THE ANNALS OF "DUNAREA DE JOS" UNIVERSITY OF GALATI
FASCICLE IX. METALLURGY AND MATERIALS SCIENCE
N°. 3 - 2020, ISSN 2668-4748; e-ISSN 2668-4756
Article DOI: https://doi.org/10.35219/mms.2020.3.01

UTILIZATION OF SMARTPHONE LIGHT SENSORS AS LIGHT TRANSMISSION ANALYZER DURING THE OBTAINING OF POLYMERIC PHASE INVERSION MEMBRANES

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ABSTRACT

Smartphone sensors are gaining research interest due to continuous sensor upgrades, leading to more precise readings of these sensors. For this study, the light sensor of a smartphone was used in determining light transmittance during the phase inversion process of ultrafiltration polysulfone-membrane manufacturing. Membrane separation is one of the best available technologies when it comes to water and wastewater treatment. The purpose of this study was to correlate light transmittance, at certain demixing steps during phase inversion, with membrane porosity, pure water flux and cross-sectional SEM images. Results show close relation between light transmittance and the mentioned membrane properties.

KEYWORDS: membrane, flux, porosity, light transmittance, smartphone, light sensor

1. Introduction

Filtration using membranes have started gaining interest since the year 1907 [1]. Unfortunately, it was not until 6 decades later that membranes showed enormous potential in filtration-based applications, especially from a commercial point of view [2]. Since then, due to the efficiency of separation and lower energy consumption compared with conventional treatment methods, the membrane technology has been implemented in almost all sectors of industry that use water as a prime material and process water.

According to the International Union of Pure and Applied Chemistry, a membrane is defined as a porous or dense structure, with lateral dimensions much greater than its thickness that acts as a selective barrier between two phases, through which transfer occurs under a variety of driving forces [3].

Pressure-driven membranes are among the most used types of membrane filters [4]. Polymeric membranes cover 90% of the produced membranes sum total scale [5]. The most common method used to prepare polymeric membranes is the phase inversion process [6].

The phase inversion is a very simple process that consists in the exchange between solvent and non-solvent in the polymeric matrix, often called demixing. It is a process that covers a wide range of different techniques, the most used being immersion precipitation [7].

Different techniques are used in order to understand the membrane performance, structure and formation. Although light transmission is an acceptable method in studying membrane formation during phase inversion, little emphasis was laid emphasis was placed on using it in membrane research [6-10].

Smartphones are becoming an important tool in laboratory-related experiments, for teaching and scientific purposes [11-15], primarily due to their built-in sensors. Examples of such sensors are: the accelerometer, magnetometer, ambient light sensor, GPS, microphone, barometer, thermometer, air humidity and, in very rare situations, the Geiger counter [16].

As sensors are starting to become smaller and more performant, they are gradually transforming smartphones in devices that could replace some laboratory equipment. The most advantageous aspect in using smartphones in such means is mainly related to costs and multifunctionality.

The purpose of this study is to verify whether the smartphone sensor is a good way to measure light transmittance of a polymeric thin film during phase inversion, in order to observe the different stages of polymerization, for ultrafiltration membranes composed of 3 polymer concentrations (polysulfone).

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From the best of our knowledge, this technique has never been used before.

2. Materials and methods

2.1. Membrane materials and preparation

2.1.1. Membrane materials

The polymer of choice was polysulfone (PSf, Mw ~35000), and the solvent was 1-methyl-2-pyrrolidinone (NMP, C_5H_9NO, M_w ~99.13, ACS reagent grade: 99%). Both components were purchased from Sigma Aldrich.

2.1.2. Membrane preparation

The studied membranes were manufactured by phase inversion, the immersion precipitation technique. The cast solution was obtained by mixing NMP solvent with PSf polymer of different concentration.

The solutions were stirred at 1000 rpm until homogeneity was achieved. After that, the solutions were casted on a glass support, and with the help of a casting knife, the solution was casted to a fixed thickness of 250 μm. In order to produce the actual membrane, the thin film solution was immersed in a coagulation bath, composed of distilled water. Here, phase inversion takes place, leading to the demixing between the pure water and the NMP. During demixing, pores are produced in the polymeric matrix. During this procedure, light transmission analysis will be investigated. After complete demixing, phase inversion ends and the membranes are transferred in a distilled water container in order to dissolve the remaining NMP residue.

The resulted membranes were labelled according to PSf concentration, as presented in Table 1.

Table 1. *Membrane designation according to polymer concentration*

Membrane	Polymer type	Polymer conc. [wt.%]
23PSf	Polysulfone (PSf)	23
25PSf		25
27PSf		27

2.2. Methods of characterization

2.2.1. Light transmission set-up

The set-up for light transmission tests was as shown in Figure 1, which is a homebuilt device. The principle is simple, consisting in an emitting visible light beam which passes through the casted polymer solution during the whole procedure of phase inversion. This beam is recorded as light intensity [lux], being afterwards transformed in transmittance percentage.

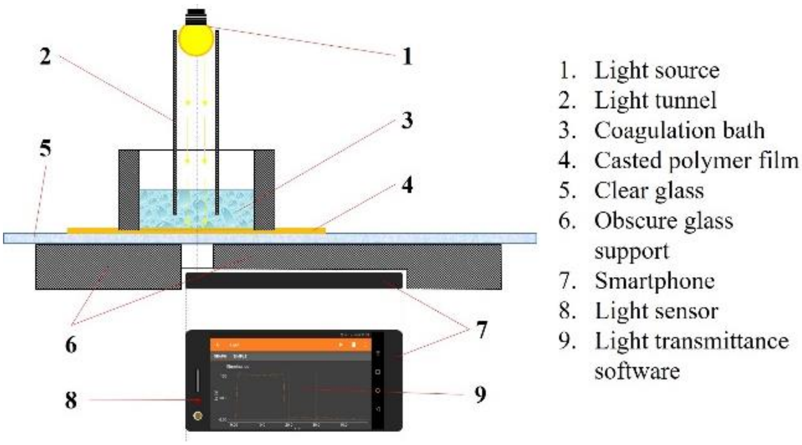

Fig. 1. *Light transmittance set-up for membrane formation monitorization*

The light sensor (TCS3701) is an integrated part of a smartphone device (SM-A705FD), which is positioned under the casted polymeric film. The data received from the sensor were recorded with a special open-source software (Phyphox, RWTH Aachen University). Some technical specifications of the TCS3701 light sensor are presented in Table 2.

Table 2. *TCS3701 sensor features*

Minimum detectable illuminance: 1 mlux (100 ms
UV/IR blocking filter
14-bit data output
Maximum range (lux): 32657.0
Resolution: 1.0

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The recorded values were exposed as light transmittance vs. time curves.

2.2.2. Porosity

The light transmission results were correlated with the membrane porosity. In order to calculate porosity, a membrane sample with a known surface (A) and thickness (h) was weighed in dry state (m_{dry}) and afterwards immersed in a liquid for at least 24h to make sure that the sample is fully wetted (m_{wet}). The liquid of choice is distilled water, which has a specific density ρ.

$$\varepsilon = \frac{m_{wet} - m_{dry}}{A \times \rho \times h} \quad (1)$$

In simple terms, porosity represents the total spaces from inside the structure of a membrane. Porosity is linked to permeation properties.

2.2.3. Pure water flux

The pure water flux, J [L m^{-2} h^{-1}], expresses the permeation performance in ideal circumstances, where the feed liquid has no impurities in its composition. In this study, pure water was used for convenience.

The flux is calculated as follows:

$$J = \frac{V}{A \times t} \quad (2)$$

Where V is the total volume [L] that passed through a known membrane area, A [m^2], within a set time, t [h].

2.2.4. Cross-section SEM microscopy

The sample cross-section morphologies were investigated using FEI Quanta 200 scanning electron microscope. All samples were sputter-coated with gold before observation to obtain conductivity during analysis.

3. Results and discussions

After the transmittance experiments of thin films during the phase inversion process, as can be seen in Figure 2, several stages can be identified. Four main stages were identified during phase inversion process. The first stage represents the instant demixing, which is the first contact between the polymeric solution film and coagulation bath. This stage is observed as a steep slope, denoting the very fast water-solvent demixing. The second stage is represented by the start in stabilization of the aforementioned demixing. Stage III will not be discussed because he fact that this step is a constant polymerization slope until full demixing is achieved. The complete solidification is reached at the last stage, where the membrane is fully polymerized.

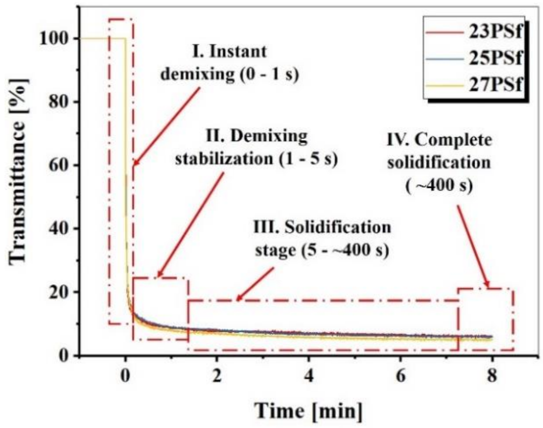

Fig. 2. *Overall light transmittances during phase inversion of the studied membranes*

It will be observed that stages I and IV are linked to the overall properties of membranes.

Although the overall graph is expressed in minutes, the other graphs will be expressed in seconds due to the fast demixing of the polymer film during phase inversion.

Every stage in light transmission is linked to special areas of the membranes cross-section, for all the studied membranes. In Figure 3 one can observe that, throughout the demixing direction, the width of the elongated pores, called macrovoids, is gradually increasing.

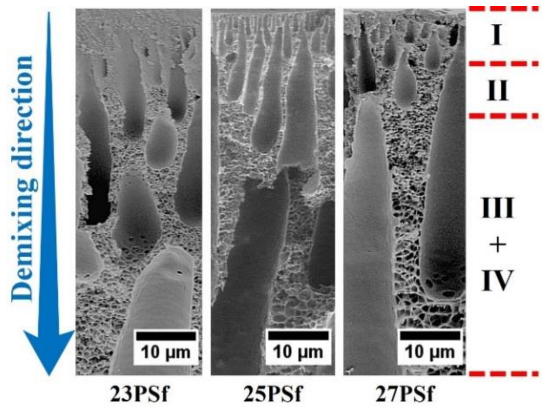

Fig. 3. *Cross-section SEM images of the studied membranes and the possible hypothesis with the regions where the four stages may occur*

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Based on width increase, the positioning of the four stages can be estimated. Hence, the region where the smallest macrovoids occur is at the top of the cross-section, noted by stage I of the demixing process. As the region progresses downward, it is observed that in the other stages the thickness of macrovoids gradually increases, meaning a slowing down of the demixing momentum.

In the first stage (Figure 4), the reactions between solvent and non-solvent take place at the membrane surface, which is considered as the most important stage in membrane fabrication. This happens because it establishes the permeation properties of the membrane, higher or lower flux and retention, also.

A lower transmittance during the first second of phase inversion means a fast demixing of the solvent with the water bath, which is directly proportional with the pore formation in the membrane active layer.

The sample corresponding to membrane 23PSf, the lowest polysulfone concentration, displayed the lowest light transmittance, which is indicative of fastest solvent-water demixing.

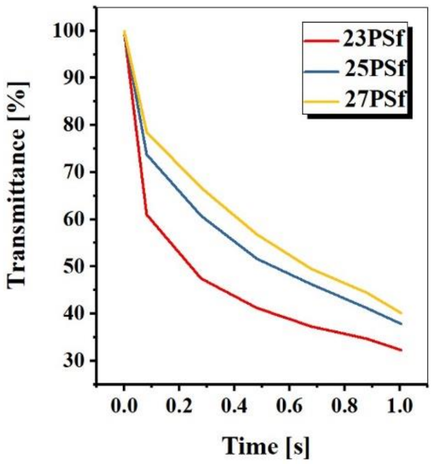

Fig. 4. *Instant demixing stage during phase inversion of the studied membranes*

Comparing transmittance with membrane structure, it is observed that with fast demixing the pore formation at membrane surface is thinner, difficult to distinguish (Figure 3 – 23PSf), while slower demixing led to observable thicker active layers (Figure 3 – 25PSf and 27PSf).

Continuing the reading to the interval of 1 to 5 seconds (Figure 5), the ability of the middle morphologies of membrane sections to stabilize is observed. Stage two shows the stabilization time of the membrane. The membrane with 27 wt% PSf show a faster demixing which result in a thinner middle layer.

Also, in stage II, the light transmittances are observed to stabilize and keep the trend up to the end of phase inversion.

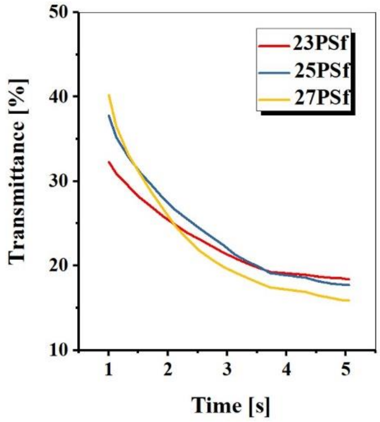

Fig. 5. *Demixing stabilization stage during phase inversion of the studied membranes*

In the final stage of the phase inversion process (Figure 6), after approx. 8 minutes, the transmittance was observed to be higher for lower polymer concentration. This phenomenon is strongly related with membrane porosity. The higher porosity leads to higher passage of light through the membrane, due to the higher frequency of macrovoids in structure.

Higher polymer concentration membranes, which is understood as the samples with lower amount of macrovoids, led to lower light transmittance.

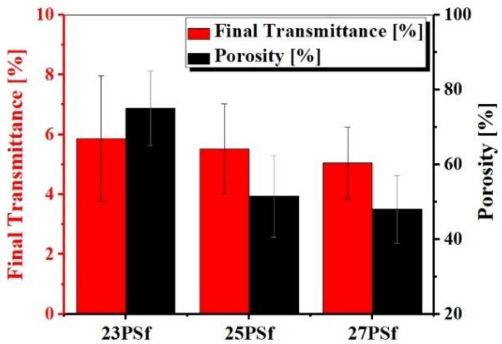

Fig. 6. *Complete stabilization stage in correlation with calculated porosities of the studied membranes*

As explained in the first stage of phase inversion, demixing is related with pore formation at membrane surface. The demixing time is directly proportional to pore formation, in which a faster demixing will lead to a higher flux due to higher number of pores.

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As can be observed in Figure 7, a faster demixing lead to higher water flux, represented by the 23wt% PSf membrane.

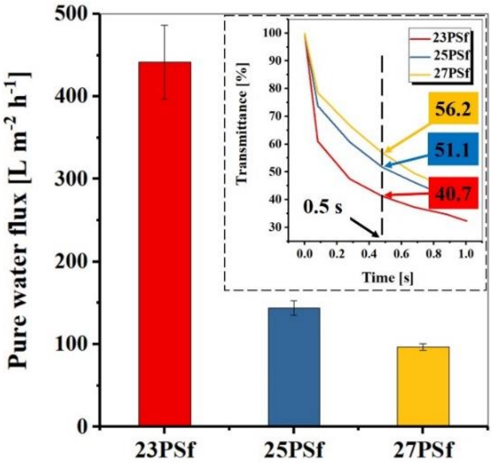

Fig. 7. *Pure water flux correlated with the transmittance occurred at 0.5 s at the stage of instant demixing (extracted from Figure 4) of the prepared membranes*

The values of the light transmittance at time of 0.5 s were extracted from Figure 4, also present in Figure 7 as an insert graph. It is obvious that the light transmittance-flux trend is inversely proportional. The highest difference, in terms of light transmittance, is between 23PSf (40.7%) and 27PSf (56.2%), respecting the pure water flux trend, also. On the other hand, the lowest difference is between the membranes with higher polymer concentration, namely 25PSf and 27PSf, with 51.1% and 56.2%, respectively.

Demixing is inversely proportional to water flux and directly proportional to polymer concentration.

4. Conclusions

Membrane characterization is very important for understanding future performances of the material. Light transmission is a good tool in determining several membrane properties and performances, before they can be used in the actual separation processes. The pure water flux and porosity were successfully correlated with the light transmittances obtained by the smartphone light sensor. The results were synchronous, showing similar trends at different polysulfone concentrations. In the near future, smartphones will become essential devices in lab research.

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