FABRICATION OF FOLDING POLYMERIC THIN FILMS WITH HYDROPHILIC PROPERTIES

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Polydimethylsiloxane (PDMS) sheets were used as a mold for the generation of low cost polymeric thin films. Poly(ethylene glycol) diacrylate average Mn 575, Poly(ethylene glycol) diacrylate average Mn 250 and Phenylbis (2, 4, 6-trimethylbenzoyl) phosphine oxide were used to create hydrophilic and hydrophobic thin films. The thickness of the films was highly dependent on the amount of the polymeric mixture and the pressure between the PDMS sheets. The hydrophobicity of the films was measured by observing the contact angle of a water drop on the film, as well as the degrees of folding when the film absorbed humidity. The films produced with Poly(ethylene glycol) diacrylate average Mn 575 were found to be hydrophobic, while the films produced with Poly(ethylene glycol) diacrylate average Mn 250 were found to be hydrophilic. Hydrophobic films fold when exposed to a drop of water on their surface. The time and the angle of folding were directly dependent on the thickness of the film. A relation between the relative mass of the thin film and the angle of folding was found.

Keywords: contact angle; polymeric mixture; humidity absorption.

1 INTRODUCTION

The manufacture of synthetic membranes has lately become a growing interest due to its multiple applications in covering surfaces and separation processes, among others. Synthetic membranes can be classified in accordance to their material into inorganic thin films (e.g. ceramics) and organic thin films (e.g. polymers) [1] . Among these, polymeric thin films have been preferred due to their relatively simple fabrication and low cost [2]. In the same way, polymer-based films can be categorized in accordance to their capacity to absorb or repel water as hydrophilic or hydrophobic [3]. Many studies are being made upon how to change the hydrophilic characteristics of polymeric membranes in order to overcome problems as fouling and permeability [2, 4-6]

The hydrophobic and hydrophilic characteristics of a particular material are a way of determining its wetting phenomena. Surface chemistry is often applied to determine the wetting behavior of a surface and the ways it can be modified [2, 3, 5, 7]. Short-ranged interactions such as van der Waals and electrostatic forces will determine whether or not a fluid wets a given surface [8]. When a droplet rests upon a solid surface, three interfaces are exhibit; solid-liquid, liquid-gas and solid-gas. The interactions between these ones raise three surface tensions that generate what is known as the contact angle [9]. If the three phases in the system (solid, liquid and gas) are in mechanical and chemical equilibrium (i.e. exhibit a force balance and there is a chemical potential matching for all of the present components) and there is a thermal

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equilibrium between the liquid and gas phases (i.e. there is a temperature matching between the liquid and gas phases so that the gas phase represents the saturated vapour of the liquid), the system is said to be in thermodynamic equilibrium [8] and therefore, it is represented by Young's relationship [9], which is shown in Equation 1.

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV}\cos(\theta_{eq}) \quad (1)$$

where γ_{SV}, γ_{SL} and γ_{LV} represent the solid-vapor, solid-liquid and liquid-vapor surface tensions respectively.

Theoretically, the shape of a droplet resting upon a solid surface is determined by the surface tensions acting on the liquid. However, in practice, the drop is often deformed by external forces such as gravity and, therefore, the contact angle is determined by a combination of both surface tension and inertial forces [10]. In order to know the ratio in which inertial and surface forces affect a drop of a particular liquid over a particular surface, the Weber number should be calculated. If the characteristic Weber number is > 1, inertial forces dominate over surface tension forces. If the characteristic Weber number is < 1, surface tension forces dominate over inertial forces, and therefore, Young's equation may be applied [9].

The equilibrium contact angle can then be associated to the wetting characteristics. Small contact angles, < 90°, describe high wettability and are related to hydrophilic surfaces, while large contact angles, > 90°, describe low wettability and are often related to hydrophobic surfaces. The range of how far the measured angle is from the 90° boundary will determine if it can be classified as a superhydrophilic or superhydrophobic surface [10].

In the current study, two sets of polymeric thin films were fabricated by ultraviolet A (UVA) polymerization using Poly(ethylene glycol) diacrylate average Mn 575 and Poly(ethylene glycol) diacrylate average Mn 250 respectively, with Phenylbis (2, 4, 6-trimethylbenzoyl) phosphine oxide as a photoinitiator. All the manufactured thin films were characterized in terms of their hydrophilic or hydrophobic behavior by means of their contact angle with a drop of water. The second set of thin films was found to be sensible to water due to an immediate folding reaction after the droplet touches the surface. A characterization study of the folding parameters such as folding angle, folding time and recovery time was performed. Further liquids, organic and inorganic, such as ethanol C_2H_6O, dichloromethane CH_2Cl_2, hydrochloric acid HCl, ammonia NH_3, acetic acid $C_2H_4O_2$, linseed oil and hydrogen peroxide H_2O_2, were also dropped on both sets of membranes to observe any possible reaction.

2 MATERIALS AND METHODS

2.1 POLYMERIC THIN FILMS

Thin films were generated using Poly(ethylene glycol) diacrylate average Mn 575, Poly(ethylene glycol) diacrylate average Mn 250 and Phenylbis (2, 4, 6-trimethyl benzoyl) phosphine oxide provided by Sigma-Aldrich®. Two types of solutions were studied. Solution A was made with 98mL of Poly(ethylene glycol) diacrylate average Mn 250 and 2g of Phenylbis (2, 4, 6-trimethylben-zoyl). Solution B was made with 98mL of Poly(ethylene glycol) diacrylate average Mn 575 and 2g of Phenylbis (2, 4, 6-trimethylbenzoyl).

A 5mL syringe was used to place 4, 6 or 8 drops of the desired solution between two polydimethylsiloxane (PDMS) sheets attached to a glass substrate as shown in Figure 1. No external pressure in addition to the upper PDMS sheet weight was exerted. The entire system was exposed to UVA radiation for 1 minute.

Figure 1 - Diagram of the thin films fabrication process.

The superficial area was set to 25mmx25mm for all created thin films. 3 different thicknesses, corresponding to 4, 6 and 8 drops of solution, were fabricated for each solution. 10 different thin films were obtained per thickness. A total of 60 thin films were studied.

2.2 EXPERIMENTAL SET-UP

Water drops were generated using a 5mL syringe (gauge 21, 38mm). The drops were released from a fixed height of 114.5 mm above the thin film surface. Repeated tests were made to determine the drop weight, which was found to be approximately 39.15mg. The characteristic drop diameter was calculated using Equation 2.

$$D_0 = \left(\frac{6m}{\pi r}\right)^{\frac{1}{3}} \quad (2)$$

where *m* is the drop mass.

The characteristic diameter was found to be 3.41 $\pm$ 0.1mm. This value was used to calculate the free fall velocity of the droplet and the Weber number associated to it according to Equations 3 and 4.

$$v = \sqrt{2g(H - D_0)} \quad (3)$$

$$We = \frac{\rho v^2 D_0}{\sigma} \quad (4)$$

where H is the height of release of the drop of water, ρ is the density of water and σ is the surface tension.

The obtained Weber number was 0.5287 $\pm$ 0.001, which means that surface forces dominate over inertial forces and, therefore, the shape of the drop of water on the surface can be assumed spherical.

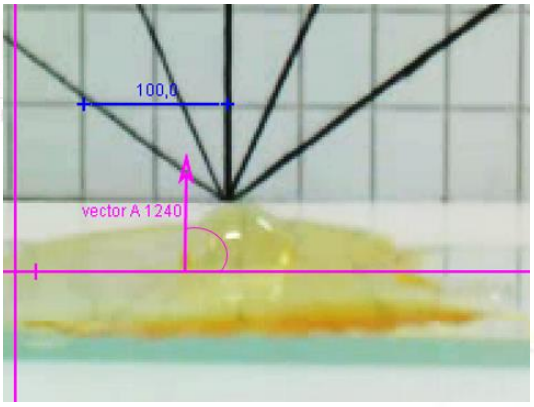
A digital camera was set horizontally aligned to the surface of the thin films and was used to record the impact and consequences of the water drop. The camera position, magnification and the thin film position were kept constant through all the experiments. A known length was used for calibration and forward data processing.

All 60 thin films were weighted using a ML 204 analytic balance from Mettler Toledo.

2.3 CONTACT ANGLE MEASUREMENT

Equilibrium contact angles were determined by video processing using the software Tracker 4.90. The contact angle of the sessile drop was measured in accordance to Young's equation as illustrated in Figure 2, where the contact angle is determined as the angle formed between the solid-liquid and the liquid-vapor interfaces. Young's model was assumed due to the fact that the thin films were characterized as a solid homogeneous system in thermodynamic equilibrium [8].

Figure 2 - Data processing for determining the equilibrium contact angle using the software Tracker 4.90.

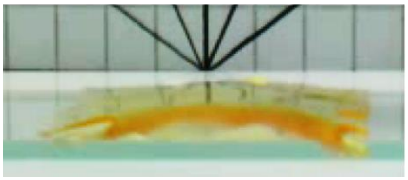


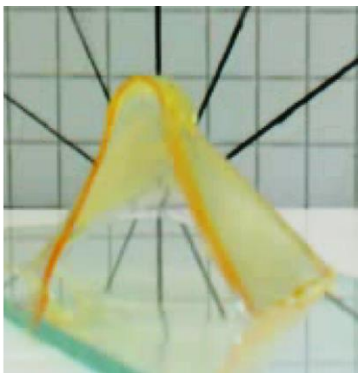
As shown in Figure 1, vector A818 represents the liquid-vapor interface, while the x axis represents the solid-liquid interface. A 100mm calibration stick is used.

2.4 FOLDING ANGLE, FOLDING TIME AND RECOVERY TIME FOR SOLUTION B THIN FILMS

When in contact with the drop of water, solution B thin films presented a mechanical reaction shown in Figure 3. In order to characterize their properties, the folding and recovery time were measured. Folding time was determined from the instant of contact between the droplet and the film's surface to the point in which the system reached mechanical equilibrium. Recovery time was measured from the moment when the drop of water was removed from the surface of the film till it returned completely to its initial position. Measurements were obtained using the software Tacker 4.90. A study of the maximum folding angle of solution B thin films was also conducted.

Figure 3 - Initial (left) and final (right) position of solution B thin films when exposed to a drop of water on their surface.





4 RESULTS AND DISCUSSION

4.1 THIN FILMS CHARACTERISTIC PROPERTIES

All thin films were characterized in terms of surface dimensions, mass and surface aspect before being exposed to water. Descriptive statistics of the mass are shown in Table 1 and surface dimensions descriptive statistics are shown in Table 2.

Table 1 - Descriptive Statistic Indicators of the Thin Film's Mass Before Water Exposure

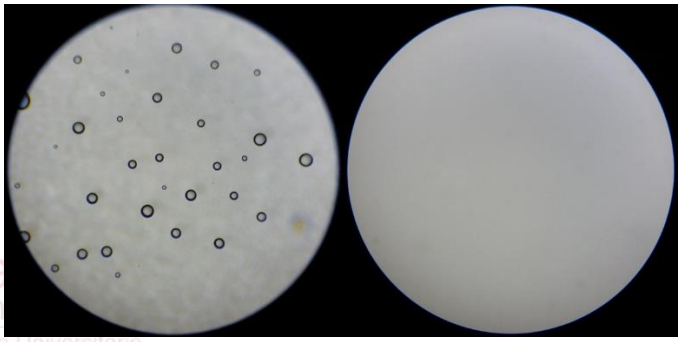
Mass (mg)						
Statistic Indicators	Solution A			Solution B		
	4	6	8	4	6	8
Mean (μ)	92.1	121.6	205.1	117.74	156.34	216.33
Standard deviation (s)	12.1	10.8	11.7	24.01	29.81	35.65
Variance (σ^2)	146.41	116.64	136.89	576.71	888.82	1270.99

Table 2 - Descriptive Statistic Indicators of the Thin Film's Dimensions Before Water Exposure

Dimensions (mm)						
Statistic Indicators	Solution A			Solution B		
	4	6	8	4	6	8
Mean (μ)	25.01x24.98	24.99x25.01	25.02x24.98	25.01x25.01	25.00x24.99	24.99x24.99
Standard deviation (s)	0.12	0.02	0.08	0.11	0.09	0.06
Variance (σ^2)	0.0144	0.00004	0.0064	0.0121	0.0081	0.0036

The surface aspect was observed using a Euromex BioBlue binocular microscope with a 100x magnification, 0.25 aperture achromatic objective. Two types of surfaces were found randomly distributed among all 60 thin films. As observed in Figure 4, the surface was either completely smooth and homogeneous, or with small holes on it.

Figure 4 - Microscopic images of the different types of surfaces randomly distributed among the 60 thin films. Left shows a surface with small holes. Right shows a flat homogeneous surface.



An analysis of variance (ANOVA) with a 0.05 significance was realized to determine if the 3 studied widths were statistical different. A Fisher's least significant difference (LSD) test was studied. All three used thicknesses were found to be different in terms of the mass of the film.

4.2 CONTACT ANGLE MEASUREMENTS

The contact angle values for solution A films oscillated between 28.7° and 36.4°. For solution B, the observed contact angle was between 55.6° and 94.4°. Since the contact angle of solution A thin films was < 90°, the thin films are said to be hydrophilic. In the same way, solution B films presented a contact angle which, except for 2 films, was 90°, which means that they can be said to be hydrophilic. However, the mean value of the contact angle was 84.6°, which means that solution B thin films are just slightly hydrophilic.

An ANOVA was used to determine if there was a dependence between the thickness of the membrane and the contact angle of the drop. The statistical analysis indicated that there is no significant difference (α=0.05) between the three used thicknesses in terms of the contact angle, which suggests that the contact angle is a property of the used solution rather than the mass of the film.

Similarly, an ANOVA was calculated to determine a relation between the aspect of the surface of the film and its contact angle. It was found that there is no effect of the surface being smooth or with holes in terms of the contact angle of the drop of water.

4.3 FOLDING ANGLE OF SOLUTION B FILMS

The maximum folding angle of the 30 films made up with solution B was determined at the point where the film reaches thermodynamic equilibrium with the drop of water. The average

and the standard deviation of the observed folding angle are shown in Table 3.

Table 3 - Descriptive Statistic Indicators of the Folding Angle of Solution B Thin Films

Folding Angle (°)			
Statistic Indicators	4	6	8
Mean (μ)	49.54	115.48	49.94
Standard deviation (s)	17.06	24.66	10.62

The ANOVA test revealed that the folding angle of the three different thicknesses is statistically different with a significance of 0.05. As shown by the data set, the folding angle of the intermediate thickness was greater than the ones corresponding to thicknesses 4 and 8. In addition, LSD test showed that only this angle was significantly different from the other two, which means that the films manufactured with 6 drops of solution B folded less than the ones fabricated with 4 and 8 drops.

4.4 FOLDING AND RECOVERY TIME OF SOLUTION B THIN FILMS

Since solution B thin films presented a folding reaction when exposed to a drop of water, a study was performed in terms of their folding and recovery time. Tables 4 and 5 show the mean and standard deviation of the acquired data.

Table 4 - Descriptive Statistic Indicators of the Folding Time of Solution B Thin Films

Folding Time (s)			
Statistic Indicators	4	6	8
Mean (μ)	50.1	42.9	58.5
Standard deviation (s)	15.47	16.89	19.69

Table 5 - Descriptive Statistic Indicators of the Recovery Time of Solution B Thin Films

Recovery Time (s)			
Statistic Indicators	4	6	8
Mean (μ)	250.9	143.2	420.3
Standard deviation (s)	30.49	45.39	140.47

An ANOVA test was performed for each of the observed times. Results show that while the recovery time between the films was different, the mean folding time of the 3 studied thicknesses was statistically the same. Moreover, the LSD test showed that the recovery time of the films fabricated with 6 drops of solution B was smaller than that of the remaining ones. This makes sense taking into account that the folding angle of the intermediate thickness films was greater than the one of the films manufactured with 4 and 8 drops, making the movement required for returning to the initial position of the last ones greater.

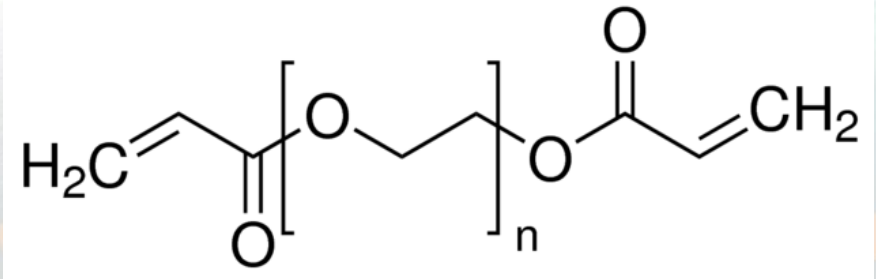
4.5 DISCUSSION

Contact angle measurements provided information of how hydrophobic or hydrophilic towards water the fabricated thin films were. Solution A thin films showed a mean contact angle of 30.1°, showing that the films are highly hydrophilic. In contrast, solution B thin films exhibited a mean contact angle of 84.6°, which means that the films are just in the border between being hydrophilic or hydrophobic. This is probably due to the fact that solution A polymer has a smaller number average molecular weight (Mn) than solution B. Having a smaller Mn implies a smaller size of the molecule, which provides a higher number of molecules per volume. Since the thickness of the polymer is controlled by the amount of volume of the used solution, it is sensitive to say that solution A thin films contain a larger number of molecules than solution B thin films, making solution A thin films more polar than those of solution B.

Solution B thin films were found to react when a drop of water touched their surfaces by folding themselves in the direction opposite to the location of the drop, while solution A thin films remained stand still after exposed to water. This was interesting to observe due to the fact that both solutions are made up from the same polymer and photoinitiator in exactly the same proportions. However, the Mn of solution A polymer was 250 while the Mn of solution B polymer was 575 as stated in the specifications provided by Sigma-Aldrich®.

The Mn indicates the statistical average molecular weight of the total number of polymer chains in the simple [11]. According to this, the number of polymer chains in solution A is smaller than that one of solution B. Figure 5 shows the chemical structure of Poly(ethylene glycol) diacrylate.

Figure 5 - Chemical structure of Poly(ethylene glycol) diacrylate (Sigma-Aldrich®).



Since the presence or not of a folding reaction on the thin film depends on the number of polymer chains in the material and not on the material itself, this reaction can be said to be a consequence of a change in a colligative property between solutions A and B. It appears to be that there is a relation between the folding reaction and the polarity of the thin film. Folding reaction of solution B thin films makes then be hygroscopic [12]. As a matter of fact, polyethylene glycol is a polar polymer and its hygroscopic properties have been already studied somewhere else [13, 14]. As observed in Figure 5, poly(ethylene glycol) diacrylate has an ester terminal functional group. These generates a dipole moment in the molecule, responsible for its polarity. It is important to take into account that the polymer is in a rigid-solid matrix that is organized randomly within the film, which limits the intermolecular movement. By definition of polarity, a polar molecule will have more affinity towards an equally polar molecule. Since water is a highly polar molecule, solution A thin films, which are more polar than solution B thin films, have a better affinity towards water. This implies that when the drop of water touches

the surface of solution B thin films, the polymer will try to decrease the contact area due to the lack of affinity as a consequence of the difference in polarity. Since the polymer is fixed in a matrix, the attempt of reorganizing itself to expose the most polar part of itself ends up in a folding reaction of the entire matrix. Moreover, the proportion of the backbone chain towards the terminal functional groups can be calculated from the Mn of each of the solutions. Each of the terminal functional groups have a molecular weight of approximately 71g/mol, which results in a 142g/mol contribution from the terminal functional groups. In addition, the backbone chain has a molecular weight of approximately 44g/mol. This means that solution A thin films contain approximately 2.45 unities in between the acrylate groups while solution B thin films contain approximately 9.8 unities. Therefore, the effect of the terminal functional groups on solution A is way higher than that of the backbone chain, which reinforces the idea that solution A thin films are more polar than the ones manufactured with solution B.

In order to confirm these ideas, both sets of thin films were exposed to different organic and inorganic liquids of diverse polarity. Table 6 shows the qualitative results of whether or not a folding reaction was observed. In every case, one drop of the studied liquid was placed on the surface of the film.

Table 6 - Qualitative Study of a Folding Reaction of Solution A and Solution B Thin Films When Exposed to Different Liquids

Liquid	**Solution A Thin Films**	**Solution B Thin Films**	**Dipole Moment**
H_2O_2	No	Yes	2.24D
H_2O	No	Yes	1.85D
$C_2H_4O_2$	No	Yes	1.74D
C_2H_6O	No	Yes	1.69D
CH_2Cl_2	No	Yes	1.60D
NH_3	No	Yes	1.47D
HCL	No	Yes	1.08D
Linseed Oil	No	No	Non-polar

As observed in Table 6, solution B thin films showed a reaction towards almost all of the tested liquids while solution A thin films didn't showed a reaction at all. This can be in part due to the fact that the tested liquids are encountered, in many cases, as a solution in water rather than in pure form. As expected, none of the thin films reacted towards linseed oil due to its non-polarity behaviour. However, solution B thin films showed a decrease in response time (i.e. the folding took place faster) when the dipole moment of the exposed liquid increased. This could be potentially characterized and used as a method of determining the polarity of unknown solvents. The observed relation between the folding rate and the polarity of the dropped liquid sustains the idea that the reaction is a consequence of polarity affinity that produces a reduction in the solid-liquid interphase area.

Moreover, both sets of thin films were exposed to vapor from boiling water and NH_3 gas. When exposed to NH_3 in gas form, neither of the thin films presented a reaction. Nevertheless, it is suspected that a significant increase in the concentration of NH_3 could be detected. Contrary, when exposed to water vapor, both sets of thin films exhibit a folding reaction, although solution B thin films showed a faster and more abrupt one. This could be due to the higher

polarity presented by vapor water in comparison with NH_3. However, since the experimental setup involved the exposure at a greater temperature, the contraction may not necessarily be a consequence of polarity. Baird et al. [14] showed that an increase in temperature of a polyethylene glycol will increase its hygroscopic effects due to the endothermic phenomena of the dissolution of the polymer in water.

5 CONCLUSIONS

An effect upon the hygroscopic properties of thin films manufactured with poly(ethylene glycol) diacrylate and phenylbis (2, 4, 6-trimethylbenzoyl) was observed when changing the Mn of the polymer from 250 to 575. Solution B thin films (Mn=575) exhibit a folding reaction when exposed to a drop of water. The response and recovery time as well as the folding angle was studied for three different thicknesses. It was found that the best reaction was produced for films above or below 6 drops of solution. In addition, solution A and solution B thin films exhibited a considerably different contact angle, which exhibit that solution A films were highly hydrophilic while solution B films were just in between the margin of being hydrophilic or hydrophobic. This gave an insight of solution A thin films being more polar than those produced from solution B. Hygroscopy was encountered to depend upon the Mn of the polymer, which had an effect upon the polarity of the polymeric matrix. In addition, it was observed that the response time of the folding reaction decreased when increasing the dipole moment of the exposed liquid. This aspect could be characterized and used as a way of sensing the polarity of unknown solutions. Finally, it would be interesting to study till what extent can solution B thin films be used as non-energized actuators due to their relatively fast response and recovery time.

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