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Exploration of shockwaves on polymeric membrane physical properties and performance

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Abstract. The Commercial polymeric membranes like Polysulfone (PSF), Polyvinylidene difluoride (PVDF) and Polyacrylonitrile (PAN) which are an integral part of water purification investigation were chosen for the shockwave (SW) exposure experiment. These membranes were prepared by blending polymer (wt. %) / DMF (solvent) followed by phase-inversion casting technique. Shockwaves are generated by using Reddy Tube lab module (Table-top Shocktube) with range of pressure (1.5, 2.5 and 5 bar). Understanding the changes in membrane before and after shock wave treatment by parameters, i.e., pure water flux (PWF), rejection (%), porosity, surface roughness (AFM), morphology (SEM) and contact angle which can significantly affect the membrane's performance. Flux values PSf membranes shows increase, 465 (pristine) to 524 (1.5wt%) LMH at 50 Psi pressure and similar enhancement was observed at 100Psi (625 to 696 LMH). Porosity also shows improvement from 73.6% to 76.84% for 15wt% PSf membranes. It was observed that membranes made of polymers such as PAN and PSF (of high w/w %) exhibits some resistance against shockwaves impact and are stable compared to other membranes. Shockwave pressure of up to 1.5 bar was sufficient enough to change properties which are crucial for performance. Membranes exposed to a maximum pressure of 5 bar completely scratched the surface and with minimum pressure of 1.5bar is optimum enough to improve the water flux and other parameters. Initial results proved that SW may be suitable alternative route to minimize/control membrane fouling and improve efficiency.

Keywords: shockwaves; polymer; membrane; fouling; surface roughness; morphology

1. Introduction

Polymeric membrane plays a pivotal role in water pollution control and potable water production around the world (Amy 2008). Due to an increase in demand for potable water and decrease in natural resources stresses the need to develop membrane with long life without decline in efficiency, especially with fouling resistance (Haan *et al.* 2020). Fouling is a universal issue faced by membranologists, technically an unwanted deposit (bio, chemical compounds) on the membrane surface, which will retard the efficiency (Wenshan *et al.* 2012). Fouling control methods aim to decrease the likelihood of membrane fouling; often by pretreatment methods (filters, coagulation, and flocculation) used as a preventive measure for controlling foulants in the feed. Membrane systems vary in design, such as pore size, membrane orientation and various mechanisms used to control fouling (Liu *et al.* 2003). Membrane surface modification may also be performed to reduce affinity between foulants and membrane surface by incorporating nanomaterial or by operating conditions (pH, temperature, pressure and hydrodynamics) (Shen *et al.* 2013). Also applying shear on the membrane surface by gas bubbling, rotating disks/ rotors, rotating membranes and vibratory system are additional viable alternatives to control fouling (Qian 2013). Though effectiveness, scale-up and instrumental cost are major challenges involved. Membrane cleaning processes are required, when fouling control methods fail to remove foulant from the membrane surface (chemical or physical methods). Chemical cleaning involves chemical agents (caustic soda, oxidants, acids, chelates and surfactants) to alter interaction between foulant and membrane surface, which requires large number of chemicals followed by which creates critical safety issues, membrane damage, and generate secondary pollution (Wang *et al.* 2014). Physical cleaning involves application of hydraulic or mechanical forces (hose-pipe, sponge and brush) which require significant physical efforts. Backwash has established as an effective physical cleaning method for flat sheet membranes but unsuitable for other modules due to high-pressure requirement (Qianqian *et al.* 2017). Hydraulic flushing (forward and reverse) also involves with surface deposits removal by solution rinsing and is effective only after pretreated with other cleaning methods (chemical cleaning and backwash) (Katsoufidou *et al.* 2005, Zakariah *et al.* 2016). Ultrasound, an alternative tool for membrane fouling control/cleaning also used mainly involved with ultrasound-assisted mitigation. Different chemical agents have different cleaning efficiencies toward different foulants, combining chemical cleaning agents not advisable process (Clémence *et al.* 2018, Ang *et al.* 2006, Guglielmi *et al.* 2003, Edwin 2007). Li *et al.* reported a new ultrasonic-chemical cleaning system to control organic and

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inorganic fouling (Garcia-Fayos *et al*. 2015, Filloux *et al*. 2015, Li *et al*. 2014, Li *et al*. 2016, Qasim *et al*. 2018, Isabelle *et al*. 2001, Orooji *et al*. 2017, 2018, 2020). In addition to the existing cleaning methods, shock waves (SWs) appear to be an effective tool falls under the physical process category. Shock waves (SW) appear in nature whenever different elements in a fluid approach one another with velocities greater than the local speed of sound (Jagadeesh 2008). SW's are observed during explosions, super-sonic flights and various other processes (bursting a balloon to an exploding star). When a large amount of energy has to be dissipated in a very short time in any mechanical, chemical or nuclear processes shock waves are invariably produced. They need a medium for generation as well as propagation, having the ability to instantaneously enhance the pressure, temperature and density of a medium (in limited space and time) in which they propagate (Chintoo 2014). The ability of SW to instantaneously increase the pressure and temperature in propagation medium enable their use in novel industrial applications. In some sense, the presence of a SW propagating in an enclosed medium can be similar to a furnace where, in addition to temperature, even pressure can go up instantaneously and remain at elevated levels for a short duration (in the order of μs to ms depending on the strength of the shock) and then return to ambient conditions (STP). There is no other method by which you can achieve high pressure and temperature in a medium so rapid than SW. With the large interdisciplinary applications in various field, we attempted to change polymeric membrane's properties with the advantage of using shockwaves. Surface modifications provide vital lead to control membrane fouling by changing properties such as surface roughness, porosity and hydrophobic nature (Shanxue 2017, (Arefi-Oskoui *et al*. 2019, Choi *et al*. 2016). This may be a one major breakthrough in fouling control by combining polymer membranes with suitable shockwaves for improving membrane performance. In the current investigation, we use shockwave energy as a physical treatment technique to observe the membrane properties that would favor in improving performance. Appreciable changes such as an increase in pore size, and increase in pure water flux will enhance membrane performance (Fangang *et al*. 2017). Novel SW technique coupled with membrane materials may be a promising future for the wastewater treatment and separation applications in fouling resistance.

2. Materials and methods

Polysulfone (PSf) pellet (Udel P-3500, Solvey Advanced Polymers, USA), Polyvinylidene fluoride (PVDF), trade name SOLEF 1015/1001 (density1.78 g mL−1) Solvay Advanced Polymer (Brussels, Belgium). Polyacrylonitrile (PAN, MW. -160 kDa, IPCL, Vadodara, India) used as received. Non-Woven polyester fabric (Filtration Sciences Corp., USA), N,N dimethylformamide >99% (DMF) (Merck, India), Sodium lauryl sulphate (SLS,SD fine chemicals, India), Polyethylene Oxide (Mw 200kDa) and Bovine serum albumin >98% (BSA) (Mw 66kDa) (Sigma-Aldrich, USA) was used in membrane fabrication and characterization process. Milli-Q water was used in the experiments.

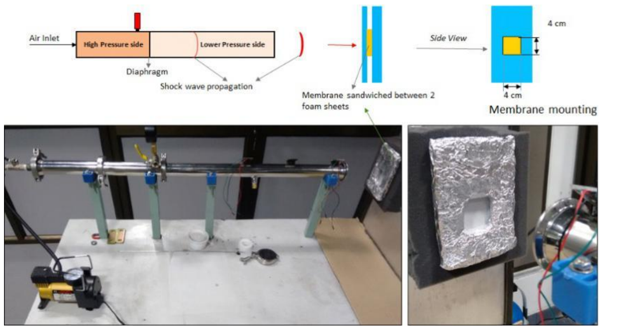


Fig. 1 Reddy Shocktube experimental module used for shockwave exposure at Physical Research Lab (PRL, Ahmedabad), India

2.1 Flat sheet membrane fabrication by phase-inversion technique and characterization

Polysulfone (PSf) (15, 20, 24 wt. %), PVDF (14wt. %) and PAN (20wt. %) in DMF was prepared by stirring coupled heating for homogenous polymer solution used in membrane casting process. The polymer solution kept at ambient condition for sufficient time to remove air bubbles before casting. Polymer/ DMF solution were casted on non-woven polyester fabric fitted on glass plate and then immediately immersed in gelation bath under controlled conditions. Membranes were washed and stored in deionized water for further investigations (pure water flux, rejection, contact angle, porosity, surface morphology (SEM, AFM)). Pristine and SW exposed membrane morphology visualized by Scanning Electron Microscope (Leo, 1430UP, Oxford Instruments), surface roughness (Atomic force microscopy NT-MDT instrument) and contact angle (water) measured by DSA100Kruss GmbH instrument.

2.2 Shock wave experimental module

In the laboratory, shock waves (SW) are produced using shock tubes by a sudden expulsion of flow from one region to another (Fig. 1). A gas driven Reddy shock tube (Reddy *et al*. 2013) was used for generating SW of peak overpressure of up to 5 bar. It consists of a driver section and driven section separated by a diaphragm made of tracing paper of thickness 100 μm. The intensity of the shock waves generated can be increased by using multiple layers of the diaphragm so as to hold more pressure before rupture (Cioanta 2017). The rupture of the diaphragm at a certain pressure results in a sudden expulsion of flow into the driven section of lower pressure resulting in the generation of shock waves. The driver side is pressurized using an air pump and when the diaphragm ruptures at a certain pressure, a shock wave is generated and propagates along the driven side and impinges on the membrane surface. SW experiments were conducted on various peak-pressures on polymeric membrane using a table-top

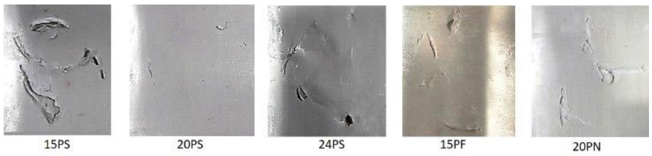
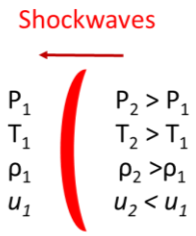


Fig. 2 Damaged membranes (visible physical changes) on the surface after applying shockwaves of ≥5 bar



u- Velocity of flow;
P - Pressure;
T - Temperature;
ρ - Density;
h - Enthalpy;
s - Entropy;
M - Mach Number;
M = Velocity of flow in a medium/Velocity of sound in the same medium.

Fig. 3 Schematic representation of change in properties of the medium across the shock wave

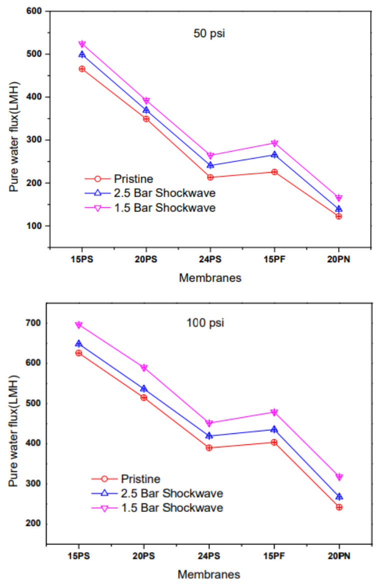


Fig. 4 Pure water permeability (PWP) of pristine and shockwave exposed polymer membranes

shocktube system.

Membrane sample of size 4 cm×4 cm are mounted on Reddy shock tube for generating shock wave for all the experiments. Considering lab safety operations and the risk involved with high pressure, we restricted the pressure range minimum by using a miniaturized shock tube available that can generate SWs of peak overpressure up to 5 bars. With the available facility 3 types or peak pressure of 1.5 bar (single diaphragm), 2.5 bar (double layer), 5.0 bar (Three layers) were explored. Shockwaves of peak pressure (1.5, 2.5 and 5 bars) were applied based on the number of diaphragms to hold the pressure and release, thus generating the shockwaves. It was observed that the membranes were completely damaged at a peak-overpressure value of ≥5 bar generated by tube. At ≥5 bar the membrane is getting damaged due to the impact of diaphragm material and not suitable for further analysis. Membrane samples before and after shockwave exposure were sent for further analysis to determine the following changes: pure water flux, rejection percentage, surface roughness, contact angle and porosity.

3. Results and discussion

3.1 Effect of shockwave on membrane surface

Membranes before and after shockwave exposure were displayed in (Figs. 2 and 3). Effect of shock wave exposure on membrane surface is a novel option to analyze the effects (visible surface and interior effects).

The shockwave generated in the experimental tube increases pressure of the medium in its path instantaneously as it propagates with a pressure jump of 1.5 bars as measured (lasting for 250-350 μs). During the experiment, SW was generated by filling gas (feed from external source) inside the driver side which is separated from the driven side by a diaphragm which ruptures at a static pressure of 2.5 bar (rupture pressure) but the shockwave thus generated imparts a pressure jump of 1.5 bar-equivalent on the membrane surface. When the rupture pressure is 4 bars, the generated SW imparts a pressure jump of 2.5 bars to the membrane. At a rupture pressure of 6 bar, the generated shockwave imparts a pressure of 5 bar which results in damaging the membrane. It was observed that the maximum changes have occurred at a pressure of 1.5 bars, then at 2.5 bar or 5.0 bar which requires more power or more volume of gas to fill the driver before the rupture which is a measure of enthalpy of the system (Fig. 4). Thus, it is effective to enhance membrane performance at a lower enthalpy value which will define the energy requirement for running a system and related maintenance cost. Considering the complete process time (μs) and energy required to develop the SW effect on membrane surface altogether provide an alternate route to monitor and change the membrane surface which is vital for fouling control. It was observed that membrane surface damage generated from broken diaphragm pieces and not from shock waves. We optimized the pressure 1.5 and 2.5 bar for the current investigation to understand membrane surface changes with these low-pressure exposures (Bartman *et al*. 2011).

3.2 Membrane performance with shockwave pressure variation

Effect of SW on pure water flux clearly observed from the values, 1.5 bar SW exposed membranes exhibits superiority at both 50 and 100 Psi range. Compared with pristine membrane, shockwave (physical forces) does have certain impact on membrane surface that is substantiated by the pure water flux performance (Cen *et al.* 2015). Rejection percentage (%) or Molecular weight cut off (MWCO) tested using the macromolecule polyethylene oxide (PEO Mw 100kDa) by cross-flow filtration system at 50,100 Psi pressure using and samples (Fig. 6) were analyzed by HPLC-GPC Waters, 2695 module 241 (Eric *et al*. 2001).

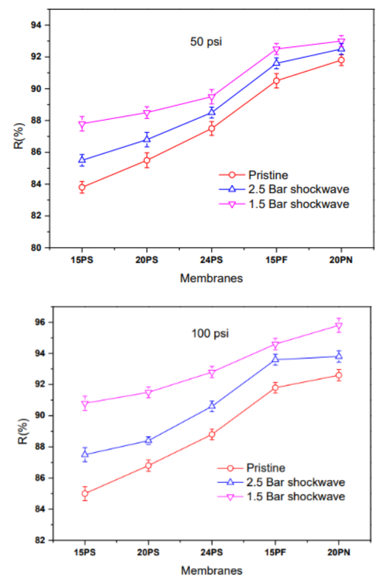


Fig. 5 Rejection (*R* %) of pristine and shockwave exposed polymer membranes

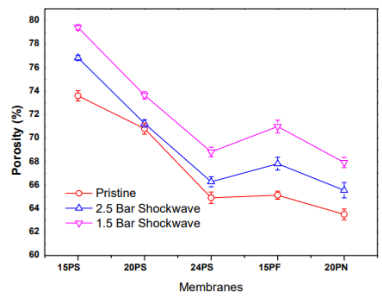


Fig. 6 Porosity of pristine and shockwave exposed polymer membranes

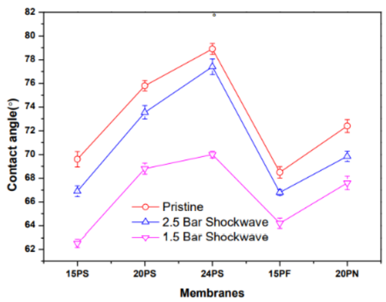


Fig. 7 Contact angle of pristine and shockwave exposed polymer membranes

For porosity measurements, pristine and SW exposed membranes were cut with an area of 4×4 cm soaked in water and after 24h membranes were removed and wiped with tissue paper to remove moisture adsorbed on the surface (Creber *et al*. 2010). Weight was measured and then dried in an oven at 70°C and after reaching constant weight was noted again (Fig. 7).

Wet and dry weights were analyzed using the following equation

$$\text{Porosity (\%)} = \frac{W_{wet} - W_{dry}}{A \times d \times \rho} \times 100 \qquad (1)$$

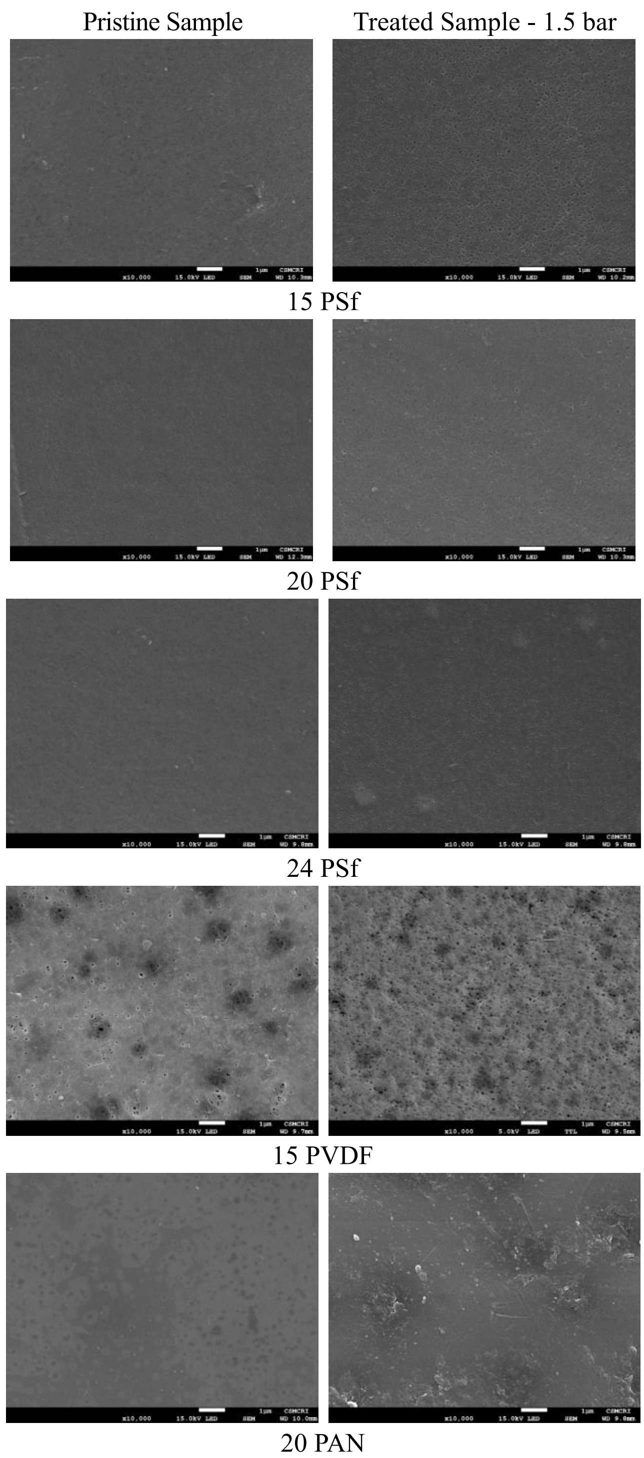


Fig. 8 (A) SEM micrographs-Top view

Wwet (membrane weight at wet condition) and *Wdry* (membrane weight at dry condition), *A* is the membrane area, *ρ* is the density of water and *d* is membranes thickness. Porosity before and after *SW* exposure were plotted which will give a clear indication about the pore opening process when the membrane surface exposed to various pressure range of shock waves. It is a good indicator that with minimal energy (*SW*) of 1.5 bar is sufficient enough to improve porosity. Contact angle of membranes before and after *SW* exposure reveals some

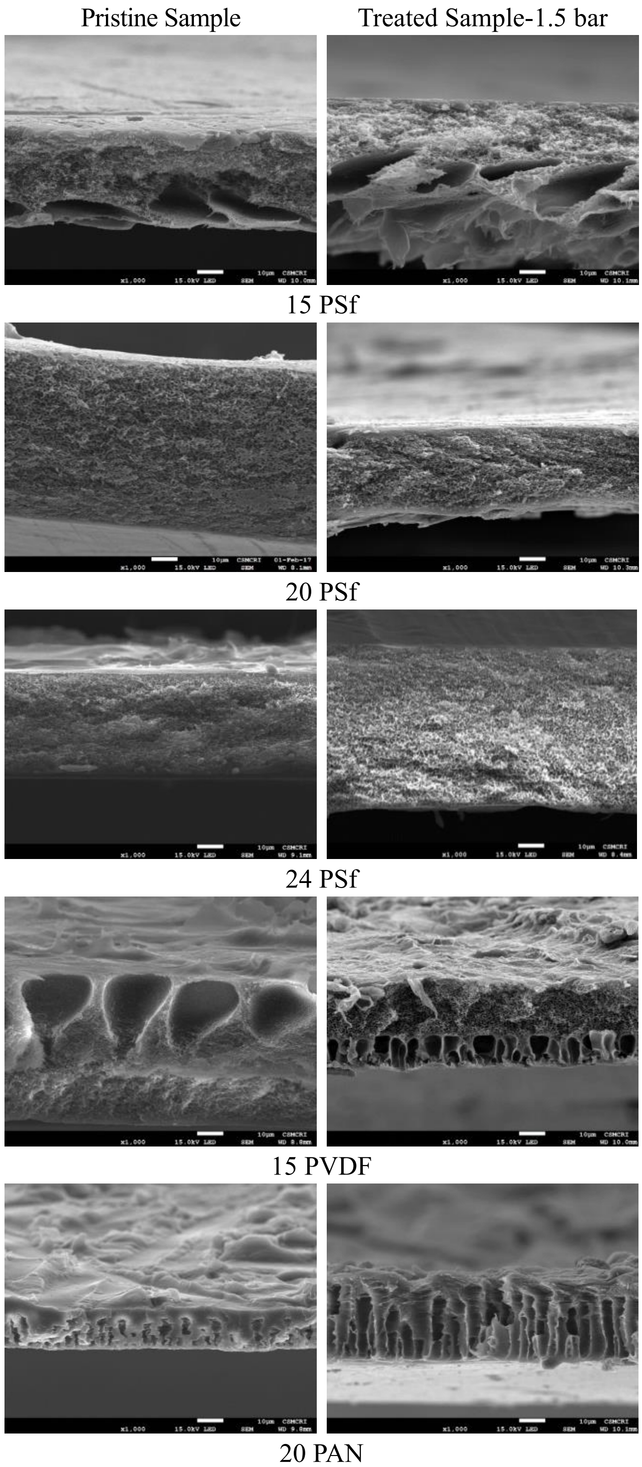


Fig. 8 (B) Cross-section view of polymer membranes (Pristine and SW exposed @1.5bar)

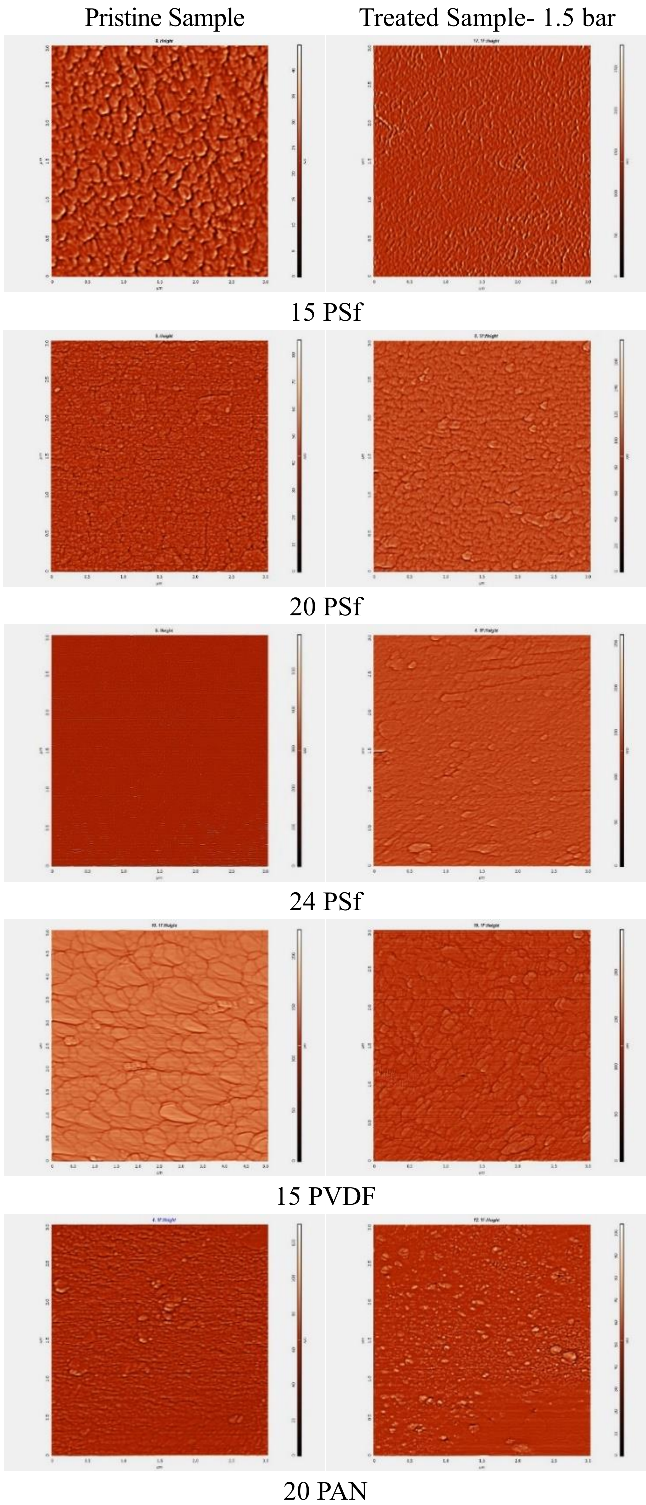


Fig. 9 (A) AFM 2D images

changes in hydrophobicity, specifically values slightly decreases after *SW* treatment process, may be due to physical turbulence created by the pressure. To further support the surface changes by *SEM* and *AFM* images, after shockwave treatment membrane surface pores are more visible (*PSf*) than pristine surface, whereas *PVDF* shows more open surface structure and *PAN* retains the original surface morphology (Kim *et al*. 2014, Creber *et al*. 2010b).

The cross-section image confirms that shock wave of 1.5 bar applied substantially modified the surface without affecting the cross-sectional morphology (Fig. 8, 8(b)). Surface roughness values obtained from AFM images for membranes before and after shockwave treatments, which clearly confirms the surface changes developed by shockwaves (Fig. 9(a), 9(b)).

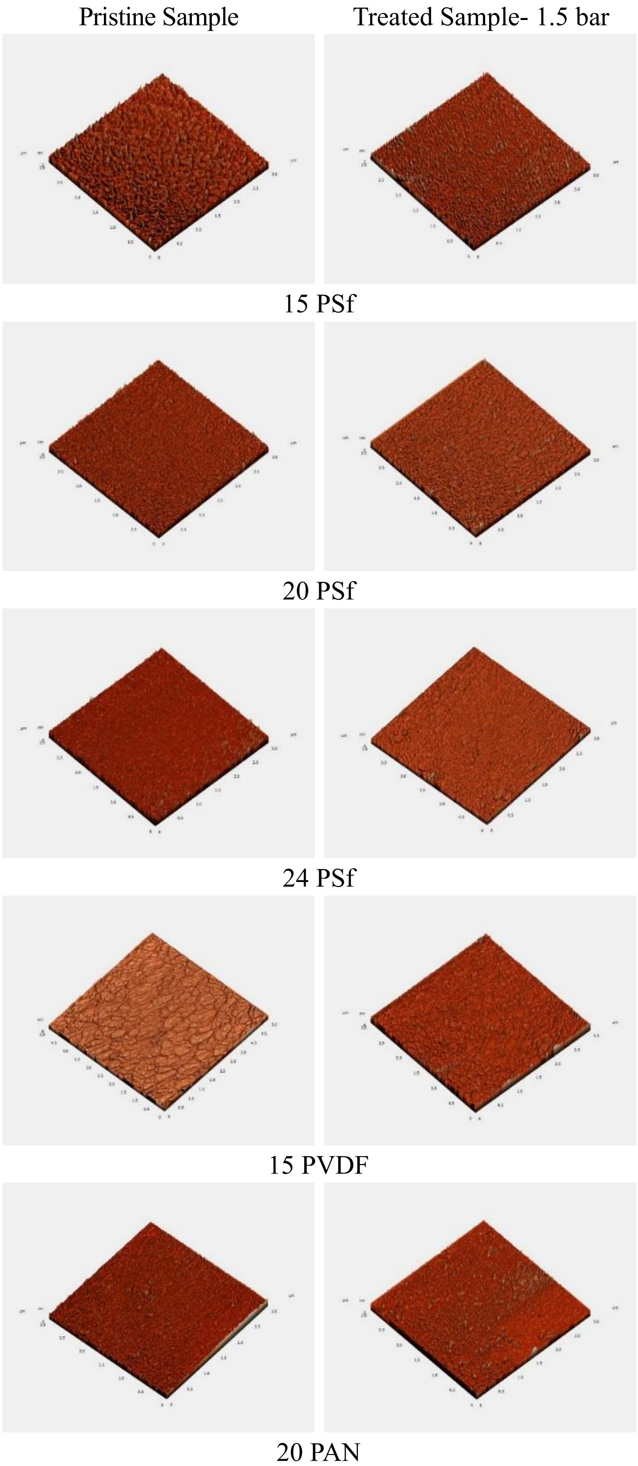


Fig. 9 (B) AFM 3D images of polymer membranes before and after shockwave exposed @1.5 bar

4. Conclusions

Shockwave (SW) experiments were conducted using a table-top shocktube to explore surface morphologies of PSf, PVDF and PAN flat sheet membranes. From the preliminary results it was observed that minimal use of energy (pressure 1.5 bar) was sufficient enough to impact membrane surface and efficiency, and beyond this pressure it does not guarantee better values and damage the membranes. Considering cost economics and energy involved in the process, we restricted our analysis with 2.5 bar maximum, because very low pressure provides the expected outcome. Porosity values increase from 74 to 80% for 15 wt. % PSf membrane and similar enhancement were observed in all types of membranes investigated. Membrane's performance after shockwave exposure gave vital information about membrane properties which may be useful to overcome the membrane fouling effect and determine efficient ways to treat and reuse fouled membranes. This may be one major breakthrough in surface modification, membrane performance by combining polymer membranes with suitable shockwaves for possibly minimizing fouling effects (removal/rupture of biofilms). Initial results are promising enough to explore the area in detail for further investigation. Based on these encouraging results with usage of minimum energy, which paves way for novel treatment tool in membrane fouling issues. it is possible to modify and enhance membrane surface, morphology. Encouraging flux performance supports us to carry further biological fouling analysis by simulating conditions. SW technique can be extended to bulk level after executing required energy and cost-economics involved.

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