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A Film of Polystyrene Hydroxyl end Group Supported on SiO₂ Monoliths: Thermal Conductivity and Micro-Indentation

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Keywords : Polystyrene film, SiO₂ monolith, thermal conductivity, thickness, Lee's Disk.

1. INTRODUCTION

More and more countries around the world, with predominantly hot or cold climates, tend to experience more extreme weather during most of the year. In this case, the use of air conditioning, ventilation and heating systems can help maintain a comfortable temperature [1]. However, the energy consumption is directly affected by glass windows that are a means by which energy is gained or lost from the surrounding environment [2]. The estimated energy consumption by this means has generated high costs in environmental and economic terms [3]. This limitation can be overcome by using a thermal insulation barrier on a conventional glass [4-6].

Thin organic polymeric films are receiving more and more attention in the field of thermal insulation

barriers because they present special properties, such as transparency, light-weight, easy transportability and installation, and low cost. Due to this, thin organic polymeric films have had a considerable technological importance in a wide variety of fields; most of them related to glass coating, used as solar- protective and anti-reflection coating. In both cases, these coatings have had a major impact on the temperature behavior [7, 8]. Equally important are the coating's physical and chemical properties, related to the adhesion phenomena, humidity and friction surface [9, 10]. Polymeric thin films based on polystyrene have offered advantages in optical and thermal properties (no absorption of visible light). By using this film, acoustic insulation, as well as excellent electrical and mechanical properties can be reached [11, 12, 13]. In addition to this, polystyrene chains having a low-molecular weight can improve its orientation and inter-chain dispersion with gases, liquids, particles and polymers. This is considered to be particularly advantageous in the current study; because with the addition of a functional group as -OH, a better adhesion of polystyrene chains can be achieved, improving its coating properties. This hydroxyl group attracts other polar groups as hydroxyl and alkoxy groups. Therefore, short-chains containing a hydroxyl end group (PSOH, Scheme 1) [14-16] provide an ideal structure that can be capable of reacting with fragments that appear after the Sol-gel process.

To provide evidence between PSOH and common glass window interaction, this common glass was interchanged by a silicon dioxide (SiO₂) substrate. This is because most commercial or common glass substrates have a complex chemical composition, which contain for the most part SiO₂, alkali and alkaline earth ions. They also contain the presence of impurities, making them usually unsuitable for a research protocol. In order to obtain SiO₂ glasses with high purity and homogeneity, the Sol-gel method (Scheme 1) was used [17, 18]. This process was performed at low temperature, allowing the researchers to obtain SiO₂ monolith disks (SiO₂-D's) [19]. Thus, by controlling the hydrolysis and the rate of the gel drying, transparent and low-density SiO₂-D's were obtained [20, 21]. These monoliths contain a -Si-O-Si-O- backbone and represent

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Si-OH groups, depending on the thermal treatment used. These fragments gain importance in presence of PSOH as result of polarity of the three functional groups. Given the context discussed above, the goal of this study was to examine the possibility of using a PSOH of medium molecular weight to provide a thermal insulation barrier on conventional glass windows, thus certain aspects were analyzed such as nitrogen sorption isotherm, molecular weight, FTIR spectra, hardness (microindentation test) and thermal conductivity (using the Lee's disk method).

II. EXPERIMENTAL

a) Materials and Methods

All reagents used in the study were purchased from Sigma-Aldrich Co.: tetraethyl orthosilicate (TEOS), ethanol (EtOH), nitric acid (HNO₃), toluene and 2-mercaptoethanol (2-ME). Styrene was purified by treating it with 5 wt% aqueous NaOH to remove the inhibitor and then washed with ion-free water until a pH=7 was achieved. It was later dried by using anhydrous. 2-2' Azobisisobutyronitrile (AIBN) was purified with methanol by recrystallization.

b) Synthesis of polystyrene with hydroxyl end group (PSOH)

The PSOH was prepared using the following procedure: (1) styrene (5x10⁻² moles), 2-ME (5x10⁻³ moles) and AIBN (2.1x10⁻⁴ moles) were dissolved in toluene. (2) This mixture was degassed by several freeze-thaw cycles under high vacuum, and then polymerized for 5 hours under nitrogen gas at 70°C.

Finally, (3) the solution was diluted in toluene and precipitated in methanol [22]. According to the reaction, the hydroxyl group -S(CH₂)₂OH should be present at the end of the polystyrene chain (Scheme 1).

c) Silica monoliths disks (SiO₂-D's)

SiO₂-D's were obtained by the sol-gel process, which was completed in three stages: (1) A first solution of TEOS/ETOH with a molar ratio of 4.5x10⁻³:69.0x10⁻³ (1/4 volume relation) was heated for 15 minutes under magnetic stirring at 60°C [23]. (2) A second solution of HNO₃/H₂O with a molar ratio of 4.7x10⁻³:89.0 x10⁻³ (1.0/80 volume relation) was prepared at 18°C. In the end, (3) both solutions were mixed and stirred for 1 hour at 60°C. The resulting mixture was left to cool at 18°C and then transferred to 10 ml plastic vials (drilled with small holes to permit a slow solvent). The plastic vials were subsequently put under vacuum for different time periods during which the temperature was gradually raised from 20 to 250°C.

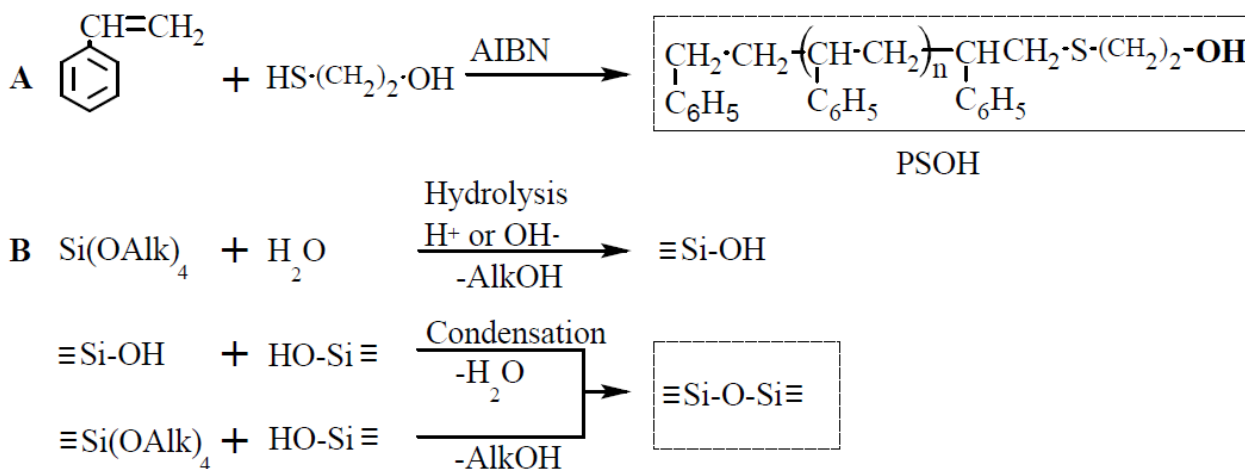
d) Coating of PSOH on SiO₂-D

By dipping SiO₂-D (on one of its sides) partly into the PSOH/toluene solution once, twice or three times, SiO₂-D's covered with PSOH were obtained. (Figure1). The dipping processes were carried out in three replications, which were conducted at a rate of 10 cm/min for 1 second, using a digital immersion apparatus.

e) Instrumentation and characterization methods

The nitrogen sorption isotherm was obtained by the static volumetric method at 196 °C.

Escheme 1 : A) PSOH preparation using MEOH as transfer agent and B) SiO₂-D matrix preparation by the Sol-gel method.



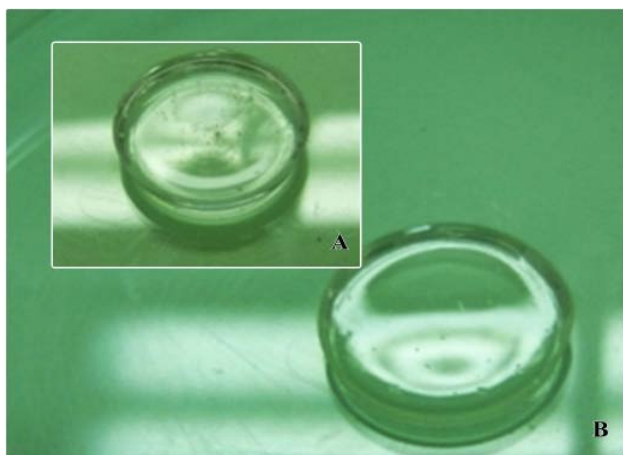


Figure 1 : Transparent silica monoliths of 1.5 cm in diameter and 2 cm along: A) uncoated and B) uncoated with PSOH.

The specific surface area (m²/g) was determined using the BET (Braunauer, Emmet and Teller) method.

The molecular weights of the PSOH were measured using gel permeation chromatography (GPC) Waters 1525, Binary HPLC. The equipment was calibrated with polystyrene standards, and tetrahydrofuran was used as a solvent.

FTIR spectra were obtained using a Tensor 27 Bruker spectrometer in the 2000-400 cm⁻¹ spectral range, at a resolution of 4 cm⁻¹ in absorption mode.

The average film thickness was calculated using the polystyrene density (1.05 gcm³) and it was assumed that the film coated totally and uniformly the SiO₂-D surface.

The microindentation tests to determine hardness (H_v) behavior were carried out using a Vickers microindentation tester (Durimet, Leitz, Wetzlar Germany), with a 10, 25 and 100 gf load (P) in the diamond shaped indentation tip (pyramid form).

Through the tester microscope, it was possible to observe (10x) the mark on the surface of SiO₂-D with and without coating.

Characterization of the experimental arrangement through Lee's disk method was carried out using *VirtualBench-Logger 2.5 Report National Instruments* software, an Analog Input, National Instrument TC-2190 data acquisition board, 5 "k" type thermocouples with chromel and alumel joints, the hot plate, two cylindrical lead pieces (1.5 cm in diameter and 2 cm along), five firebricks (silica-alumina) and diatomaceous earth. Figure 2 shows a schematic representation of the experimental arrangement of Lee's disk method, where there are two systems: one that uses pure SiO₂-D and another one that uses SiO₂-D coated with PSOH (system I and II respectively). In both

systems, there was a piece of copper on both sides of the specimen having four thermocouples A, B, C and D which gave information about the interface. Each system was isolated with 5 firebricks forming a chamber filled with diatomaceous earth, where a last thermocouple (E) was inserted to monitor its temperature.

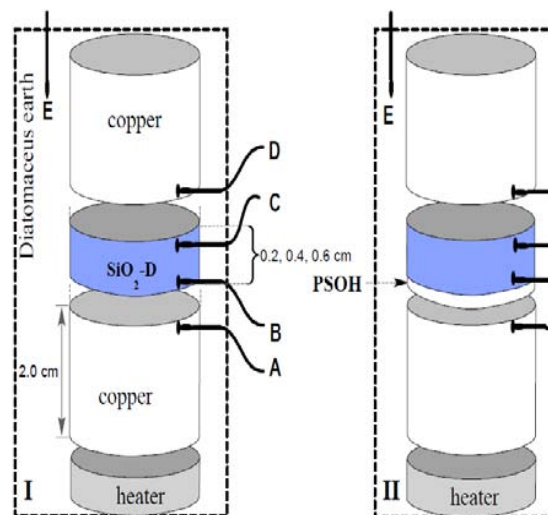


Figure 2 : Arrangement of the Lee's disk method: System I, SiO₂-D without PSOH film and System II, SiO₂-D with PSOH.

Each experiment was carried out under stable operating conditions for 40 minutes. While a plane surface heat source was set at a fixed temperature of 110°C, the diatomaceous earth temperature was kept constant at 20°C. It also revealed that there was no heat lost in the systems (I and II) toward the surroundings, and an appropriate isolate system was built.

The equation that describes heat transfer through conduction (Fourier Law) can be expressed as a linear approximation, as follows:

$$(q/A)_z \approx -k(\Delta T/\Delta z) \quad (1)$$

where $(q/A)_z$ is the heat flux in the z-direction in W/m², k is the thermal conductivity in W/m K, and $\Delta T/\Delta z$ is the temperature gradient $T_n - T_{n+1}$; $T_n > T_{n+1}$ as a function of z-thickness ($z_{n+1} - z_n$; $z_{n+1} > 0$ y $z_n = 0$) in °C/m. In regard to the equation describing specific heat, it can be expressed as heat transfer in transitory state:

$$q = mC_p(dT/dt) \quad (2)$$

where q is the power in the transitory state, m is the mass of material in g, CP is the specific heat of material in J/g °C, and dT/dt is the differential of temperature depending on the t-time in °C/min. In addition, the cross-sectional area of a cylinder can be expressed:

$$A = \pi D^2/4 \quad (3)$$

where D is the diameter of material in cm.

III. RESULTS AND DISCUSSION

a) PSOH molecular weight and monolith characteristics

The weight-average molecular weight of PSOH was about MW = 70345 g mol⁻¹, having a molecular weight distribution of MW/Mn = 1.2. This value indicated that a low molecular weight PSOH was obtained.

SiO₂-D's with a single diameter of 1.5 cm and thicknesses of 0.2, 0.4, and 0.6 cm were obtained, with a relative standard deviation of 3.4 and 3.0 % for diameter and thickness respectively. These thicknesses were used to label each SiO₂-D, as SiO₂-D_{0.2}, SiO₂-D_{0.4}, and SiO₂-D_{0.6}, each one having an average weight of 0.8250, 1.6498 and 2.4748 g respectively, with a relative standard deviation of 2.5, 2.8 and 3.2 % respectively. Table 1 shows the relations between reagents and solvents that were used in each case.

Furthermore, according to the standard BET method, the SiO₂-D's were composed of pores having a diameter of 1.8 nm.

b) FTIR analysis

Figure 3 shows the infrared absorption spectra of the PSOH and pure SiO₂-D. The presence of the PSOH was determined by the characteristic bands of polystyrene (Figure 3A). The aromatic PSOH fraction gave a C-H stretching for the sp² carbon at 3067 cm⁻¹; this fraction exhibited the characteristic overtone bands in the region from 2000-1667 cm⁻¹ because of the monosubstituted ring. Additionally, the phenyl ring vibrations at 1601, 1452, 753 and 695 cm⁻¹ were clearly observed.

Table 1 : Initial concentration of the reactant for SiO₂-D preparation

First solution		Second solution	
TEOS	ETOH	H ₂ O	HNO ₃
4.5x10 ⁻³	6.9x10 ⁻²	8.9x10 ⁻¹	4.7x10 ⁻³
0.9330	3.1600	16.0000	0.2980
1	4	16	0.2

Peaks at 3278 and 2628 cm⁻¹ were associated with the alkyl substituent group. Additionally, due to OH, the PSOH showed a broad stretched band centered at 3465 cm⁻¹, originated by the H...O-H bond.

The spectrum presented in the Figure 3B illustrates the situation of SiO₂-D. This includes intense absorption peaks at 1170, 1070, 950 and 800 cm⁻¹.

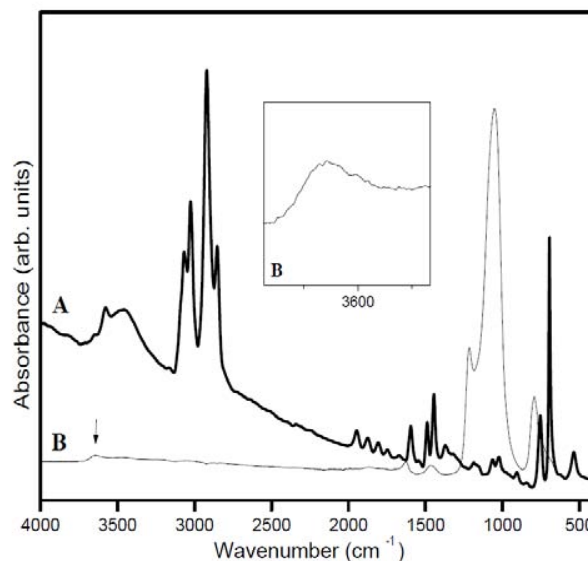


Figure 3 : FTIR spectra of A) PSOH having a OH group and B) SiO₂-D.

These peaks were assigned to the absorption of the longitudinal optic Si-O-Si asymmetric, Si-O-Si asymmetric stretching, Si-OH group, and Si-O-Si symmetric stretching, respectively. Finally, through zooming into the spectrum, a very weak broadband signal at 3645 cm⁻¹, due to a O-H stretching can be easily observed. These results conceived the existence of an intermolecular hydrogen bonding between PSOH and SiO₂-D.

c) Coating of monolith by the polymer

Table 2 shows the PSOH films thickness value (PSOH_E) and the weight percentage (wt%) of PSOH on SiO₂-D, in relation to the number of dips. The results showed that all SiO₂-D's coated were transparent, as well as hard and plain in consistency.

According to the experimental data (Table 2), the film thickness was independent of SiO₂-D thickness, because SiO₂-D's of different thicknesses absorbed almost the same amount of PSOH after each dipping. This shows that the PSOH chains were absorbed on identical surfaces.

As a consequence, after each immersion, more chains were added (increasing the monolith weight) which suggests the existence of a strong chain-chain attraction. These new chain depositions were done without significant detachment (by solvation) from the previous chains.

Table 2 : PSOHE and gain weight percent (wt%) as function of SiO₂-D thickness and numbers of dips

Numbers of dips	1		2		3	
	PSOHE* ^a	wt%	PSOHE* ^a	wt%	PSOHE* ^a	wt%
SiO ₂ -D _{0.2}	1130	0.049	2246	0.098	3782	0.215
SiO ₂ -D _{0.4}	1139	0.031	2206	0.049	3772	0.107
SiO ₂ -D _{0.6}	1128	0.015	2256	0.029	3801	0.074
Averages	1132		2236		3785	

* PSOH films thickness value in nm.

d) Hardness determination

Figure 4 shows photos obtained after the microindentation hardness tests.

These tests determined the effect of PSOH film thickness on SiO₂-D_{0.2} and SiO₂-D_{0.6}.

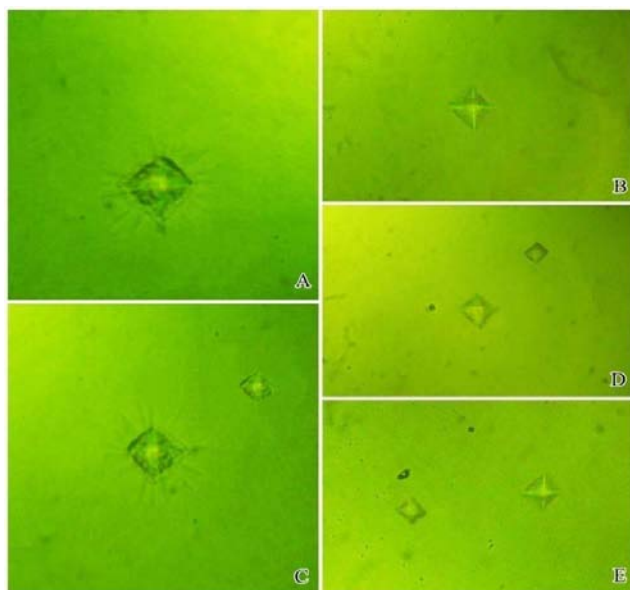


Figure 4 : Micrographs of specimens: A) pure SiO₂-D_{0.2} (100 gf), B) SiO₂-D_{0.2} with PSOHE= 3782 nm (100 gf), C) pure SiO₂-D_{0.6} (10 gf), D) SiO₂-D_{0.6} with PSOHE= 1128nm (10 gf), and E) SiO₂-D_{0.6} with PSOHE= 3801nm (10 gf).

The first Vickers indentation was performed on pure SiO₂-D_{0.2}, and it was made with 100 gf *P*. In this monolith, a large number of cracks around the perimeter mark were observed (Figure 4A).

As a result of PSOH deposition, PSOHE=3782 nm, shorter cracks and a good geometrical definition of the mark were determined (Figure 4B).

A variant was found on the surface of the pure SiO₂-D_{0.6} after applying a load of 10 gf *P* (Figure 4C). The cracks around the perimeter mark and the geometrical definition were lower than that of the pure

SiO₂-D_{0.2}. This situation changed when the SiO₂-D_{0.6} was coated with a PSOH film thickness of 1128 nm (Figure 4D). In this circumstance, the cracks around the perimeter mark, as well as the geometry were better defined after applying the same load. Moreover, as the PSOH film thickness was increased to 3801 nm, shorter fissures around the perimeter mark and a high geometry definition were observed (Figure 4E).

According to the above mentioned results, there was a decrease in fissure size after the PSOH film addition, which was more evident as the film thickness was increased. This was due to the lamination effect, which is produced by the strong intermolecular interaction between PSOH chains.

Table 3 indicated that *Hv* values were direct function of the geometrical mark dimension and *P* in the tip of the diamond shaped indentation. For all coated SiO₂-D's, the *Hv* values were increased as the load and PSOH thickness were increased.

Additionally, the *Hv* values were direct function of the PSOH film and the SiO₂-D's thickness. This behavior is mostly related to the uniform dispersion, orientation and attraction of PSOH chains on the SiO₂-D surface, giving it an increase of the ductile strength. This situation may be due to the medium molecular weight of PSOH, which introduced a notorious increase in the *Hv* values.

e) Temperature evaluation (Lee's Disk method)

From the System I developed for an uncoated monolith test (Figure 2), the following notes were obtained: for instance, for SiO₂-D_{0.2}: A₁, B₁, C₁, and D₁ and so forth, this monolith had a temperature reduction from An to Bn, and from Cn to Dn that were barely 0.4°C and 0.35°C respectively (Figure 5A).

Table 3 : Microindentation values of pure SiO₂-D and with PSOH films thickness values.

	P (gf)	Monolith		PSOH films on SiO ₂ -D				
		H _v (GPa)	PSOHC	H _v (GPa)	PSOHC	H _v (GPa)	PSOHC	H _v (GPa)
SiO ₂ -D _{0.2}	100	23.0220	1130	25.8370	2246	29.6870	3782	31.5720
SiO ₂ -D _{0.4}	25	92.0408	1139	103.3380	2206	114.8042	3772	126.2701
SiO ₂ -D _{0.6}	10	230.1021	1128	258.3451	2256	287.0106	3801	315.6753

Consequently, two values were omitted from each pair, which only left B and C. Similarly, all B temperature values were assumed to be the same for all studies (Figure 5B).

System I and II took similar considerations, except that A' was not omitted due to its temperature difference with B'. On the other hand, because A's value is independent of the tested monolith (A'=A), it was used as an indicator in the increase of the temperature.

Table 4 shows the analysis carried out in pure SiO₂-D specimens, showing that the temperature decreased due to an increase of the SiO₂-D thickness.

This tendency was expected, however, the heat reduction was very significant, reaching up to 60% at 31.5 minutes for the monolith of higher thickness, SiO₂-D_{0.6}.

The difference of insulating temperature between B and C offered the temperature gradient ΔT, which was established for each thickness of the SiO₂-D. Subsequently, the temperature quotient was

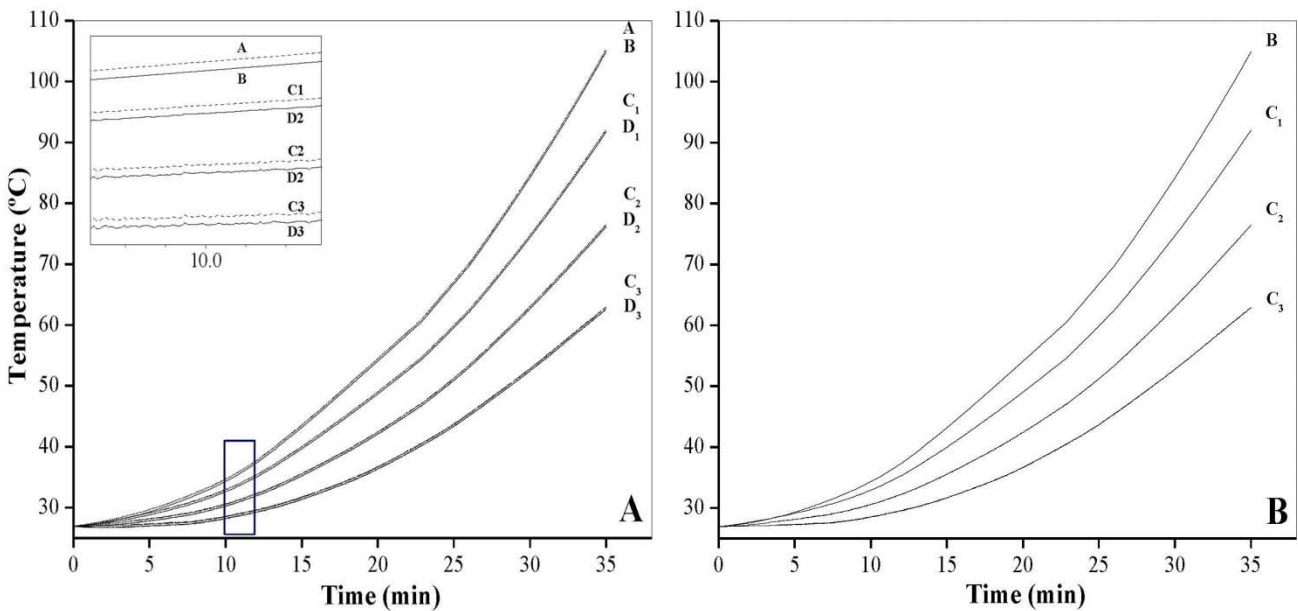


Figure 5 : System I: A) SiO₂-D with A and Dn and B) without A and Dn dates.

Table 4 : Temperature values (°C) and gradient, ΔT (°C) of pure SiO₂-D from B to C as function of time and SiO₂-D thickness.

Time, min.	SiO ₂ -D	SiO ₂ -D _{0.2} , ΔT	SiO ₂ -D _{0.4} , ΔT	SiO ₂ -D _{0.6} , ΔT
	B	C ₁	C ₂	C ₃
13.4	40	37.4, 2.6	33.7, 6.3	30.4, 9.6
24.4	65	58.4, 6.6	50.1, 14.9	42.8, 22.2
31.5	90	79.6, 10.4	66.8, 23.2	55.8, 34.2

Obtained in relation to the time in the transitory state (dT/dt; slope) which was associated to the Cn value.

Figure 6 shows the thermal analysis corresponding to SiO₂-D having a PSOH film. The Figures 6A, 6B, and 6C indicate the analysis of SiO₂-D_{0.2}, SiO₂-D_{0.4} and SiO₂-D_{0.6} respectively. The subscript numbers correspond to the monolith thickness and new additional subscript numbers correspond to the PSOH thickness values, which are 1130, 2206, and 3801 nm.

Table 5 separately shows the PSOH film performance, without considering the SiO₂-D thickness, indicating the constant A' as well as B's values for different PSOHC. ΔT was calculated to obtain the temperature quotient for each B₁', B₂' and B₃' as a function of time in a transitory state (dT/dt). The insulating temperatures by PSOH thickness and by SiO₂-D were A' - B' and B' - C' (is equal to B - C) respectively. Thus, the difference in the insulation total (reduction temperature) is given by (A' - B') + (B' - C'). For the systems I and II, as the experiment time runs in the interval of 0 to 35 minutes, there was an increase of ΔT as SiO₂-D and PSOH thickness were increased. This tendency was associated with the dielectric properties of both SiO₂ and PSOH.

Figure 7 shows the heat flux behavior as a function of temperature gradient and thickness in SiO₂-D (Figure 7A) and PSOH (Figure 7B). The heat flux increases linearly due to the thickness increase of SiO₂-D and PSOH. This was attributed to a higher temperature required to transfer heat from A' to B' and

from B' to C'. The thermal conductivity value (k; slope) in each SiO₂-D thickness (Figure 7A) and in each PSOH (Figure 7B) was almost the same.

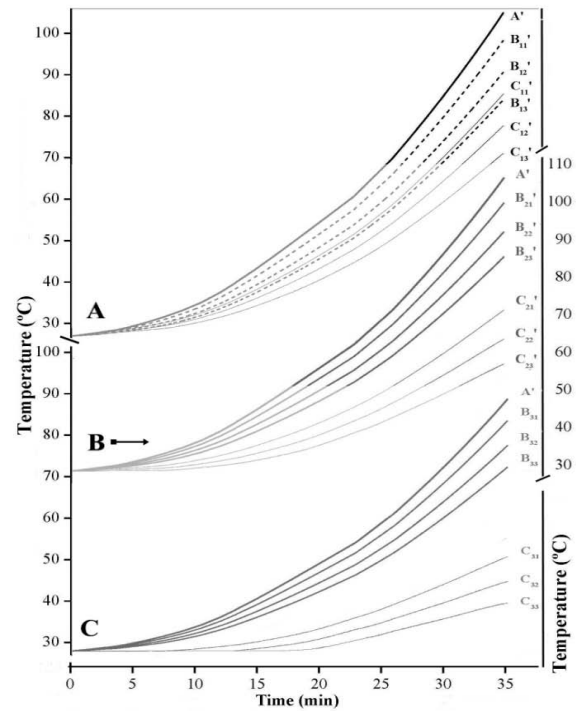


Figure 6 : System II: A) SiO₂-D_{0.2} B) SiO₂-D_{0.4} and C) SiO₂-D_{0.6}, having each monolith a PSOHC of 1130, 2385 and 3703nm respectively.

Table 5 : Temperature values (°C) and gradient, ΔT (°C) of PSOH films from A' to B' as function of time and PSOHC for no specific SiO₂-D.

Time, min.	PSOH ε = 1130, ΔT			PSOH ε = 2206, ΔT		PSOH ε = 3801, ΔT	
	A'	B ₁₁ '		B ₁₂ '		B ₁₃ '	
13.4	40	38.5, 1.5		36.7, 3.3		35.2, 4.8	
24.4	65	61.5, 3.5		57.4, 7.6		53.9, 11.1	
31.5	90	84.6, 5.4		78.3, 11.7		72.9, 17.1	

All thickness values (ε) in nm.

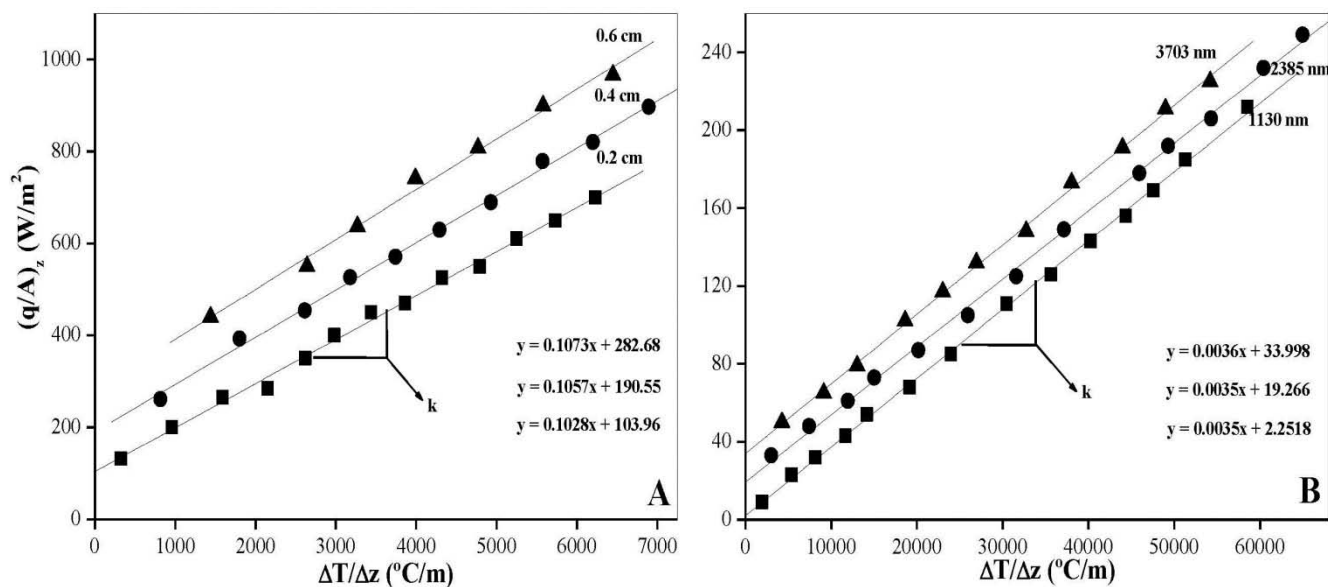


Figure 7: Heat flux behavior as a function of temperature gradient and thickness in: A) SiO₂-D, and B) PSOH film.

This was related to the fact that in certain materials such as SiO₂ and PSOH, k does not depend on its thickness, so average SiO₂-D and PSOH of k values were 0.1053 and 0.0035 W/m K, respectively. The SiO₂-D coated and uncoated with PSOH showed a high transparency and well defined geometry. The maximum Hv values and ductile behavior were found as the thickness values in PSOH increased. The individual values of SiO₂-D and PSOH film of k (0.1053 and 0.0035 W/m K) were lower compared to the k values coming from bibliographic references of pure SiO₂ and polystyrene (1.46 and 0.12 W/m K). It may be a result of a substantial air (0.02 W/m K) presence in the silica pore structure and the dispersion of polymer chains within the inorganic matrix (monolith pore size diameter 1.8 nm). Consequently, the PSOH increases its thermal insulation properties when it is supported on SiO₂-D. Preliminary, this could be attributed to the strong interfacial interaction between the polymer and the substrate, where the PSOH chains can be disassociated as a consequence of a possible affinity.

IV. ACKNOWLEDGMENTS

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