on C5 Fibers

As shown in Table 4, compared to C1 to C4 fiber spinning solutions, the C5 solution had a higher PES concentration, and hence a higher viscosity, but its PVP content and PVP/(PES + PVP) ratio were closer to those of the C2 solution. Further, in the production of C5 fibers, the EP used was in the 20 to 60 psig range together with a WFR of 5 mL/min and a LAG of 80 cm; these conditions are closer to those used in the production of C4 fibers. Therefore, some comparison on the characteristics of C5 fibers with those of C2 and C4 fibers is possible.

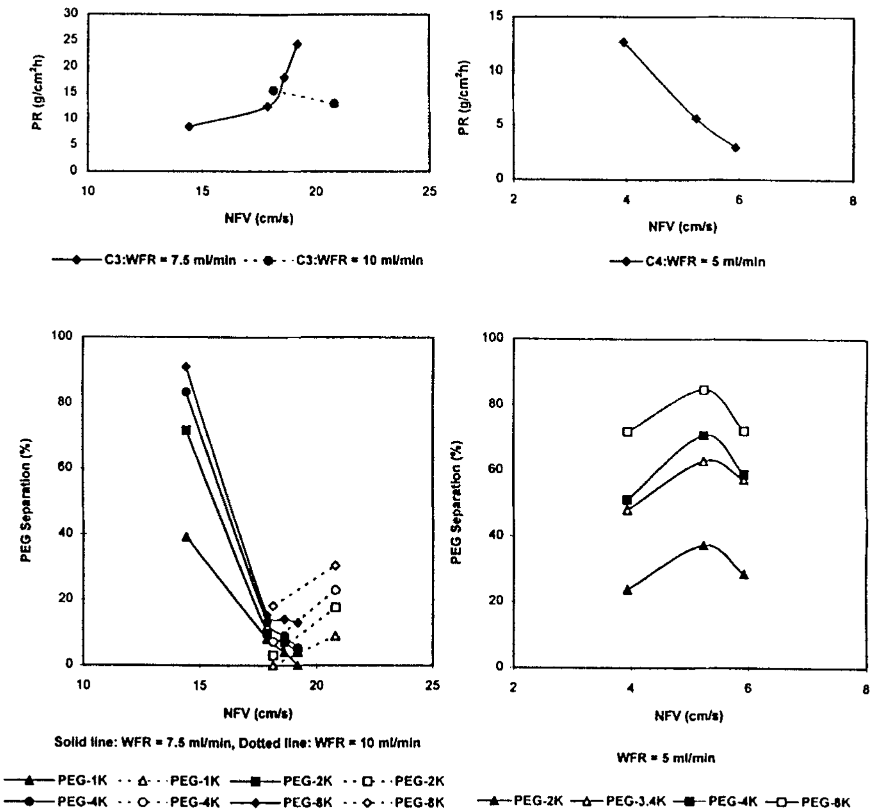


FIG. 9 Effects of NFV and WFR on UF performance of C3 and C4 fibers.

The data on C5 fibers obtained under the experimental conditions of EP, WFR, and LAG used in their production are given in Table 4 and Figs. 10 to 12. They illustrate the combined effects of desolvation, fiber swelling, and fiber stretching on fiber dimensions and skin layer morphology.

Compared to C2 fibers, C5 fibers had higher values for OD, ID, and the wall thickness, which can be attributed to the higher EP used in the production of C5 fibers, the higher PVP concentration used in the C5 spinning solution, and consequently the higher fiber stretching and fiber swelling effects on fiber dimensions. On the other hand, compared to C4 fibers, C5 fibers had lower values for OD, ID, and the wall thickness, which can be attributed to the relatively lower PVP concentration and higher NMP concentration in the C5 spinning solution, and consequently the greater desolvation effect on the dimensions of the resulting C5 fibers.

Figure 10 shows that for C5 fibers the OD and ID increased with an increase in EP in the 20 to 60 psig range, while those of fiber wall thickness remained unchanged. The OD values increased by almost 11% (1.46 to 1.62 mm), those of ID increased by about 19% (0.81 to 0.96 mm), and the wall thickness remained constant at 0.33 mm (Table 4). These data illustrate that the increase in OD due to the combined effects of fiber swelling and fiber stretching (EP effect) is partially compensated by the decrease in OD due to the desolvation effect, the increase in ID due to the fiber stretching effect of EP is more than the decrease in ID due to the desolvation and fiber swelling effects, and the decrease in wall thickness due to the desolvation and fiber stretching effects is exactly compensated by the



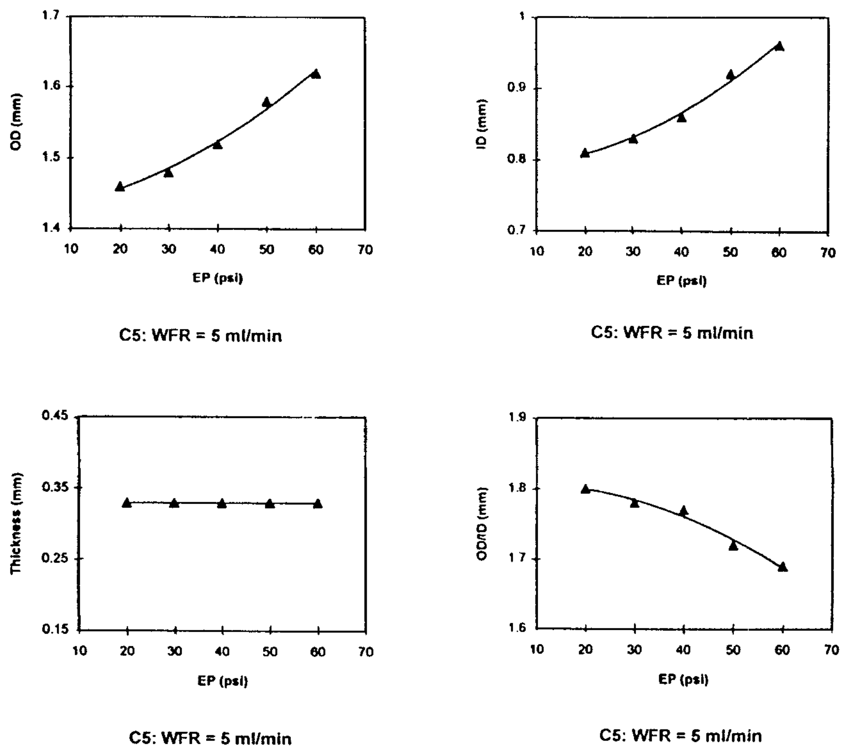


FIG. 10 Effects of EP and WFR on C5 fiber dimensions.

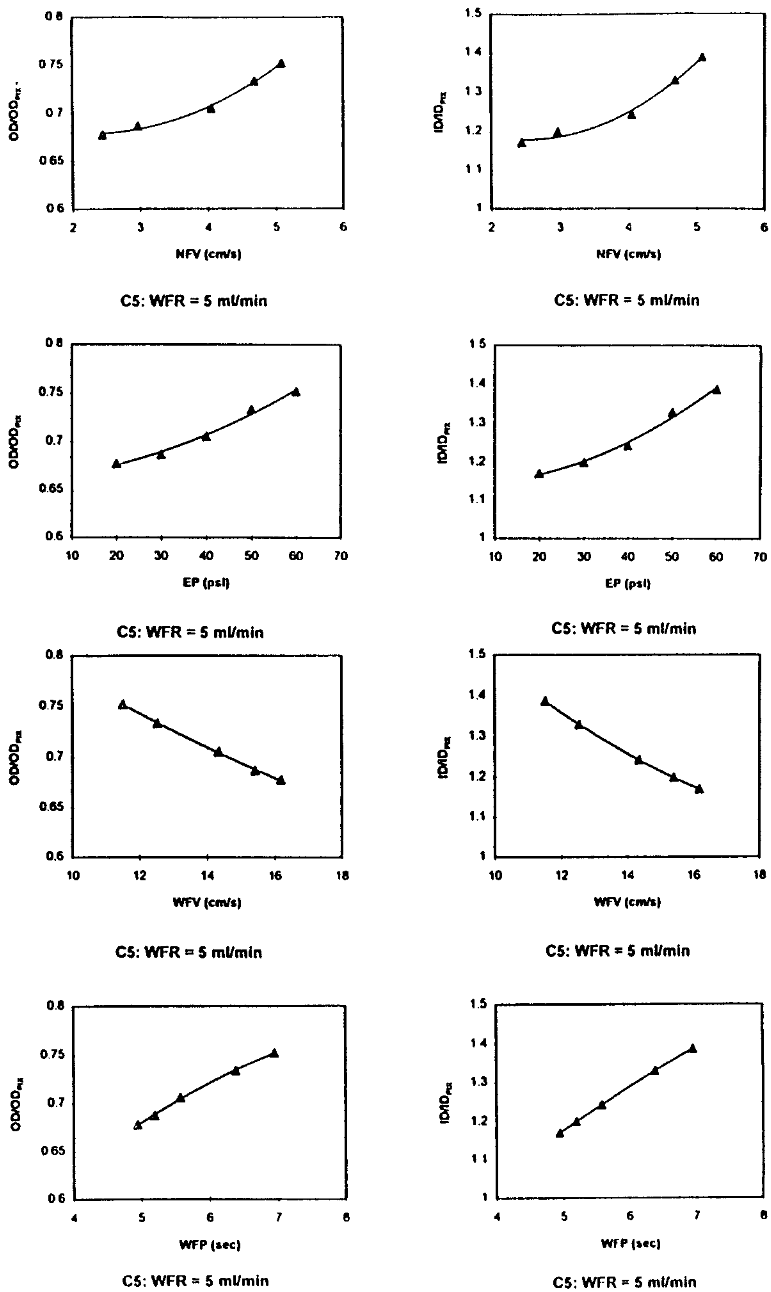


FIG. 11 Effects of NFV, EP, WFV, and WFP on OD/OD$_{FIX}$ and ID/ID$_{FIX}$ for C5 fibers.

increase in wall thickness due to the fiber swelling effect. The observed 6% decrease in the OD/ID ratio (1.80 to 1.69) in the EP range of 20 to 60 psig (Fig. 10) is simply the consequence of the relative changes in the respective OD and ID values.

It may be noted that the effects of EP on OD and ID were similar, but of different magnitude, compared to those for C4 fibers. In the EP range of 20 to 40 psig, OD and ID increased by 22 and 13%, respectively, for C4 fibers, whereas the corresponding increases were only 4 and 6%, respectively, for C5 fibers (Table 4). These values confirm the relatively greater desolvation effect on fiber dimensions with respect to C5 fibers.

Table 4 shows that WFV decreased for C5 fibers, and the corresponding WFP, and also NFV, increased with an increase in the EP used; these variations are similar to those obtained for C4 fibers. Further, Fig. 11 shows that the values of $OD/OD_{FIX}$ were less than unity, and those of $ID/ID_{FIX}$ were greater than unity for all C5 fibers. These results are similar to those obtained for C4 fibers (Fig. 5). It seems reasonable to conclude that such results are to be expected when ID values increase with an increase in EP and/or WFR, i.e., when fiber stretching governs the ultimate fiber dimensions in fiber production.

Figure 12 shows UF performance data for the C5 fibers produced. The results show that with an increase in EP in fiber production, PEG separations tended to increase and the corresponding PR tended to decrease, both more steeply at lower EP and much less steeply at high EP. These results indicate that the average pore size on the skin layer on the bore side surface of the fiber tended to decrease with an increase in EP in the 20 to 60 psig range during fiber production.

The above results are not necessarily in contradiction with those obtained for C4 fibers, shown in Fig. 6, which exhibited a maximum in PEG separations for fibers produced at an EP of 30 psig. The data shown in Fig. 12 for C5 fibers may be considered to be similar to those observed for C4 fibers in the EP range of 20 to 30 psig. This means the possibility exists that PEG separations for C5 fibers may show a maximum at some EP higher than 60 psig; this possibility, however, needs to be proved.

As pointed out already, an appropriate desolvation rate during fiber production tends to decrease the size of skin layer pores. Fiber stretching (EP) tends to increase the size of such pores, and fiber swelling can contribute to both a decrease and an increase in the size of such pores. The UF performance data for C5 fibers shown in Fig. 12 indicate that under the conditions used for fiber production, skin layer morphology was primarily controlled by the desolvation effect which was progressively moderated by the increasing fiber stretching effect at higher EP. This conclusion is consistent with the observations made earlier on the desolvation effect on C5 fiber dimensions.

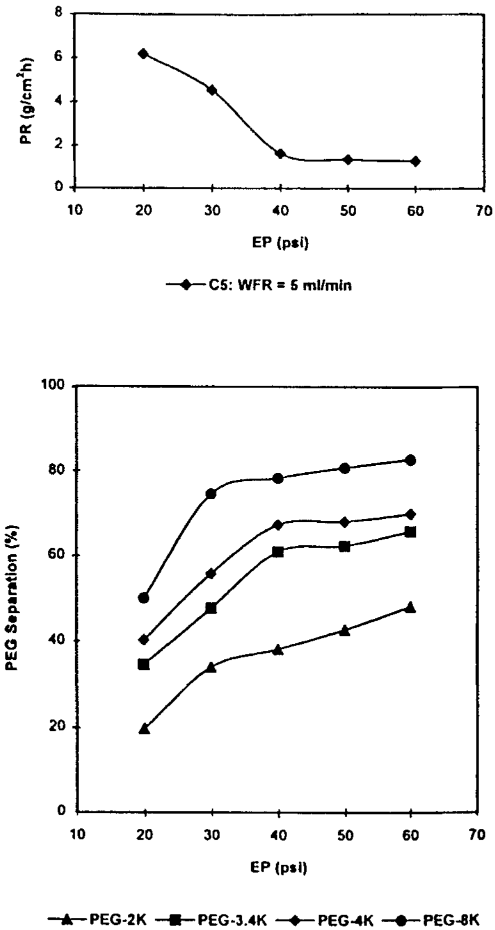


FIG. 12 Effects of EP and WFR on UF performance of C5 fibers.



## CONCLUSIONS

The following general conclusions arise from the foregoing results.

1. With an increase in EP during fiber production, OD always tends to increase for the resulting fibers.
2. An increase in PVP concentration in the fiber spinning solution contributes to a greater fiber swelling effect.
3. NFV tends to increase with an increase in EP, but it decreases consid-

erably with an increase in PVP concentration in the fiber spinning solution and the consequent increase in solution viscosity.

4. Both fiber dimensions and skin layer morphology are governed by the combined effects of desolvation, fiber swelling, and fiber stretching during fiber production.

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