

# Thermophysical and Mechanical Properties of Sprayable Polydimethyl Siloxane Coating

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**Abstract** - Recently, there has been a large increase in employing polymers in engineering applications. modified polysiloxanes are generally recognized as the newest generic class of high performance protective coating. For this reason, understanding the mechanical, thermal properties of sprayable thermal protection PDMS under different loading rates and temperatures has become increasingly important. Various methods were applied to evaluate the effect of additives on polymer thermal properties. Differential Scanning Calorimetry (DSC) was used to study the phase transitions such as glass transition temperature as well specific heat behavior and comparative thermal conductivities were measured. Mechanical properties were measured using dynamic mechanical analysis technique and the percentage of thermal expansion coefficient has been measured.

**Key Words:** Poly Dimethyl Siloxane-Flame retardant- Heat retardant- Mechanical Properties-Thermal Properties

## 1 INTRODUCTION

silicon based polymers have been used as coating binders for more than 60 years. The first silicon based coating binders were the alkali silicates used in the formulation of heat-cured zinc rich primers in the 1940's. The development of silicone resins after World War II resulted in the first major commercial applications for silicone coatings; heat-cured, high temperature resistant paints for exhaust stacks, boilers, heat exchangers, mufflers, engines and aircraft components. [1]

Silicone materials applied into a billion-dollar industry, and used in many applications in civil engineering, construction building, electrical, transportation, aerospace, textiles, and cosmetics industries.

Coating technologists have long sought to utilize the properties of silicone polymers to improve coating properties for many years.

Silicon based materials as coating binders was limited to the specialized coatings just mentioned, primarily because of poor film flexibility and toughness, incompatibility with organic polymers, the need for heat-curing and certain problems associated with film formation [2].

liquor Polydimethylsiloxane (PDMS) mixed with flame retardant and heat retardant to withstand higher temperature

than pure silicone rubber and employed this technique as sprayable thermal insulation for metallic case of aircraft. [3]

Coatings based on this chemistry overcame the need for heat curing and provided good flexibility with improved solvent and acid resistance compared to conventional epoxy coatings. Cost, stability and adhesion problems limited commercial success. [4]

Development of polysiloxane as a protective coating, polysiloxane coatings have gained market share compared to epoxy, polyurethane and other traditional organic coatings and are perhaps the fastest growing generic coating type. Of note, the number of coating manufacturers that supply polysiloxane coatings has nearly tripled in the last four years [5].

The reasons for the rapid growth of polysiloxane coatings are clear. They offer improved performance properties and cost effectiveness, lower VOC content and improved health and safety features compared to traditional organic coatings.

Siloxane chemistry and formulation technology have led to the development of temperature liquor polysiloxane coating systems with significant advantages compared to traditional inorganic and organic thermal insulation adhesive coatings application [6].

Silicones first developed in the 1950's, to use as coating, these coatings overcame incompatibility problems by pre-reaction of silanol functional silicone resin carbinol functional organic resin.

These coatings are still in use today for the protection of Navy vessels, tanks, process equipment, rail cars and other steel structures. The backbone polymer in the silicone industry is polydimethylsiloxane (PDMS) [7].

## 2 EXPERIMENTAL

### 2.1 Materials and Techniques

#### 2.1.1 PDMS (SILIKPHEN P/80/X)

Chemical formula: PDMS Resin

$\text{CH}_3[\text{Si}(\text{CH}_3)_2\text{O}]_n \text{Si}(\text{CH}_3)_3$

Appearance: viscous Liquid.

#### 2.1.2 Mica (Muscovite)

Chemical formula:  $\text{KA}_{12}(\text{AlSi}_3\text{O}_{10})(\text{FOH})_2$  Grade V

Appearance: Crystal Color Ruby / Green

### 2.1.3. Kaolinite (Kaolin)

Chemical formula:  $Al_2Si_2O_5(OH)_4$ , Grade C

Appearance: Crystal Triclinic.

### 2.1.3. Polyamide (Torlon) 66

Chemical formula:  $(C_{12}H_{22}N_2O_2)_n$

Density = (1.14) g/ml at 25°C

### 2.1.4. Zinc Borate (ZB)

Chemical formula:  $(2ZnO.3B_2O_3.3.5H_2O)$

Fire Brake Zinc Borate (Borax)

### 2.1.5 Calcium Carbonate (CC)

Chemical formula:  $CaCO_3$

Appearance: White crystalline powder

### 2.1.6 Antimony trioxide (ATO)

Flame retardant

Chemical formula:  $Sb_2O_3$

Appearance: white powder

### 2.1.7 Alumina Trihydrate (ATH)

Chemical formula:  $Al_2(OH)_3$

Appearance: white powder

All chemicals supplied by  
 Abo-Zabal company for  
 Special chemicals, Egypt

## 2.2 Techniques

### 2.2.1 Specific Gravity

The PDMS density was measured at 25 °C using Electronic Densimeter (H -300S) ASTM. No. (D-792). The specimen is weighed in air then weighed when immersed in distilled water at 25°C using a sinker and wire to hold the specimen completely submerged as required. Specific Gravity is calculated as follows:

$$\text{Specific gravity} = a / [(a + w) - b]$$

a = mass of specimen in air.

b = mass of specimen and sinker in water.

W = mass of totally immersed sinker and partially immersed wire [8].

### 2.2.2 Mechanical Properties

The mechanical properties of polydimethylsiloxane (PDMS) are inevitably important properties, because most applications involve mechanical loading under a particular service conditions. PDMS, like other thermoplastics, is a viscoelastic material. The mechanical properties depend on time, temperature [9].

Storage modulus, loss modulus, stiffness and Tan delta were measured experimentally for each of the prepared samples using the DMA Q800 V20.24 Build 43. Dynamic Mechanical Analysis determines elastic modulus (or storage modulus, E'), viscous modulus (or loss modulus, E'') and damping coefficient (tan δ) as a function of temperature, frequency or time. Results are typically

provided as a graphical plot of E', E'', and tanδ versus temperature. [10]

$$\text{Storage Modulus: } \dot{E} = \frac{\delta^0}{\epsilon^0} \cos \delta$$

$$\text{Loss Modulus: } \dot{E} = \frac{\delta^0}{\epsilon^0} \sin \delta$$

$$\text{Tan Delta: } \tan \delta = \frac{\dot{E}}{\dot{E}}$$

$$\text{Stiffness: } K = \frac{F}{\Delta}$$

Where: -

Δ is the displacement produced due to force

F is the force applied to the body

δo is the amplitude of the stress

εo is the amplitude of the strain

Stiffness (K) is the rigidity of an object, the extent to which it resists deformation in response to an applied force [11].

### 2.2.3 Hardness-Shore (A)

The hardness test is used for measuring the relative hardness of soft materials and is based on the penetration of a specific indenter forced into the material under specified conditions.

The hardness shore (A) is measured using the hardness tester ZWICK (model- 3102) at 25 °C. According to ASTM. No. (D-785) [12].

### 2.2.4 Thermal Conductivity

Thermal conductivity is the rate at which a material conducts heat energy through itself.

It is the quantity of heat that passes through a unit of the material per unite time when the temperature difference of two faces is 1 K.

The thermal conductivity of the test specimen is determined from the knowledge of the thermal conductivities of the reference materials, the temperature gradient through the reference and test samples, and the geometry of each sample from the equation [13].

$$K_{ts} = \frac{1}{2} \left( \frac{\Delta X}{\Delta T} \right)_{ts} \left[ \left( K \frac{\Delta T}{\Delta X} \right)_{tpr} + \left( K \frac{\Delta T}{\Delta X} \right)_{br} \right]$$

$K_{ts}$  = thermal conductivity of tested sample.

$K_{tpr}$  = thermal conductivity for top reference sample.

$K_{br}$  = thermal conductivity for bottom reference sample.

$\Delta X$  = distance between thermocouples in each sample  
 $\Delta T$  = temperature difference through each sample.

### 2.2.5 Differential Scanning Calorimetry

The differential scanning Calorimetry method is widely used to examine and characterize substances, mixtures, and materials. This technique is internationally standardized under ASTM D3418. The principle of operation depends on measurement of the heat flux between the sample and reference. The heat flux is measured while the temperature is changing. Thermal analysis experiments were carried out on using a (DSC-50) Shimadzu instrument. [14] The experiments were elaborated at heating rate of 10 (C/min), flow Rate: 20(ml/min). PDMS samples were heated under a nitrogen atmosphere in the DSC cell up to 400 °C. During these transitions, the sample will either absorb or radiate heat. This heat is characterized by a temperature change in the sample which is detected by a thermocouple and compared with the relative temperature of the reference cell ( $T_s - T_r = \Delta T$ ) [15].

### 2.2.6 Thermomechanical Analysis (TMA)

Thermomechanical analysis (TMA) is a useful technique used to characterize linear expansion, by applying a constant force to a specimen while varying temperature. The performance of these tests is done through the use of a magnetic force coil which applies positive or negative loads to the sample, and a linear variable displacement transformer which measures the expansion or contraction of the sample.

The TMA is almost always used to measure samples well before their point of degradation.

The coefficient of linear thermal expansion may be recorded as the mean  $\alpha$  ( $\Delta T$ ) or differential  $\alpha$  (T) and is calculated in accordance with DIN 53 752, ISO 113591 [16].

The mean coefficient of linear thermal expansion  $\alpha$  ( $\Delta T$ ) is derived as follows:

$$\alpha (\Delta T) = \frac{l}{l_0} \frac{l_2 - l_1}{T_2 - T_1} = \frac{l}{l_0} \frac{\Delta l}{\Delta T}$$

Where: -

$\alpha$  ( $\Delta T$ ): The coefficient of linear thermal expansion.

$l_0$ : reference length.

$\alpha$ : linear thermal expansion.

$\Delta l = l_2 - l_1$  = linear variable displacement.

$\Delta T = T_2 - T_1$  = temperature difference through sample.

The temperature-dependent change of length is the progressive change of length expressed in terms of the initial length/reference length  $l_0$ . [17] It is a relative measure of the linear Expansion, which always has a value of 0 at the start of the trial at the reference temperature  $T_0$ . The values may assume the dimensions [10<sup>-6</sup> °C<sup>-1</sup>] or [K<sup>-1</sup>].

ISO 11359-2 [25] recommends [K<sup>-1</sup>], we shall use the unit [ $\mu\text{m}/(\text{m}^\circ\text{C})$ ] because this conveys a better impression of the magnitudes involved [18].

## 3 RESULT AND DISCUSSION

### 3.1 FORMULATIONS

According to the thermal experimental studies experienced in the present work, the formulations can be as shown in Table (3-1).

Table (3-1) Thermal Formulation for PDMS

Samples%	MICA	ATO	ATH
S1	0	0	0
S2	2	1	1
S3	2.5	1.5	1.5
S4	3	1.7	2
S5	3.5	2	2.5
S6	4	2.5	3
S7	5	3	4
S8	7	4	5
S9	8	5	7
S10	22	7	10
S11	32	10	13
S12	40	12	16

According to the mechanical experimental studies experienced in the present work, the formulations can be as shown in Table (3-2).

Table (3-2) Mechanical Formulation for PDMS

Samples%	KW	PA	CC	ZB
S <sub>1</sub>	0	0.2	8.8	2
S <sub>3</sub>	1.5	1	15.6	2
S <sub>5</sub>	3	2.5	18.4	2
S <sub>7</sub>	5.2	6	20.3	2
S <sub>9</sub>	8	8.5	22	2
S <sub>11</sub>	12.4	10	25	2
S <sub>12</sub>	20	15	35	2

### 3.2 Specific Gravity of PDMS

The specific gravity of different samples of PDMS has been measured at 25 °C. The results are listed in Table (3-3), from which it is clear that the specific gravity increased from 0.970 to 1.32 on increasing the amount of fillers.

Table (3-3) Specific gravity of PDMS

Samples	S1	S3	S5	S6	S7	S9	S11	S12
SP. G	0.970	0.997	1.20	1.22	1.24	1.26	1.3	1.32

### 3.3 Mechanical Properties

Thermal and mechanical properties of various PDMS (polydimethylsiloxane liquor) formulations have been recorded and extensively discussed.

Such Storage modulus, loss modulus, stiffness and Tan  $\delta$  as well as hardness were measured, the results are in shown in Table (3 -4) and illustrated in Fig. (3-1) to Fig. (3-4).

Table (3-4) Mechanical Properties of PDMS

Samples Materials	S1	S2	S3	S4	S5	S6	S7
KW%	0	1.5	3	5.2	8	12.4	20
PA%	0.2	1	2.5	6	8.5	10	15
CC%	8.8	15.6	18.4	20.3	22	25	35
STM100°C (Mpa)	175	280	310	390	450	590	1340
STM200°C (Mpa)	70	80	152	153	175	290	985
LM100°C (Mpa)	90	54	50	51	48	46	44
LM200°C (Mpa)	85	47	45	43	41	32	30
STF100°C (N/m)	10300	10400	10500	10600	1750	10850	20100
STF200°C (N/m)	9700	9800	9860	9900	9970	9990	10300

3.3.1 Storage Modulus (STM)

Fig (3-1) demonstrates the effect of Kaolinite addition on the storage modulus, from which it is clear that the storage modulus increases with increasing Kaolinite (KW) %, reaches a maximum of 1340 (MPa) at 100 °c and 985 (MPa) at 200 °c, beyond maximum value of 20 % KW% the increase in storage modulus is marginal. It may be also noticed from the same figure increases the storage modulus by adding KW % up to 20 %. Which Indicates complete conserve energy and the ability of the sprayable thermal insulation coat to conservation of energy at high temperature during application.

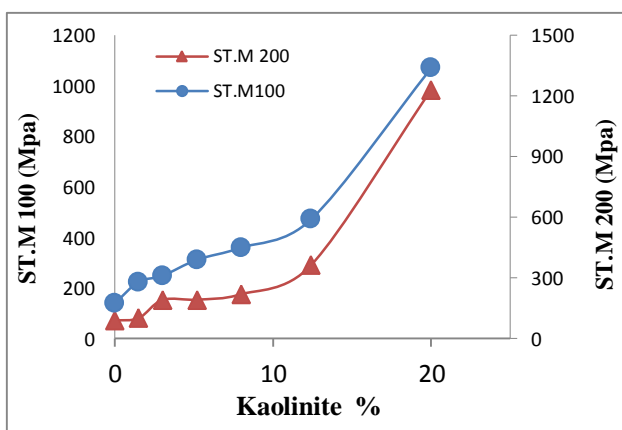


Fig. (3-1) Effect of Kaolinite addition on Storage Modulus at Fixed Temperature (100-200) °c

3.3.2 Losses Modulus (LM)

The effect of increasing the  $CaCO_3$  (%) on loss modulus is demonstrated in Fig (3-2) from which it is clear that the addition of  $CaCO_3$  (%) minimizes the losses modulus from 90 (MPa) to 44 at 100 °c and from 85 (MPa) to 30 at 200 °c, respectively at constant temperature (100,200), Illustrated at table (3-3). Decreasing the losses modulus means the high efficiency of the sprayable thermal insulation coating which conserve energy and not easy release it, which increased the mechanical stability of the thermal insulation coat.

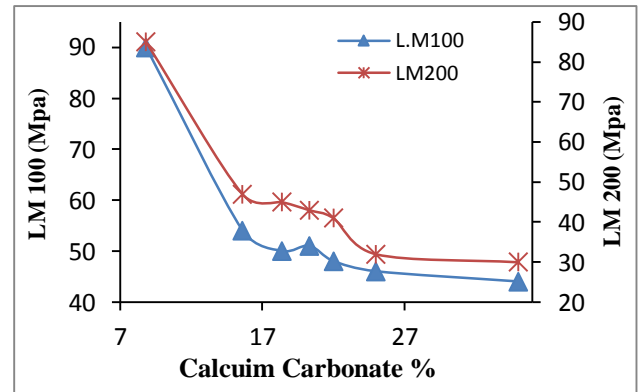


Fig. (3-2) Effect of Calcium Carbonate addition on Storage at Fixed Temperature (100-200) °c

3.3.3 Stiffness (STF)

Fig. (3-3) shows the effect of adding a (CC+PA) % mixture on stiffness, where the stiffness rises from 10300 to 20100 N/m at 100 °c and from 9700 TO 10300 N/m at 200 °c on increasing the % addition from 7% to 35%.

Fig. (3-4) shows the effect of adding polyamide to the matrix, the effect on stiffness is similar to that shown in Fig. (3-3).

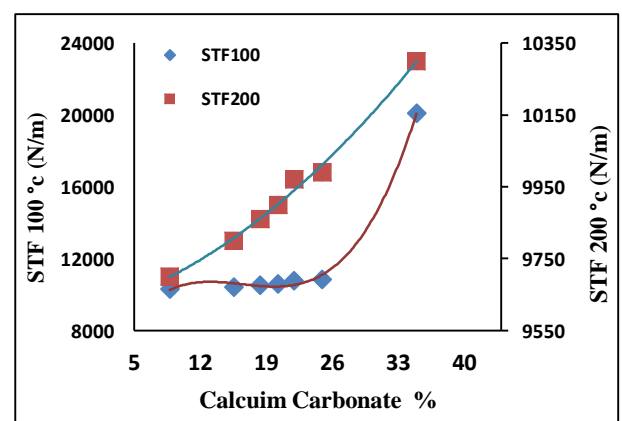


Fig. (3-3) Effect of Calcium Carbonate addition on Stiffness at Fixed Temperature (100-200) °c

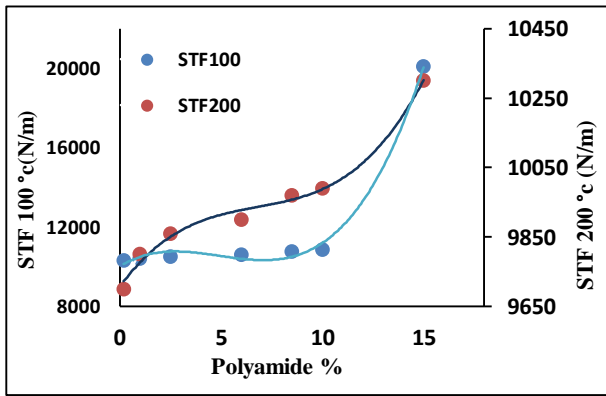


Fig. (3-4) Effect of Polyamide addition on Stiffness at Fixed Temperature (100-200) °c

### 3.3.4 TanDelta (tanδ)

The ratio between the energy viscous component and energy elastic component corresponds to the damping (tan δ) of a material. Tan δ can be defined as the dissipation of energy in a material under cyclic load and gives a measure of the capacity of a material to release energy. Fig. (3-5) and table (3-5) Show the effect of addition of kaolinite (KW%) to the matrix on the value of tanδ, which decreased the dissipation of energy in the sample S12 under cyclic load and lower the relaxation strength to be (0.253) for S12. This indicates that the conservation of energy higher than the other samples.

Table (3-5): Relaxation strength of PDMS Samples with Kaolinite

Formulation	Kaolinite %	Relaxation strength
S1	0	0.268
S3	1.5	0.264
S5	3	0.262
S7	5.2	0.260
S9	8	0.258
S11	12.4	0.256
S12	20	0.252

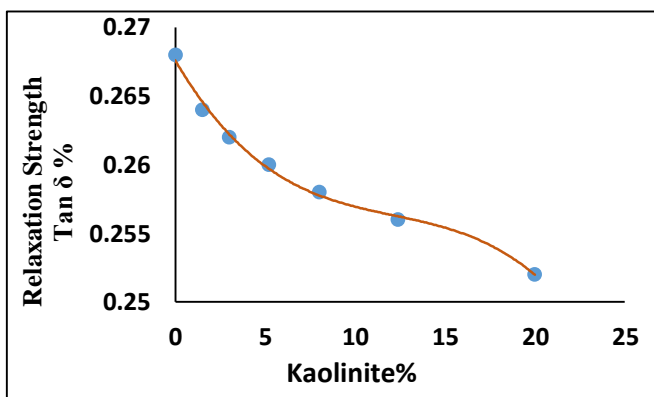


Fig. (3-5) Relaxation strength of PDMS Samples with Kaolinite

### 3.3.5 Shore (A) Hardness

Fig (3-6) shows the effect of adding Kaolinite (KW%) to PDMS on the hardness. From the figure, it obvious that the hardness increased almost linearly with the increasing of Kaolinite up to a max of (20 %), shore value illustrated in table (3-6).

Table (3-6) Shore A for PDMS

Samples	S1	S3	S5	S7	S9	S11	S12
Shore A	65	70	76	80	86	90	95
KW%	0	1.5	3	5.2	8	12.4	20

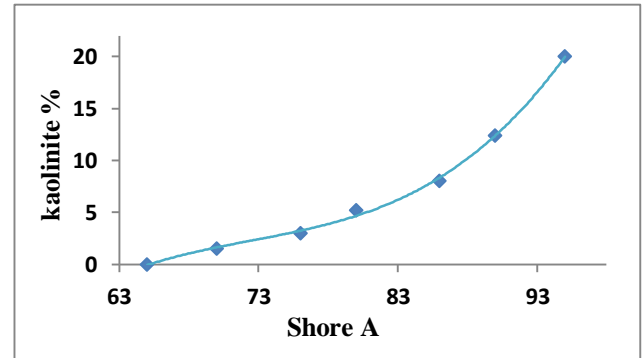


Fig. (3-6) Effect of Kaolinite addition (%) on Hardness

## 3. 4 Thermal Properties

### 3.4.1 Thermal Conductivity

The thermal conductivities of both pure and liquor PDMS were measured. The results are exhibited in Table (3-7) and Fig (3-7) from which it may be concluded that the thermal conductivity decreased to (0.122) (w/m°K) being achieved with S12 although Mix 7and Mix9 can give very close results.

Table (3-7): Thermal Conductivity of PDMS Samples

Samples	S1	S3	S5	S7	S9	S11	S12
ATO%	0.5	2	3	5	7	10	12
Thermal conductivity TC (w/m°K)	0.2	0.17	0.162	0.145	0.141	0.131	0.122

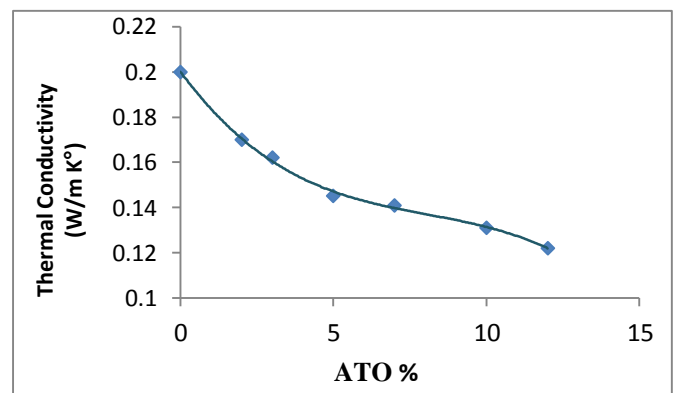


Fig. (3-7) The effect of adding ATO to PDMS on thermal Conductivity



### 3.4.2 Differential Scanning Calorimetry (DSC)

Differential scanning Calorimetry was used to study the phase transitions such as thermal decomposition, glass transition temperature, and specific heat behavior and decomposition temperature. The values of the formulated properties are listed in table (3-8).

The results illustrated from table (3-8) shown in Fig. (3-8) to (3-9) that the addition of mixture (ATH, ATO) % have a great effect on increasing the specific heat value respectively from (1.46 to 25.63) (j/g°C).

It is clear that decomposition temperature TD, specific heat Cp and melting point TM increase from mixture to another due to the combination effect of both the flame retardants (ATO, ATH) illustrated table (3-8). The maximum value is attained by respectively (12,16) %. where (Tg) decreased due to increase of Mica max value (40) % to 35.73°C.

Table (3-8): (Tg -T<sub>melt</sub>-T<sub>D</sub>-Cp) of PDMS Samples from DSC Curves

Sample	Mica %	ATO %	ATH %	Tg °c	TM °c	TD °c	Cp (j/g°C)
S1	0	0.5	0.5	130	210	302	1.46
S2	2	1	1	124.7	213	310	1.839
S3	2.5	1.5	1.5	79.75	215	315	3.023
S4	3	1.7	2	73.85	218	319	3.171
S5	3.5	2	2.5	58.70	220	321	7.229
S6	4	2.5	3	57.87	222	322	8.071
S7	5	3	4	56.87	224	324	9.199
S8	7	4	5	54.83	227	325	10.12
S9	8	5	7	48.98	230	327	10.82
S10	22	7	10	41.98	233	330	12.75
S11	32	10	13	40.02	238	335	18.75
S12	40	12	16	35.73	245	340	25.63

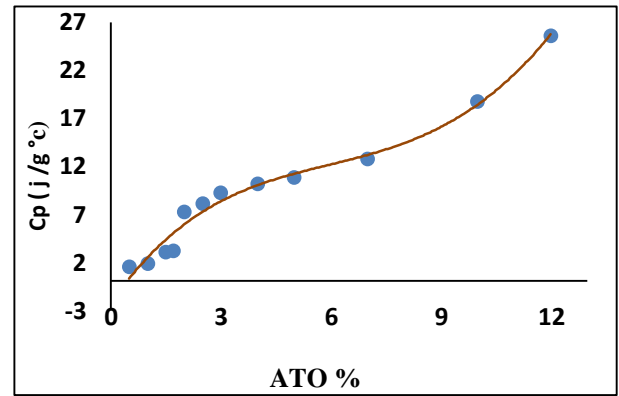


Fig. (3-9) Effect of Addition Antimony Trioxide on Specific Heat

### 3.4.3 Thermal Mechanical Analysis (TMA)

Thermal Mechanical Analysis (TMA) was introduced to study the thermal expansion behavior of (PDMS) samples in the presence of various additives. Fig (3-10).

we observed that on increasing the percentage of Mica% up to 15 %, the thermal expansion ratio% decreased from 5.59 to -268.69 % at 40% Mica. result shown in the table (3-9).

Table (3-9) Thermal Expansion Coefficient Ratio for PDMS

PDMS Samples	Mica%	TEC%
S1	0	5.59
S2	1	5.01
S3	2	4.6
S4	2.5	0.767
S5	3	-0.308
S6	4	-0.717
S7	7	-1.11
S8	10	-1.44
S9	15	-12.79
S10	22	-21.06
S12	40	-268.69

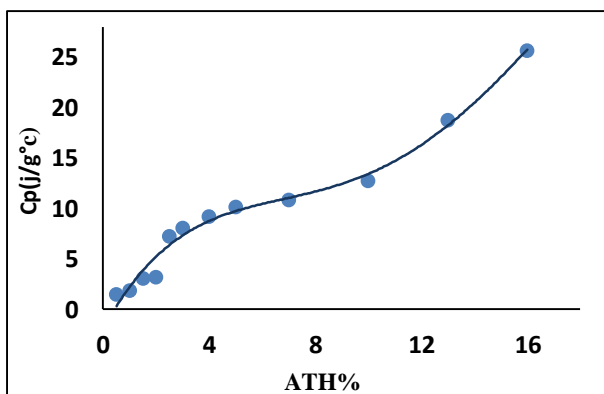


Fig. (3-8) Effect of Addition Alumina Trihydrate on Specific Heat

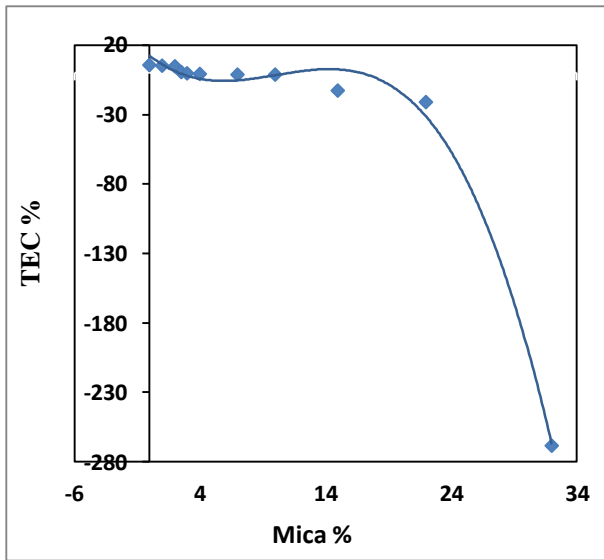


Fig. (3 -10) Thermal Expansion Coefficient Ratio for PDMS

#### 4 CONCLUSIONS

From the studies conducted, it can be concluded the following:

- 1- Addition Kaolinite% to polydimethylsiloxane liquor increases the mechanical properties, where, storage modulus increases from (175 to 1340) Mpa at 100°C and from (70 to 985) Mpa at 200°C, increasing the storage modulus by adding KW % up to 20 %. This Indicates the complete conservation of energy and the ability of the sprayable thermal insulation coat to conservation of energy at high temperature during application.
- 2- Addition CC (%) to PDMS liquor minimize the losses modulus from 90 (MPa) to 44 at 100 °c and from 85 (MPa) to 30 at 200 °c, respectively at constant temperature (100, 200) °c, means the high efficiency of the sprayable thermal insulation coating which conserve energy and not easy release it.
- 3- Addition mixture of (CC+PA) % to PDMS liquor increasing stiffness, from (10300 to 20100) N/m at 100°C and increasing stiffness from (9700 to 10300) N/m at 200 °c was attained at 35 CC % and 15%, beyond which stiffness increased marginally with CC % and PA% addition. Which mean high resistance to deformation and impact in response to an applied external force.
- 4- The difference in the value of Tanδ between samples are very close. where the addition of kaolinite decreased the dissipation of energy in the sample S12 under cyclic load. The lower relaxation strength result is (0.253) for S12. This indicates the conservation of energy higher than the other samples. It has a lower ability to release energy than other samples.
- 5- Addition of Kaolinite% to polydimethylsiloxane liquor has a major effect on hardness the hardness.

- 6- Addition of Antimony trihydroxide to PDMS liquor has a great effect in decreasing the thermal conductivity the least value being achieved with Mix 12 although Mix 7 and Mix 9 can give very close results.
- 7- Glass transition temperature decreased from (130 to 35.73) °c (T<sub>g</sub>) due to increase of MICA% max value (40) %.
- 8- Melting and Decomposition temperature increased respectively from (210-245) °c to (302-340) °c due to the effect of both the flame retardants (ATO, ATH) %.
- 9- Heat capacity increased from (1.46 to 25.63) J/g °c due to the effect of both the flame retardants (ATO, ATH) %.
- 10- Addition of Mica% to the PDMS liquor up to 15 %, the thermal expansion ratio% drops significantly from 5.59 to -12.79%, after which farther increase in Mica% leads to further decrease in expansion ratio % to -268.69 % at Mica% up to 40 %.
- 11- The improvement in the thermal and mechanical properties of PDMS liquor qualified to work as sprayable thermal insulation.

#### 5 LIST OF ABRIVIATION

- ASTM: American Society of Testing and Materials  
 ATH: Aluminum tri-hydrate  
 ATO: Antimony trioxide  
 AZC: Abo-Zabal Company for Specialty Chemicals  
 CC: Calcium carbonate  
 DMA: Dynamic mechanical analysis  
 Cp: Heat capacity  
 DSC: Differential scanner Calorimetry  
 KW: Kaolinite  
 Mix: Mixture  
 Mpa: Mega Pascal  
 PA: Polyamide  
 PDMS: polydimethylsiloxane  
 Q: heat flux  
 ST.M100 °c: Storage Modulus at 100°C  
 ST.M200 °c: Storage Modulus at 200°C  
 STF100 °c: Stiffness at 100°C  
 STF200 °c: Stiffness at 200°C  
 S: Sample  
 SP. G: Specific gravity  
 T<sub>g</sub>: Glass transition temperature  
 TD: Decomposition temperature  
 TCN: Thermal conductivity  
 TGA: Thermogravimetric analysis  
 TMA: Thermal mechanical analysis  
 Tan δ: Damping Coefficient  
 VOC: Volcanized organic compound  
 δ: Tan Delta parameter determine from dynamic mechanical analysis technique {Phase Angle:  $\delta = \arctan \ddot{E} / \dot{E}$  }.  
 ε<sub>0</sub>= is the amplitude of the strain.  
 δ<sub>0</sub>= Is the amplitude of the stress.

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