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Membrane from Technical Grade Poly(Vinyl Chloride): Preparation and Characterization

M. A. Ahmed, M. F. Uddin, M. M. Hossain, M. A. Islam*

Center for Environmental Process Engineering, Department of Chemical Engineering and Polymer Science, Shahjalal University of Science and Technology (SUST), Sylhet-3114, Bangladesh

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Abstract

Sophisticated separation without membrane technology is unthinkable in modern chemical industries using process water and discharging effluents contaminated with various solid and liquid pollutants, but Bangladesh is far behind the advanced world in terms of membrane manufacturing and uses. This work initiates research in the field and successfully develops membranes from technical grade poly(vinyl chloride) (PVC) applicable in water and wastewater treatment. Porous structure observed by Scanning Electron Microscope looks somewhat finger like channel structure bounded from both upper and lower surfaces by thin skin layers, called ‘selective layer’. This resembles partially to ultrafiltration membrane structure. However, the pore-size distribution measured by Liquid displacement porometry, shows that the pore-sizes are in the typical microfiltration ranges. The membrane permeability decreases with an increase in the casting solution concentration. It is confirmed that micro- and ultra- filtration membranes can be prepared from PVC with desired pore-size distribution and ‘selective layer’ by phase inversion method varying the preparation parameters. This is a pioneering work in the country in developing polymer membrane preparation technique and would inspire young researchers to develop it further for applications in native industries.

Keywords: Membrane; Microfiltration; Ultrafiltration; Poly(vinyl chloride); Water treatment.

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1. Introduction

Since its first applications in bacterial analysis during post- first world war times in German laboratories, membrane technology has undergone tremendous development and today it plays tremendous role in separation processes in liquid, gas and gas-liquid systems. It has got vast applications in water and wastewater treatment in different sectors such as water supply for domestic and industrial purposes [1-5], and chemical [6], pharmaceutical [7], biotechnological [8], food, beverages [9], oil [10], mine and hydrometallurgical industries [11], thermal and nuclear power plants [12], and fuel cells [13], lithium-ion battery [14] and medical application [15] and many other industries. For wastewater treatment and reuse

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* *Corresponding author*: mislam@sust.edu

also, membrane technology is successfully applied in different sectors, e.g. membrane bioreactor technology for municipal wastewater treatment [16], ultrafiltration membrane process for removing radioactive particles from nuclear wastewater [17,18], reverse osmosis membrane for treating laundry wastewater for reuse [19] and so on.

In Bangladesh, however, this technology has still limited applications. In a large scale, it is applied in preparing bottled water for drinking purposes, but the price is very high for Bangladesh standard. In some clinical laboratories also, microfiltration membrane is used for concentration of enzymes. Membrane technology could bring high quality product in Bangladesh dairy and fruit juice industries. Also, membrane bioreactor technology could bring sustainability in treatment of municipal wastewater and textile dyeing industries [20]. These are few examples of potential fields of immediate application of membrane technology in Bangladesh, but still the entrepreneurs and industries are not much interested in development of high-quality product through membrane technology. The prime reason is; the price seems high for native consumers.

The most important element in membrane technology is the membrane itself, and polymer-based membranes have got versatile application due to the flexibility of the material in module formation. The exact methodology of the preparation of polymer membrane is a secret maintained by the manufacturer, but still the preparation method, in general sense, is known in literature [21-23]. Among all the available polymer membrane preparation methods, non-solvent induced phase separation method is most practiced [24-29]. In this method, polymer is dissolved in an organic solvent, cast in a film and finally precipitated in water to leach the solvent out leaving a porous thin film called membrane. Most membranes (microfiltration, ultrafiltration and reverse osmosis) for water treatment are prepared by this method [30,31].

A number of commercially available polymers could be potential candidates as membrane materials for water treatment. Membrane producers, however, prepare their own polymers with varying characteristics and the recipe of casting solution for membrane preparation is either kept secret or defended with patents. Generally, polymer membranes prepared in water bath are brittle in dry condition and their porous structure collapses on drying making them unsuitable for application. To minimize these destructive processes, the manufacturers modify the polymer material, bring changes in recipe, and most importantly, make expensive physical and chemical treatment to the prepared membranes to preserve the integrity of the membranes and to minimize collapse of the porous structure. In membrane module the membrane pores are filled with poorly volatile liquids. Before purposeful applications, the membranes are thoroughly washed with water first. In fact, these membranes are formed in water and ultimately used in aqueous media, but the processes that they have undergone for transfer from the manufacturer to consumer make them expensive. These expenditures could be practically avoided, if the membrane and its modules are prepared in the premises of the consuming laboratories and industries i.e. if the manufacturer and consumer are the same entity. No industry in Bangladesh produces synthetic polymers that could be used as membrane material. But in the local market, various technical grade polymers are available, few of them are soluble in organic solvents, which could be extracted from the solution by water. One such polymer is poly(vinyl chloride) (PVC), which is imported in the country for small entrepreneurs or industries without any certificate of material characterization. Even it is unknown whether it represents a pure component or a composition with ingredients. This is a material used for preparation of articles for everyday uses by compression molding (melting under pressure and formation of articles on cooling). Such materials, if appear suitable for membrane preparation would certainly be highly economical for in situ membrane preparation unit in a number of industries in the country. As argued, the purpose of the present work is the preparation of membranes from locally available polymers. A number of technical grade polymers are available in local market, but for the present study, PVC has been chosen as the test case for studying its potentiality as a membrane material. Membrane properties were controlled mainly through variation in the polymer concentration in the casting solution, evaporation time, coagulation bath temperature and composition. Casting film thickness also determines ultimate membrane thickness as well as morphology [32]. With manual casting membrane thickness could hardly be precisely maintained. To ascertain optimal condition for preparation, intensive experimental work is required covering wide range of membrane preparation parameters. Since the target of the present work is to establish only the potential of a technical grade polymer, the preparation condition in this study will include only variation in casting solution concentration keeping evaporation time and coagulation bath composition constant. The membrane performance was evaluated by water flux under pressure-driven processes. The morphology was studied by Scanning Electron Micrograph (SEM) and the pore-size distribution was determined by liquid displacement porometry. The experimental results are highly encouraging and show that varying PVC-concentration in the casting solution, micro- and ultra-filtration membranes can be prepared from the technical grade PVC and through in situ preparation, these membranes could be used in water and wastewater treatment in the country. Also, to the authors' knowledge, PVC membranes prepared by non-solvent induced phase separation are not commercialized yet by membrane manufacturers. Therefore, although this work presents the primary result on the preparation and characterization of a specific polymer membrane, still it bears high scientific and technological significance. This being a pioneering work in the country, young researchers will be inspired to contribute to the development of membrane technology in the country.

2. Experimental

2.1. *Materials and methods*

Technical grade PVC was chosen as membrane material. The material was available in powder form and was purchased from a local open market. There was no certification of the material, but the material was identified and characterized by laboratory tests. Dimethyl formamide (DMF), a product of Merck Specialities Private Limited, Mumbai, and tetrahydrofuran (THF), a product of research Lab Fine Chem Industries, Mumbai, were collected from a specialized chemical shop and used without further purification.

2.2. *Polymer characterization*

The polymer material has been characterized by FTIR spectroscopy (with IRPrestige-21 FTIR Spectrometer, Shimadzu, Japan). The analysis uses infrared light to scan test samples and observe functional groups of the chemical species. At first KBr was mixed with polymer powder sample thoroughly in a mortar at a ratio of 9:1. Adequate amount of the powder-mixture is placed at the bottom of the pellet die and pressed with a hand pump at 1 to 4 bar to prepare a solid tablet. The latter is carefully removed from the die and placed in the FTIR sample holder. The FTIR instrument sends infrared radiation of about 10,000 to 100 cm^{-1} through the sample, with some radiation absorbed and some passed through. The resulting signal at the detector appears as a spectrum, typically from 4000 cm^{-1} to 400 cm^{-1}, representing the molecular fingerprint of the polymer. The spectrum so obtained is compared with the spectrogram of a known PVC sample available in literature.

Thermogravimetry analysis (TGA) of the polymer sample was performed on TGA-50 Thermogravimetry Analyzer system, Shimadzu, Japan by heating the sample in the range of 22-500 °C. The sample was burned under nitrogen atmosphere with a heating rate of 10 °C/min under a controlled dry nitrogen flow of 10 mL/min. The TGA curve so obtained is compared with the that of a known PVC sample available in literature.

2.3. *Determination of molecular weight of the PVC*

Number-averaged molecular weight was determined by a viscometric method. For the purpose, PVC solution was prepared in THF in the concentration range of 0-1.75 g/mL. Then viscosity of the solutions was measured by Ostwald viscometry by methods described in refs. [33-35]. The data were treated with the following relations:

$$\eta_{sp} = \frac{\eta - \eta_0}{\eta_0}, \qquad \frac{\eta_{sp}}{C} = A_1 + A_2 C, \qquad [\eta] = \lim_{C \to 0} \frac{\eta_{sp}}{C} \tag{1-3}$$

$$[\eta] = K M_w^{\alpha} \tag{4}$$

Where η and η_0 are the viscosity of the solution and the solvent respectively, C is the concentration of the polymer solution (g/mL), A_1 and A_2 are empirical constants, and K and α, are constants characteristics for the polymer and the solvent, and M_w is the average number of repeating units in a polymer molecule. For a η_{sp}/C vs. C plot (see Eq. 2), the intercept will give the value of the intrinsic viscosity $[\eta]$ and then applying the relation in Eq. (4), the value of M_w can be estimated.

2.4. *Membrane preparation*

The polymer is dissolved in DMF. The solution is cast on one edge of a glass plate and drawn to the other end by a glass rod and thus a liquid film is formed. The film with the glass plate is exposed to open air for 1 min for the solvent to evaporate partially and then the plate along with the film is immersed in water for the residual solvent to leach out of the film. In this process, the polymer precipitates out of the liquid film forming a porous sheet called membrane, which separates out of the glass plate. The porous membrane is kept immersed in water to preserve its porous structure from collapse on drying.

2.5. *Flux measurement*

Water flow through a membrane has been measured with an ultrafiltration cell manufactured in a workshop of the University of Duisburg-Essen, Germany. The pressure is changed from 0 to 4 Bar stepwise at an increment rate of 0.5 or 1 bar. At each pressure, the flow rate through the membrane was recorded. After measuring flow rate at 4 Bar, the pressure was withdrawn at a decrement rate of 0.5 or 1 bar and the flow rate is measured again at each pressure.

The flux, J (m/s), at a given pressure, - ΔP (Pa), is calculated by the following formula:

$$J = \frac{V}{A.t} \tag{5}$$

Where V (m^3) is the volume of the liquid passing through the membrane of area A (m^2) for time t (s).

The permeability, ξ (m^2.s^{-1}.Pa^{-1}) of the membrane is defined as follows:

$$\xi = \frac{J}{(-\Delta P)/L} \tag{6}$$

Where -ΔP (Pa) is the pressure difference across the membrane and the L(m) is the membrane thickness.

2.6. *Porosity determination*

Porosity, β, is the void fraction of the membrane, and is defined and determined as follows:

$$\beta = \frac{V_p}{V_b} = \frac{(W_w - W_d)/\rho_w}{V_b} \tag{7}$$

Where V_p is the total volume of the pores, V_b is the bulk volume of membrane (geometric volume), W_w and W_d are, respectively, the weight of the completely wet and the dried membrane, and the ρ_w is the density of water.

2.7. *Pore-structure study*

The membrane surface and the cross-sections were examined with a Field Emission Scanning Electron Microscope (FESEM) instrument, model: JSM-7610F (JEOL, Japan) at standard high-vacuum conditions. An Auto Fine Coater, model: JEC-3000FC (JEOL, USA) was used to coat the outer surface of the sample with gold. The cross-section images were obtained by cutting the membrane with a sharp knife.

2.8. *Pore-size distribution measurement*

The data for pore size distribution analyses by gas liquid displacement (GLD) porometry were acquired by a Capillary Flow Porometer (Porolux 1000, Belgium). Membrane samples with a surface area of 298.6 mm^2 were used for measuring the gas flow as a function of the trans-membrane pressure, first through the wet membrane with gradual drying of the membrane, and then through a completely dried membrane. A fluoro-derivative of hydrocarbon, namely, 1,1,2,3,3,3-hexafluoro-propene ("Galwick", surface tension 16 $dyn\cdot cm^{-1}$) was used as the pore-filling liquid for the wet membrane. The data treatment method is described in detail in refs. [36-38].

3. Results and Discussion

3.1. *Identification and characterization of the material as PVC powder*

As the material is assumed to be PVC powder, three types of chemical bonds, namely, C-H, C-C and C-Cl bonds must be available in the polymer material. Fig. 1 presents the FTIR spectrogram of the material under study. There are distinct peaks: i) at 2909 cm^{-1} attributed to the C-H stretching vibrations of the $-CH_2$ groups, ii) at 1427 cm^{-1} attributed to the deformation wagging vibrations and twisting of the $-CH_2$ groups, iii) at around 1254 and 1331 cm^{-1} corresponding to the deformation of C-H near Cl, iv) at around 963 cm^{-1} corresponding to the C-C stretching vibrations of PVC backbone chain, and v) strong peaks in the range of 600-800 cm^{-1} attributed to the stretching vibrations of the C-Cl bonds present in PVC. FTIR peaks at nearby positions have been observed for pure PVC material by Bodîrlău *et al*. [39] also. The authors attributed their observed peaks at i) 2929 cm^{-1} to C-H stretching from $-CH_2$, ii) 1430 cm^{-1} to wagging $-CH_2$, iii) 1272 cm^{-1} to C-H stretching from CH-Cl) iv) 958 cm^{-1} to rocking $-CH_2$, and v) 703, 639 and 616 cm^{-1} to C-Cl stretching. Definitely, the characteristic peaks available in the FTIR spectrogram of the polymer material under study are quite adequate for the material to be identified as PVC.

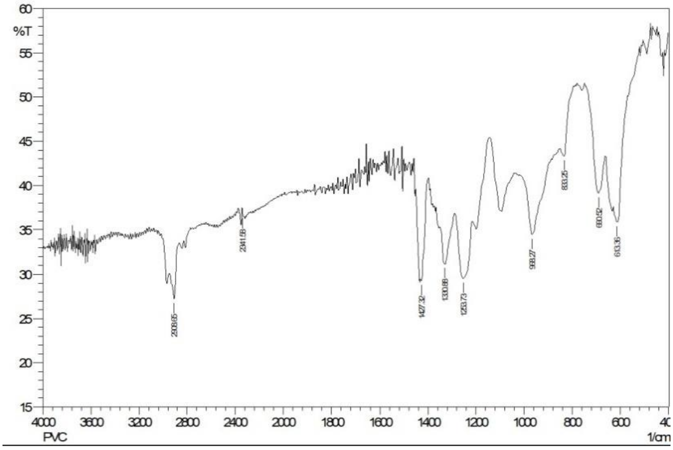


Fig. 1. FTIR spectrum of PVC.

3.3. *Thermal stability of the polymer material*

Thermal stability of the membrane material plays important role during preservation and application of the membrane, and this property is tested through TGA. The present material is also subjected to thermogravimetric analysis and the corresponding integral and differential thermal degradation curves are presented in Fig. 2.

As seen in the Fig. 2, the present material shows thermal stability up to 200 °C. In the short range of 200-250 °C, the material degrades slowly, and in the range of 250-300, there is a sharp degradation rate, and beyond that the degradation continues at a slower rate (monitored up to 500 °C). Similar picture was observed by Fathy *et al.* [40] in TGA study of PVC. The authors reported a thermal stability of the PVC material up to 180 °C and a sharp degradation in the temperature range of 200-250 °C followed by slow rate of degradation till 600 °C. This physical property also gives evidence for identity of chemical nature of the material under study and the standard PVC samples.

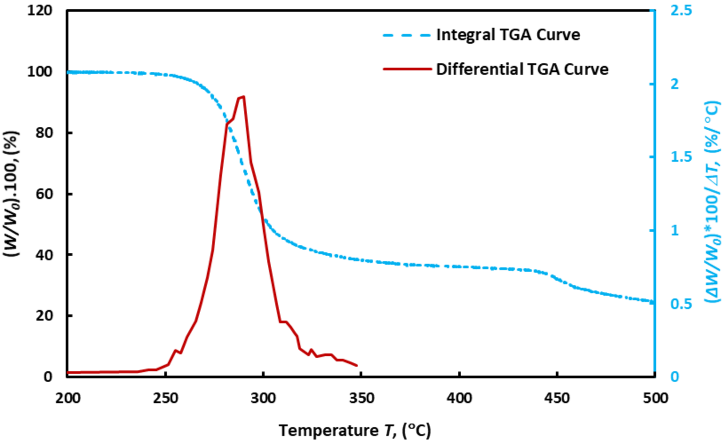


Fig. 2. Integral and differential thermodegradation curves of the polymer material under scrutiny. W- mass at temperature T, W_0 - initial mass of the sample.

3.4. *Molecular mass of the PVC under study*

Molecular weight of commercial grade PVC varies in a wide range and some important physical properties like mechanical strength, brittleness and solubility of the material are highly depend on the molecular weight of a polymer. These physical properties determine in a great extent the membrane properties and their reproducibility. For this reason, it is important to specify the molecular weight of the polymer from which membrane is formed. Fig. 3 presents the η_{sp}/C vs. C plot for PVC solution in THF.

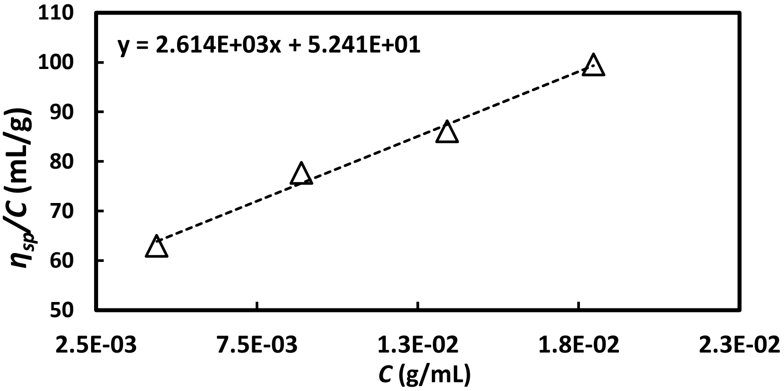


Fig. 3. Reduced specific viscosity vs. concentration plot.

As per the Eqs. (2) and (3), the intrinsic viscosity, $[\eta]$, equals to the intercept of the linear plot in Fig. 3. Then applying the relation in Eq. (4), the average molecule weight, M_w, is estimated assuming the value of K and α as 1.63×10^{-2} mL/g and 0.77 [34] respectively. The value of M_w of used PVC is 4.0×10^4 g/mol.

3.5. *Membrane preparation*

Four types of membranes are prepared for investigation. The preparation conditions and external characteristics of the membranes are presented in Table 1.

Table 1. External characteristics of membranes depending on preparation conditions.

Membrane type	Preparation condition		External characteristics of ultimate membrane	
designation	Concentration in DMF, wt %	Evaporation time τ, s	Membrane thickness, mm	External appearances
M9	9	60	0.04	White smooth sheet with no visible defect, suitable for handling
M10	10	60	0.03	
M11	11	60	0.04	
M12	12	60	0.04	

Although the casting procedure is the same for all the four types of membranes, the thickness of M10 membrane appears lower. This is attributed to unintentional variation of thickness of the liquid film during manual casting.

3.6. *Membrane characterization*

Basic properties of a membrane are its flux or permeability, and selectivity. For microfiltration membranes, selectivity is determined by the pore-size distribution of the membrane. Flux is a pressure-dependent parameter for a given membrane. For this reason, flux is reported along with the pressure under which it has been obtained. For membranes with identical pore characteristics, the fluxes will differ due to the difference in the membrane thickness, as for the same applied pressure, the pressure-gradient will be higher for membrane with lower thickness. To account for effect of the membrane thickness on the flow rate under pressure, the concept permeability has also been defined as an additional membrane characteristic. Porosity is also a parameter determining membrane flux. Higher porosity ensures higher flux, but polymer membrane with high porosity is susceptible to compaction under pressure, and that results in lower flux than expected at a given pressure.

Fig. 4. presents the flux vs. transmembrane pressure relation for the membrane prepared with casting solution concentration of 10 wt% and evaporation time τ = 60 s. The relation is similar for membranes prepared from 9, 11 and 12 wt% PVC solution at evaporation time τ = 60 s. For saving spaces, only the flux data for a membrane from10 wt% PVC solution has been presented in the paper.

As seen in the Figure, the flux increases with pressure, which is reasonable. Also, the flux curve acquired during ascending pressure cycle lies above that acquired during descending pressure cycle. Non-coincidence of the two curve and formation of hysteresis is attributed to the compaction undergone by higher pressure, which is at least pseudo-irreversible within the timeframe of observation. One interesting phenomenon, which has been observed in case of the membranes M9, M10, and M12, is that the flux curves acquired during ascending pressure cycle are convex in nature, i.e., the flux increment rate increases with an increase in the pressure. This is a bit unusual, as with an increase in pressure, the membranes undergo compaction and thus the porosity decreases. Therefore, to achieve a flux curve with concave nature was more expected. The unusual convex nature of the curve could be explained with the assumption that some dead-end pores open up with increasing pressure forming an improved communicating transport path between the two sides of the membranes. Thus, formation of ultimate pore-structure of the membrane continues during application as well.

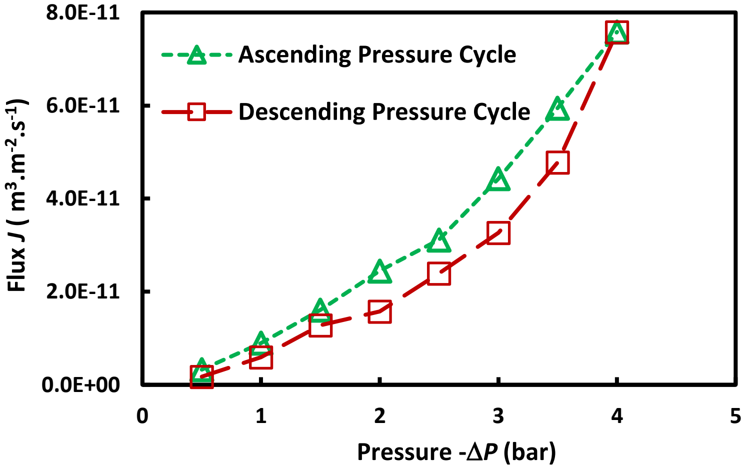


Fig. 4. Flux vs. transmembrane pressure for PVC membrane prepared from 9 wt% solution (M9) and evaporation time τ= 60 s.

Fig. 5 presents the flux and permeability (measured at 4 bar), and porosity (unpressurized) as a function of casting solution concentration. The real values of the flux and the permeability have been multiplied by a factor of 10^{12} and 10^{17} respectively to accommodate the data on the same scale (5 to 30) for presentation.

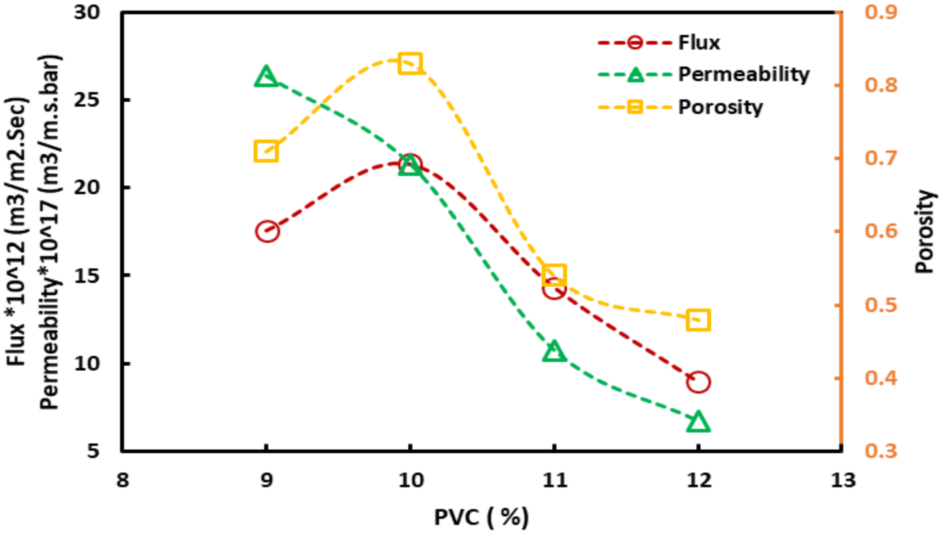


Fig. 5. Flux, permeability and porosity vs. casting solution concentration. The membranes were with different thicknesses (Table1).

There is a thumb rule that for a given evaporation time during formation (without undergoing phase inversion on the glass plate itself), the flux through the membranes decreases with the increase in the casting solution concentration, *C*, but in the present case, the flux passes through maximum for M10 (at *C* = 10 wt%). This is due to the fact that the thickness of this membrane was 0.03 mm, while the others were 0.04 mm thick (Table 1). The porosity of the membrane M10, prepared from *C* = 10 wt% is also much higher that other membranes. In defining the permeability, the thickness is taken into consideration and consequently, the permeability is monotonously decreasing with the increase in casting solution concentration. This works explores only the potential of the technical grade polymer and not the optimal condition for membrane preparation. Still, it becomes evident that preparing thinner membrane with satisfactory mechanical strength would give high flux, and that bears significance for practical purposes.

Fig. 6 presents the differential flow distribution vs. pore sizes of the membranes under study. The flow distribution of the membrane M12 has been multiplied by a factor of 0.25 to accommodate it in the distribution scale along with those of the other membranes

As seen in the Fig. 6, the pore sizes of the four membranes under study are in the microfiltration ranges [41,42], but they are well distinguished from one another. There is no sequence in arranging the pore sizes according to the casting solution concentration. As seen in the Figure, the M12 membrane has the lowest pore sizes, which might seem reasonable as higher casting solution concentration would lead more compact mass. Such logic, however, fails for the membranes M9, M10 and M11, for which higher casting solution concentration resulted in larger pore sizes. It appears that evaporation time plays an important role for different casting solutions to undergo phase inversion in the coagulation bath in different manner. The answer of this anomaly would get answer, if the effect of evaporation time is studied in detail for different casting solution concentration. The present pore distribution has been obtained for constant evaporation time, $\tau = 60$ s.

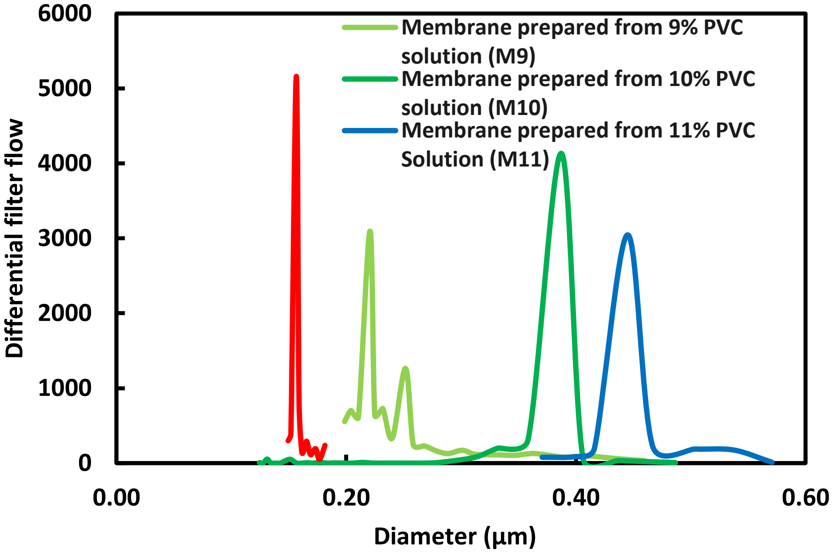


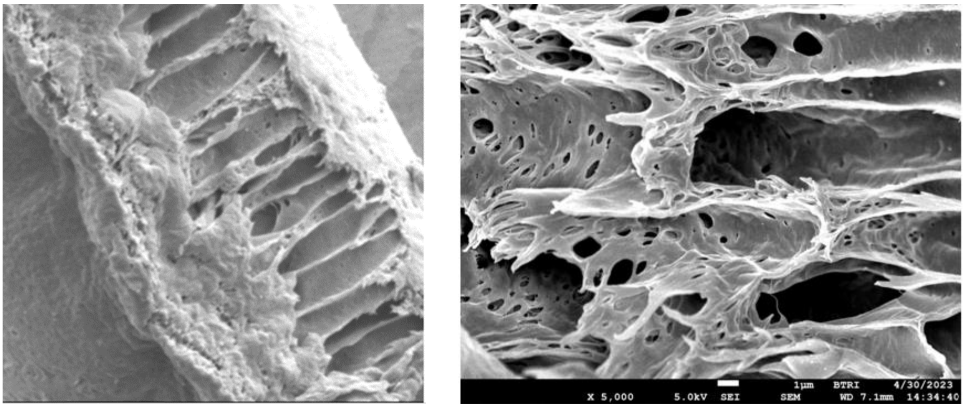
Fig. 6. Differential flow distribution vs. pore sizes of the PVC membranes.

The results of the porometry study have been summarized in Table 2. The bubble point pressure characterizing the minimum pressure for an air bubble to penetrate through a completely wetted membrane shows anomalous behavior. The same could be said for the mean flow diameter as well. An intensive study on the condition of phase inversion would give answer to these anomalies, and that is included in our continuing research program on the topic. But the membranes appear very promising from the viewpoint of narrowness of the pore-size ranges, and except M8 membrane, they are almost equivalent to nucleopore membranes prepared by neutron-bombardment.

Table 2. Summary of the results obtained from porometry data analysis.

Membrane type (see Table 1)	Bubble point pressure, Bar	Diameter range for 90% flow, µm	Mean Flow diameter, µm
M9	0.37	0.15-0.55	0.23
M10	0.32	0.33-0.43	0.40
M11	0.86	0.39-0.55	0.46
M12	2.84	0.15-0.21	0.16

Fig. 7 presents the scanning electron micrograph (SEM) of a PVC membrane prepared from a 12 wt% casting solution. Although the average pore-diameter ($D = 0.16$ µm) is lying in a standard microfiltration range [43], the pore-structure resembles partially to that of an ultrafiltration membrane [44,45]. This is an asymmetric structure with finger-like parallel pores propagating through the whole cross-section of the membrane, but bounded by two thin selective layers on both the bottom and the top sides of the membrane (Fig. 6a). Such microfiltration membrane would show mechanical strength comparable to ultrafiltration ones and also high selectivity in application. The Fig. 6b presents an extended view of the SEM image of that in Fig. 6a. The finger-like pore structure is still clearly visible, the pore-walls, however, appear not to be continuous solid mass isolating one pore from another, rather they are perforated ensuring intercommunication between the pores. Such interacting asymmetric pore-structure would ensure high flux in industrial processes.



a) Multiplication 2,000 times b) Multiplication 5,000 times

Fig. 7. SEM image of a M12 membrane (membrane prepared from 12 wt% PVC solution).

From the porometry data and the corresponding SEM images of the PVC membranes, it has become quite clear that micro- and ultrafiltration membranes can be prepared from technical grade PVC available in local market for mass usage. Through variation of casting solution concentration and evaporation time, an operating membrane team could develop their own recipe for membrane that is most convenient for their work. It should be noted that commercial membranes, that are available in the market are manufactured to serve generalized purposes, and as such they may not always be optimal for specific objects to be treated. Optimal membrane with high flux and selectivity for a specific application could be prepared varying the preparation conditions, and that can be done, if the membranes are ordered to specific purposed to serve. In the event, a membrane manufacturer does not produce the relevant polymer material, but has to collect it from local market, the characteristics of the polymer available in the market might not be of same characteristics, but would vary within a certain range. Thus, the optimal membrane could always be prepared, if for each batch of available polymer material, the preparation parameters are synchronized with the requirements. A specialized R&D team in an industry-user could always adjust the membrane preparation parameters to the properties needed for the membranes. They are a number of polymers other than PVC, and that are readily available in the local market. These polymers could also be tested as potential membrane materials. No membrane material is universally sustainable in all water chemistries. An adverse phenomenon like membrane fouling depends mostly on the water chemistry and the nature of particles to be retained. Thus, membranes with similar pore-characteristics, but prepared from different membranes would have different performances and longevity. Thus, technology development for a variety of membranes with different pore-sizes and different membrane material would be very supportive for Bangladesh industries.

4. Conclusion

From the discussion on the primary results obtained from limited experimental database, it can be concluded that micro- and ultra- filtration membranes could be prepared from the technical grade PVC available in the local market. The membranes are with very narrow pore-size distribution, and the mean flow diameter could be varied adjusting the membrane preparation parameters. The experimental results are highly inspiring for developing membranes from a number of polymers available in open market. The present work proves that membrane could be developed in situ in native industries by their own R & D teams, and the technology would be highly sustainable and the membrane-treated product will be of cost compatible with native socio-economic standard.

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