Experimental Design Applied to Fabrication of PSf Membranes via NIPS Method

Part2: Influential Parameters on Pure Water Permeability and Selection Criteria for Optimum Membrane in MF Process

Parya Amirabedi and Reza Yegani

Abstract—**Polysulfone flat sheet membranes are fabricated by using solvent-non solvent exchange method. NMP is used as solvent and PEG 20000 as additive. Full factorial design is used to examine the individual and mutual impacts of parameters on the membranes. To that effect, the individual and mutual impacts of such parameters as polymer materials concentration, PEG 20000 concentration as well as non-solvent bath composition on the structure and function of membranes are studied. The structural analyses include porosity measurement and observation of SEM images. The membranes are subjected to pure water permeability for assessing their functions and operational evaluations. The effective parameters in the porosity analysis include, in terms of priority, coagulation bath composition, polymer concentration and PEG concentration while in pure water permeability analysis, PEG concentration overtakes polymer concentration.**

The results show that when PEG concentration in dope solution and NMP concentration in coagulation bath exceed than 9% (wt/wt) and 30% (wt/wt) while polymer concentration is respectively between 14 and 15% (wt/wt), the fabricated membrane would be suitable for microfiltration (MF) process.

Key words: Full factorial design, porosity, pure water permeability, finger-like pores

I. INTRODUCTION

Using membranes for separation is a breakthrough in engineering and environmental processes. It is important to know that the membrane used in a membrane process largely contributes to the quality of final product. Therefore, the proper membrane is required for an effective separation process [1, 2]. It is important to note that, the structure and function of the membrane depend on the materials and circumstances [3]. It is clear that, the mechanical features of the membrane rely on the structure and distribution of pores in membrane. Finger-like pores are inappropriate due to their low tensile and mechanical strength, which easily causes the membrane tear apart in presence of any tensile tensions and/or high-pressure operations [4]. Although in some articles, mechanical strength is referred as an important index for assessing the function of membranes notably in high-pressure processes and membrane contractors, water permeability could be much important than mechanical strength in water treatment processes. Furthermore, since porosity and water permeability are directly proportional, few studies have been conducted to examine their mutual impacts. That is why a handful of scientific endeavors have been undertaken in view of fabricating membranes with few finger-like pores, high permeability and selectivity as well as appropriate tensile and mechanical strength [5]. It is important to know that, the viscosity of polymer solution depends on the materials used in fabrication of polymer solution and plays a significant role in fabrication of membranes devoid of finger-like pores and subsequently improvement of the process and tensile features. The conducted studies on these issues show that, the viscosity of the polymer solution significantly affects the solvent-non solvent exchange in the polymer solution [6-9]. Kinetic surveys indicate that, additives modify the exchange speed by changing viscosity in the solution. Using of polymer additives like PVP and PEG are suggested to achieve a spongy structure, reduce finger-like pores and increasing of porosity [10-18]. Chang *et al.* [19] studied the impact of polymer concentration on PVDF membranes structure and concluded that high polymer concentration slows the penetration of non-solvent down into the polymer solution and would delay phase separation. The immediate result is the decline of number of finger-like pores in the membrane. The coagulation bath conditions also constitute a key parameter affecting the structure of membrane. Wijmans *et al.* [20] studied the impact of additive on PSf flat sheet membranes. They concluded that the membrane surface structure changes when the bath composition changes from water to 80 wt% NMP aqueous solution. In the absence of solvent, the outer surface was non- porous, however, its presence in the coagulation caused porosity on the surface. Thus, many studies have been conducted on the fabrication of polysulfone membranes; however, no concrete results have been achieved about the mutual impact and significance of parameters. Experimental design is a branch of knowledge helping to measure the impact of influential factors (X1, X2, X3,...) on the outputs (Y1, Y2, Y3,...) under Yi=F(Xi) equation [21]. Examining the individual and mutual impacts of parameters in order to reduce finger-like pores and increase surface pores were among the main objectives of this research. Measuring F values, indicative

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of the significance of parameters, can meet this goal. Full factorial design was the best option to achieve this objective as it minimizes errors. Khayet *et al.* [22] reported the fractional factorial design for possible fabrication of membranes by the dry/wet spinning technique. Seven spinning factors were taken into account. An optimal membrane was finally fabricated using the determined optimum spinning conditions. This membrane exhibits the highest performance index and the greatest global desirability (i.e. high permeate flux and salt rejection factor).

In this paper, in continuation of the previous article[23], the effect and significance of influential parameters like polymer concentration, the composition percentage of coagulation bath and PEG concentration on the pure water permeability are examined. Moreover, the individual and mutual impacts of influential parameters on the micro filtration (MF) process as target functions are discussed. Finally a proper strategy for simultaneous optimization of the three features in MF process is proposed.

II. Materials and Methods

A. Materials

Polysulfone (ρ =1250 (kg/m³), T_g=190°C) was purchased from Slovay, N-Methyl-2-Pyrrolidon (NMP) (ρ = 1030 (kg/m^3) was obtained from Daejung and poly (ethylene glycol) (PEG) (Mw = 20000(g/g mol)) was purchased from Merck. All chemical and reagents were used with no more treatment, unless otherwise described.

B. Membrane fabrication

Proper amounts of polymer, additive and solvent were measured for weight and placed in a closed glass container. The mixture was stirred with a magnet stirrer to give a homogenous solution and then enough time was given for bubbles to be completely released. Then, a portion of the achieved solution was poured onto flat plate glass and spread out using an automatic casting knife at the speed of 5 mm/sec. The thickness of all membranes was kept constant at 150 micrometers. The glass plate containing polymeric solution was then immersed into the non-solvent coagulation bath for sufficient period of time where phase separation took place to give a porous structure. Coagulation bath solution was changed every 24 h.

C. Scanning Electron Microscopy (SEM)

In order to create cross-section and outer-surface images of the membranes, a scanning electron microscope (SEM LEO 440-I) method was used. The samples were broken in liquid nitrogen after being dried in the ambient temperature. The samples were then stuck on specific holders, gold-sputtered and imaged.

D. Porosity of membrane

In this method, the samples were cut in specific sizes before being weighed in a digital balance. After noting their dry weights, the samples were dipped into isobutanol for 24 h until all their pores were filled with isobutanol. For each membrane, four samples were taken for porosity analysis. The porosity of membranes was calculated using the following equation [21]:

$$\varepsilon = \frac{(w_w - w_d)/\rho_w}{(w_w - w_d)/\rho_w + \left(w_d/\rho_p\right)} \times 100 \quad (1)$$

where, w_w is the mass of wet membrane in g, w_d is the mass of dry membrane in g, ρ_w and ρ_p are the density of isobutanol and polysulfone in (g/cm^3), respectively. In the determination of the membrane porosity, it was assumed that all the pores in the membranes were completely filled with isobutanol. This analysis gave overall porosity of membrane included bulk porosity and surface porosity.

E. Pure Water Permeability Measurement

This test involves a batch process throughout which water flows into membranes. Due to the microstructure of fabricated membranes which could be confirmed by SEM images [23], these membranes are suitable for microfiltration process. Therefore during the permeability measurement, distilled water permeated the surface of membranes at a 2.5 bar pressure, which was set at 2 bar when the sampling began. The flow of water for ten minutes was weighed. The test was repeated for all four samples. Eq. 2 shows the flow and measurement of water permeation.

$$J_w = \frac{Q}{A\Delta P} \quad (2)$$

where, Q is the flow of water in (l/m².bar.hr), A is the effective membrane surface area in square meters and ΔP shows the difference in pressure.

F. Experimental Design

Three parameters; i.e. polymer concentration, PEG additive concentration and NMP solvent concentration in the coagulation bath concentration were selected to examine their roles in water permeation. The results are available in Table I.

TABLE I
FACTORS AND LEVELS TO BE USED IN FABRICATION OF FLAT PLATE MEMBRANES

Factors for flat sheet membranes	levels
Polymer concentration (wt%)	12, 15, 18
PEG concentration (wt%)	0, 5, 10
NMP concentration in coagulation bath (wt%)	0, 60

Similar to the previous article, variance analysis table, degree of freedom for each factor, total response squares and F as well as P values were calculated for a statistical analysis of results and study of the impact of parameters on the pure water permeation.

Again, P value was used to assess the impact of a single parameter on a specific response and F value helped to find out the impact of all parameters on a specific response.

III. Results and Discussions

Results of analyses are fully available in Table II.

A. Porosity Analysis

As show in Table III, the P value calculations show that

all parameters, except for PEG and polymer concentrations mutual impact, are influential on the membrane porosity. F-value is maximal for coagulation bath composition and therefore this parameter is the most influential factor. Polymer and PEG concentrations come next so that the less F-value, the less degree of importance of parameters. As illustrated in Table III, the impact of mutual parameters is ignorable in comparison to individual parameters on the porosity.

TABLE II
OBTAINED RESULTS FROM PERFORMED ANALYSIS ON THE MEMBRANES

Sample	PSf conc. (wt%)	PEG conc. (wt%)	NMP conc. (wt%)	Porosity (%)	Permeability (l/m².bar.hr)
1	12	0	0	86.4211	118.45
2	12	0	60	83.5402	48.57
3	12	5	0	90.3231	134.25
4	12	5	60	85.7251	457.18
5	12	10	0	91.1663	195.25
6	12	10	60	86.2013	648.80
7	15	0	0	85.6220	18.40
8	15	0	60	80.5281	25.20
9	15	5	0	88.6112	98.73
10	15	5	60	82.3253	146.45
11	15	10	0	89.5164	138.11
12	15	10	60	83.3891	236.45
13	18	0	0	82.4550	14.61
14	18	0	60	78.0252	17.57
15	18	5	0	85.2052	76.78
16	18	5	60	80.6603	106.63
17	18	10	0	85.9802	131.50
18	18	10	60	81.7201	146.15

B. *Pure Water Permeation Analysis*

Since membrane porosity plays the most important role in the performance of fabricated membrane especially from water permeation point of view, it is very important to clearly show the impacts of the types of the porosity on the water permeation as well as on the membrane rigidity. The latter case was completely discussed in the previous article; however, we need to examine the impact of the type of porosity on the pure water permeation, too.

The operational evaluation of a fabricated membrane, along with its structural assessment, can offer an accurate analysis of the impact of parameters on the fabrication of porous membranes. Water permeation is one of the tests indicating changes in the porosity and mechanical strength. Table IV shows the results of variance analysis of data after water permeability. All P values equal zero, indicating that the individual and interactional impacts of all parameters are identical. F values calculations show that the most important factor in increasing pure water permeability is PEG concentration in the fabrication of polymer solution. Polymer concentration and the composition of coagulation bath come next in terms of significance. In other words, the impacts of parameters on the water permeability and porosity are totally reverse. This issue endorses the significance of the type of porosity on the water permeability. On the other hand, it would be wrong to imagine that higher porosity would result in higher water permeability. The individual and mutual impacts of parameters on water permeability are discussed in the following paragraphs.

1) Impact of PEG Concentration on Water Permeability of Membranes

As illustrated in Figure 1, permeability is low in the membranes where PEG additive concentration is zero. The diagrams show identical results for all three polymer concentrations, however, the significance of interaction between influential parameters do not allow similar changes in the water permeability after PEG has been added at the same amount.

TABLE III
OBTAINED RESULTS FROM ANALYSIS OF VARIANCE OF DATA FOR POROSITY

Parameters	DF	AdjSS	AdjMS	F	P
NMP concentration in bath	1	207.026	207.026	1741.440	0.000
Polymer concentration	2	143.528	71.764	603.660	0.000
PEG concentration	2	83.244	41.622	350.110	0.000
Polymer concentration* NMP concentration in bath	2	4.974	2.487	20.920	0.000
PEG concentration* NMP concentration in bath	2	2.009	1.005	8.450	0.003
Polymer concentration* PEG concentration* NMP concentration in bath	4	1.398	0.349	2.940	0.049
Polymer concentration* PEG concentration	4	0.450	0.112	0.950	0.460
Error	18	2.140	0.119		
Total	35				

The conducted studies so far show that surface porosity is a key parameter in determining pure water permeation of membranes. Bulk porosity, however does not significantly affect permeability [6]. Given the fact that the surface porosity of membranes increases with PEG concentration, the permeability increases.

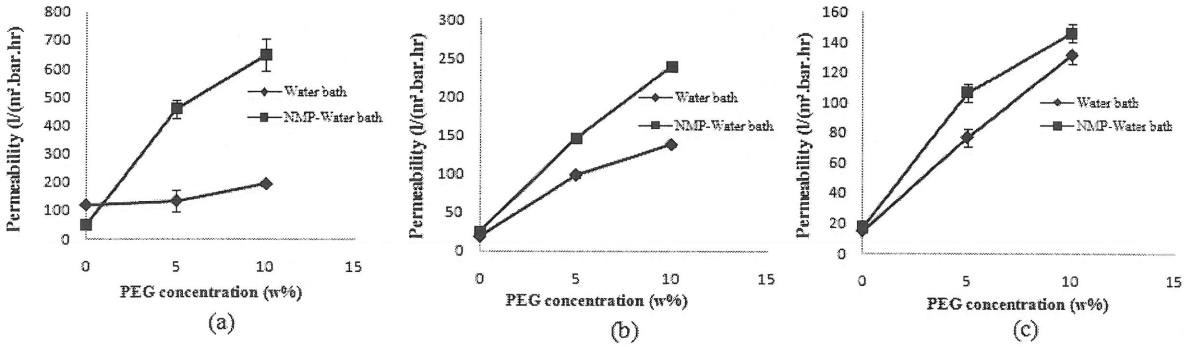


Fig.1. Diagram of pure water permeability vs. PEG concentration for three polysulfone concentrations (a) 12 wt%, (b) 15 wt%, (c) 18 wt%, and for two types of coagulation bath; pure water and mixture of NMP –Water.

TABLE IV
OBTAINED RESULTS FROM ANALYSIS OF VARIANCE OF DATA FOR PURE WATER PERMEABILITY

Parameters	DF	AdjSS	AdjMS	F	P
NMP concentration in bath	2	266886.001	133443.014	393.480	0.000
PEG concentration	2	237942.024	118971.020	350.810	0.000
Polymer concentration	1	91386.101	91386.001	269.471	0.000
PEG concentration* NMP concentration in bath	2	83581.006	41790.023	123.231	0.000
Polymer concentration* NMP concentration in bath	2	70271.012	35136.041	103.600	0.000
Polymer concentration* PEG concentration* NMP concentration in bath	4	82743.103	20686.101	61.002	0.000
Polymer concentration* PEG concentration	4	53516.110	13379.112	39.451	0.000
Error	18	6104.012	339.056		
Total	35				

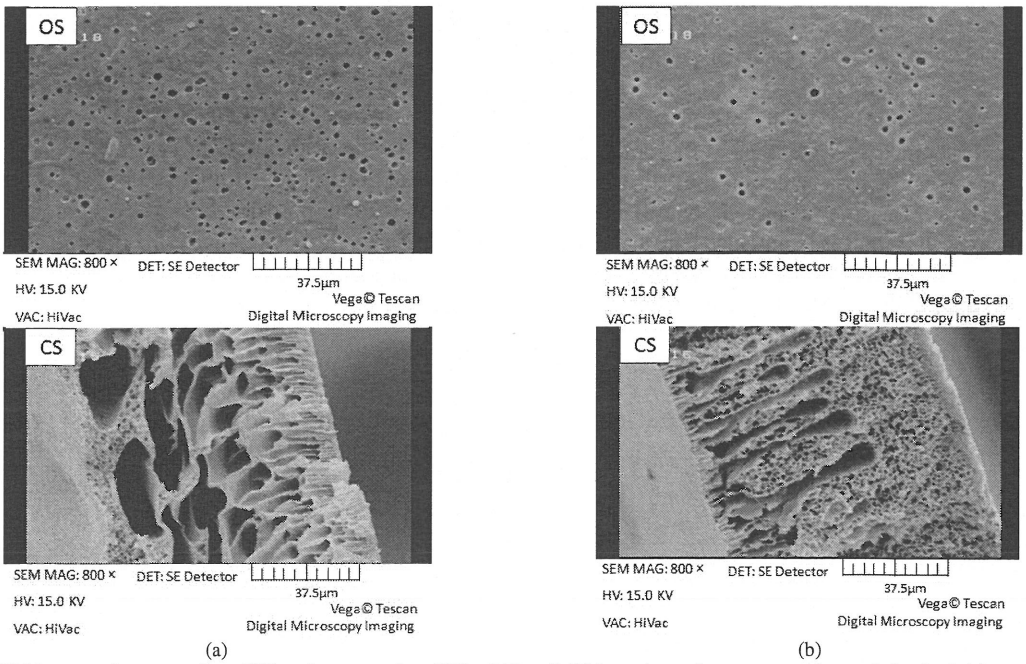
The point deduced from these diagrams is that the permeability in water bath is more than in water-solvent bath for polymer of 12% concentration when PEG concentration is zero. However, in other samples, the result is contrary. It could be explained by the fact that in this sample, due to low viscosity in the solutions lacking PEG, the solvent-non solvent exchange occurs quickly in the water bath, leading to formation of big finger-like pores in the interior structure of the membrane so that the pores are extended up to the outer surface. Furthermore, in the presence of solvent in coagulation bath, due to slow exchange, finger-like pores are fewer and they cannot leave significant impacts on the pores. Therefore, the high level of permeability in water bath could be attributed to the existence of these large finger-like pores up to the outer surface of the membrane. SEM images in Fig. 2 illustrate this conclusion. The point is that, higher PEG concentration increases the viscosity of polymer solution and reduces the possibility of formation of such pores when phase separation occurs in coagulation bath containing only water. It is then clear that in high PEG concentrations, permeability is high due to more surface pores. When the polymer concentration rises from 12 to 15 and 18 percent, the viscosity of polymer-solvent solution increases to such a level that pure water could no longer cause finger-like pores extending up to the outer surface of the membrane. Therefore, this parameter loses its significance in high polymer concentrations and PEG alone justifies the high permeability of water. It could be deduced from Table IV that the mutual impact of polymer concentration and PEG concentration is meaningful and when the polymer concentration is above 12 percent, the mutual impact is bigger than the impact of bath composition.

Effect of Polymer Concentration on Water Permeability

The results summarized in Table II show that the permeability has declined with polymer concentration enhancement. As mentioned earlier, the number of surface pores is a key parameter in determining the pure water permeability from the membrane. Since more surface pores take shape in 12% concentration, it accounts for the higher amount of permeability. As polymer concentration rises, surface pores and pure water permeability gradually decline.

Impact of Bath Composition on Water Permeability

The diagrams in Fig. 1 show that existence of NMP



(a) (b)

Fig.2. SEM images of cross sections (CS) and outer surface (OS) of 12 wt% PSf membrane in two types of coagulation bath (a) pure water and (b) mixture of NMP –Water.

solvent in coagulation bath often increases pure water permeability.

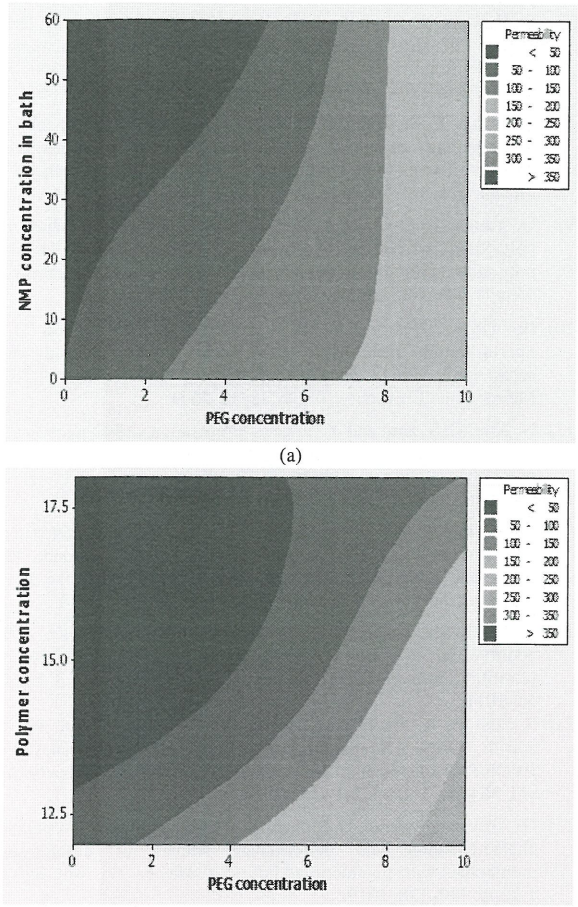


Fig.3. Contour plots of pure water permeability vs. (a) PEG concentration in dope solution and NMP concentration in coagulation bath (b) PEG and polymer concentrations in dope solution.

Given the results obtained from porosity analysis, it could be concluded that existence of solvent in the coagulation bath increases surface porosity, which in turn, would effectively increase pure water permeability. SEM images in Fig.2 confirm such a conclusion. As seen in these images, the surface porosity is higher in the membranes using solvent-containing coagulation bath. Moreover, a spongy structure is seen in the outer layer and the cross-section of the membrane. This structure plays a significant role in water permeability.

C. Selection Criteria for Suitable Membrane in Microfiltration (MF) Process

Fabricated membrane should meet all required specifications of each process. In other words, due to the required conditions, it is necessary to design and fabricate suitable membrane. Required operational conditions for each membrane related process are described in some references [1]. Due to SEM images in present and previous published articles [23] it is easy to understand that fabricated membranes in these works are suitable for microfiltration (MF) process. As explained earlier, pure water permeation and mechanical strength are two main membrane features which show their impact in all membrane related processes. Taking into account the fact that in MF process, pure water permeation and mechanical strength should exceed 50 l/(m².bar.hr) and 2 MPa, respectively, it becomes possible to modify those membrane's features, simultaneously.

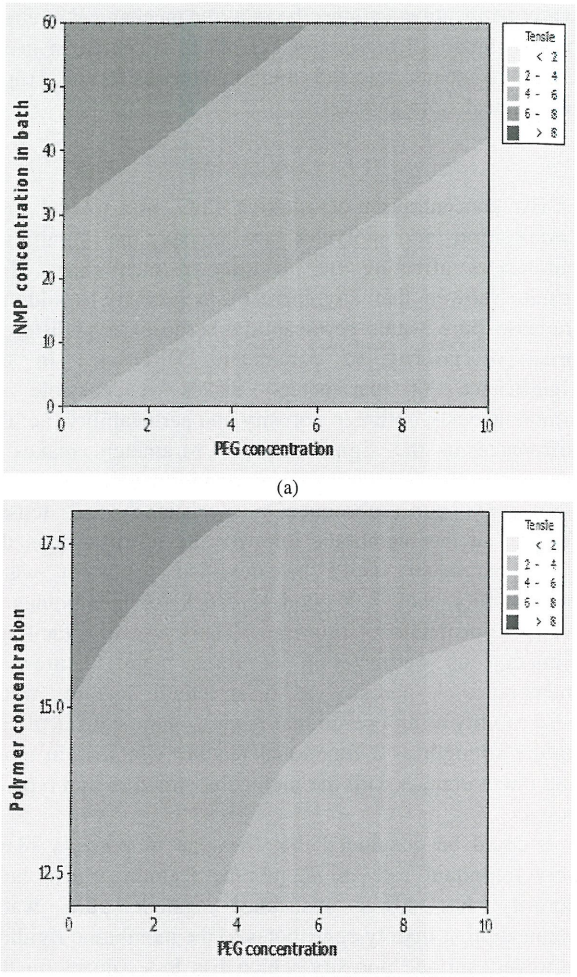


Fig.4. Contour plots of mechanical strength vs. (a) PEG concentration in dope solution and NMP concentration in coagulation bath (b) PEG and polymer concentrations in dope solution.

Contour diagrams of three influential parameters such as PEG concentration, polymer concentration and coagulation bath compositions on the pure water permeation (PWP) and mechanical strength are shown in Figs. 3 and 4 respectively. To get better specification of fabricated membrane for MF processes, PWP and mechanical strength are considered to be above 200 l/(m².bar.hr) and 2 MPa, respectively to completely meet all required conditions.

Three factors involve in contour plots for water permeability and each plot show relation of two factors in which the third factor is kept constant. Comparing two contour plots, optimum region is easily determined. As shown in Fig. 3, green colored area (please see the

http://www.itast.org/ for color print) are suitable region for pure water permeation which implies that membrane should contain PEG concentration more than 9% (wt/wt), NMP more than 30% (wt/wt) in coagulation bath and polymer concentration between 14 and 15% (wt/wt).

Fig. 4 shows various regions for mechanical strength of fabricated membrane. It is very important to pay attention to the point that whether the modified membrane for pure water permeation satisfies the required mechanical strength or not. Since all regions in Fig. 4 are appeared in green color, it means selected membrane consists of proper mechanical strength level.

IV. Conclusion

The concentration of additive PEG, non-solvent bath composition and polymer are among the significant parameters affecting the features of membranes. The results showed that any increase in porosity would not increase pure water permeability because of a different order of priority of parameters. Difference in the significance of parameters makes it possible to simultaneously optimize porosity and permeability, i.e. the difference in the significance of parameters makes it possible to use one parameter to avoid formation of finger-like pores and subsequently mechanical and tensile features of the membrane improve. In the meantime, the other parameter could be modified to boost water permeability, that is, a good yardstick for membranes in water purification process. That would facilitate simultaneous optimization of three main features of membranes – porosity as a structural feature, water permeability as an operational feature, and mechanical and tensile strength as a functional feature, which is in close and direct contact with the molecular structure and type of porosity.

It could be concluded that the type of porosity plays very important role on membrane features. Since bulk porosity has lower resistance against pure water permeation, it mainly contributes to the membrane rigidity, however, surface porosity which has less impact on the mechanical strength, plays very important role in pure water permeation. In fact, higher surface porosity provides higher water permeation.

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A Novel Approach in Geometrical-Mechanical Analysis of Plain Woven Fabrics; Initial Load-Extension Behavior

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Abstract—**A theoretical analysis for the initial state of load-extension behavior in plain woven fabrics is presented. For this purpose, a new approach for geometrical modeling of woven fabrics consisting of its structure in inclined and float regions is developed which results in theoretical estimation of all the structural parameters of plain fabrics including its weave angle. Then, by applying the strain energy method and considering a virtual spring in the unit cell, a modified model for predicting the initial tensile modulus of plain woven fabrics is proposed. The results are shown better agreement with experimental data than previous models.**

Key words: Load-extension, weave angle, strain energy, initial modulus

I. INTRODUCTION

The mechanical behavior of woven fabrics is of interest in numerous applications, including apparel and technical usages. Engineered designing of textile fabrics for specific mechanical properties requires the ability to predict its behavior in various loading conditions which among them, tensile properties has been analyzed by numerous researchers [1-7]. However, due to the geometrical model and the approach in mechanical analysis, most of the available works are resulted in complicated elliptic integrals or needs numerical solutions even in the domain of small deformation analysis.

There are numerous applications of woven fabrics involved with small extensions which have received much attention by some scientists [8-10]. The work developed by Leaf and Kandil [10] that considering this state of deformation is the basic approach in this work which is tractable and amenable to predict the initial load extension behavior of plain woven fabrics in a close form solution.

In this work, a new approach for the geometrical-mechanical analysis of the plain woven fabrics is developed which is also applicable to other woven structures.

It is assumed that the yarns cross section are always circular along the yarn path as was developed by Peirce [11] and the yarns center line obeys the saw tooth geometry which was developed by Kawabata [12]. On the base of these assumptions, a new geometrical modelling is proposed which includes both inclined and float regions. The mechanical approach in this work employs the strain energy method through applying the Castigliano's theorem. A virtual spring is defined in the unit cell of the structure and used in mechanical analysis which results in better prediction of initial tensile modulus of plain woven fabrics. The effect of friction is ignored in this work. It is shown that the proposed theory does lead to results that are in agreement with experimental data.

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II. GEOMETRICAL MODELLING

Geometrical modelling of woven structures has been the subject of many works [13-16]. Weaves geometry is determined by defining a unit cell representing the whole fabric characteristics. The proposed model and its structural parameters for the weave repeat of plain woven fabrics is shown in Figure 1. The indices i and j are used to denote warp and weft yarns sequentially and the indices f and c represent float and inclined regions respectively. The weave repeat in Figure 1 is shown for the weft yarns length which is included by the cross section of warp yarns. So, this structure can be also developed for warp lengthwise by substituting the index i instead of j. Moreover, Y_i is used to denote the spacing between warp yarns while P_j is the width of unit cell. In the unit cell, P_{fj} and P_{cj} are used to denote the projection of weft yarns float and inclined length, respectively. It should be noted here that in this work, all the lengths are considered to be in mm and the angels are in degree.

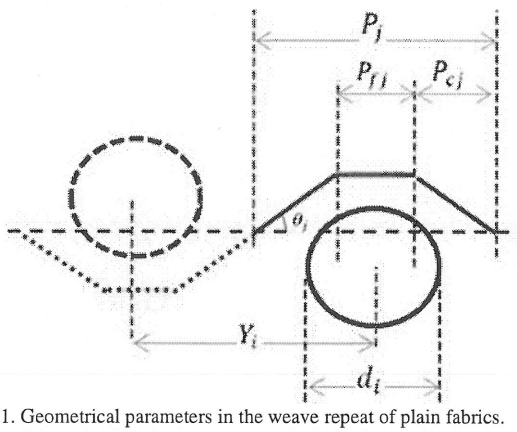

Fig. 1. Geometrical parameters in the weave repeat of plain fabrics.

The yarns diameter (d) is calculated by Eq. (1) given by Peirce [11].