Flammability and Thermal Degradation Properties of Sprayable Polydimethyl Siloxane Coating

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Abstract- **Recent years, 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 sprayable coating.**
This paper introduces a background, which highlights the application of Polydimethylsiloxane as a thermal spray protection coating. The flammability behavior as well as kinetics of the degradation process are discussed. The activation energy for degradation will also be presented.
Where the mixed liquor Polydimethylsiloxane (PDMA) with Mica, flame retardant (ATO, ATH) is adding to withstand higher temperature than silicone rubber pure and to employ this technique as sprayable thermal insulation coating for metallic case of aircraft outer surface.

Key Words: Polydimethylsiloxane; Flammability; Thermal degradation; kinetic approach; Activation Energy

1 INTRODUCTION

Polydimethylsiloxane (PDMS) containing Si –O-Si main chain and methyl side groups processes many excellent properties, such as high flexibility, high hydrophobicity and excellent thermal stability. It is widely used in the aerospace, construction, electronics and automotive [1].

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.

Commercial development of Polysiloxanes occurred during World War II, when the need increased for a new breed of materials that could be suitable for a wide range of applications.

Polysiloxanes were used for waterproofing and sealant greases, as well as instrument damping liquids and thermoset engine gaskets [2].

Silicone rubber exhibits a list of excellent characteristics including biocompatibility, oxidation resistance, thermal stability, climate resistance. Because of the unique structure of Polysiloxanes. Therefore, it is made to high performance

thermal shielding, weather resistance coatings and used widely in plastics industry, automobile industry, mold and die industry, electronic industry. Silicones are greatly acknowledged for their better thermal and thermo-oxidative stabilities compared to most carbon-based polymers. This acute resistance against flame has put PDMS in the top list of polymers for applications at high temperature where flame appears [3].

But Polysiloxanes are macromolecules composed of repeating silicon–oxygen bonds (Si-O-) along the backbone.
The strong Si-O bond itself aids the thermal and oxidative stability of siloxanes. The bond energy of a Si-O bond (~107 kcal) is more than 20 kcal higher than that of C-C bonds (83 kcal) and C-O bonds (85 kcal). Therefore, the amount of thermal energy needed to break the Polysiloxanes backbone, under neutral pH conditions, is much greater than that of most polymeric materials (silicone rubber) [4].

Therefore, Polysiloxanes are one of the most unique materials available today. As a class of polymers, Polydimethylsiloxane display an unusually wide range of properties [5].

They can be viscous yet lubricating as liquids, while an apparently solid form can be either rigid or elastomeric. They are highly thermally and oxidatively stable, display a high degree of chemical inertness, show high UV, resistance have low surface energies, have good dielectric strength (making them good insulators), and have attractive physical properties over a wide range of temperatures [6].

They have shown the ability to withstand short term exposure to severe conditions > 400°C (in inert atmospheres) and extended exposures at200°C (in air) without any significant changes in their properties [7].

That is the main aim for using this new technique of thermal protection by sprayable coating to overcome problems using thermal insulation by adhesive technique. We used mixed sprayable liquor Polydimethylsiloxane coating (PDMA) as thermal protection coatings.

Adhesion has all been tried, but none has been found entirely satisfactory. Organic modified polysiloxanes are generally recognized as the newest generic class of high performance protective coating.

2 EXPERIMENTAL

Experiments were carried out in order to determine the optimum formulations and additives for the preparation of PDMS liquor as a thermal insulation coating for coating the metallic surface of an air craft body.

2.1. Spray Coating Equipment

Automatic Spraying Equipment with water purification system was used for mixing and spray coating of PDMS coating, an electric oven was used for curing range (80-130) °C.

2.1.1. Materials and Techniques
2.1.1.1 PDMS (SILIKPHEN P/80/X)
Chemical formula: PDMS Resin
$CH_3[Si(CH_3)_2O]_n Si(CH_3)_3$
Appearance: viscous Liquid.

2.1.1.2 Mica (Muscovite)
Chemical formula: $KA_{l2} (Al Si_3 O_{10}) (FOH)_2$ Grade V
Appearance: Crystal Color Ruby / Green

2.1.1.3 Antimony trioxide (ATO)
Flame retardant
Chemical formula: Sb_2O_3
Appearance: white powder

2.1.1.4 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 Thermal Gravimetric Analysis (TGA)

The thermal gravimetric analysis was carried out using a Shimadzu instrument Thermogravimetric Analyzer instrument (TGA-50) with platinum crucibles [8].

The tests are performed in a dynamic mode, going from room temperature to 1000°C. Experiments are carried out under nitrogen, with a flow rate of 20 ml/min in order to remove the evolved corrosive gases rapidly [9]. Heating rates of (5,10, 15and 20) $^{\circ}$C/min were used for selected samples [10].

2.2.2 Flammability Analysis

Limiting oxygen index and vertical flame test are widely used to evaluate flame retardant properties of materials and to screen flame retardant formulations.

2.2.2.1. Limiting Oxygen Index Measurements (LOI %)

The LOI test is probably the most well-known test for flammability. The limiting oxygen index is defined as the minimum percentage of oxygen that is required to maintain flaming combustion of a specimen under specified laboratory conditions. The apparatus applied is the Model HC-2 Flammability Unit Oxygen gas, Nitrogen gas, and precision pressure regulator systems. The applicability of using the oxygen index test (ASTM D 2863-76) to obtain an indication of the relative flammability of fire-retardant-treated PDMS mixtures polymer was investigated. The limiting oxygen index apparatus is designed to allow a candle like burning of the specimen in a slowly rising mixture of oxygen and nitrogen. In the test, a specimen is placed in the holder at the center of the base of the test column. The flow valves are adjusted to obtain the desired initial oxygen concentration and total flow rate. The Oxygen Index, in percent, is calculated from the final oxygen concentrations tested [11].

$$\text{Limit oxygen index (\%)} = \frac{100 * (\text{Volumetric Flow of Oxygen})}{(\text{Total Volumetric Flow of Oxygen and Nitrogen})}$$

2.2.2.2 Vertical Flame Test (UL94V)
The apparatus used is the Vertical Testing Model (CZF-1), indicating the vertical ratings requirements (V-0, V-1, V-2). In the mentioned test a specimen is supported in a vertical position and a flame is applied to the bottom of the specimen. The flame is applied for ten seconds and then removed until flaming stops at which time the flame is reapplied for another ten seconds and then removed.

Two sets of five specimens are tested. The two sets are conditioned under different conditions. Test is run with bars one half inch wide and five inches long. These are held vertically and exposed to a laboratory burner flame three quarters of an inch high. Each sample is ignited for ten seconds, the flame allowed to go out, and ignited for a second time often seconds [12].

3 RESULT AND DISCUSSION

3.1 FORMULATIONS
Thermal and flammability properties of various PDMS coating mixtures formulations have been recorded and extensively discussed. Kinetics of thermal degradation has been studied and the activation energies of degradation of the specimens under investigation by using two different kinetic methods (Kissinger-AKahira-Sunose Method, Flynn-Wall-Ozawa). The samples of different compositions are illustrated in table (3-1).

Table (3-1) Different PDMS Mixtures

MICA%	0	2	2.5	3	3.5	4	5	7	8	22	32	40
ATO %	0.5	1	1.5	1.7	2	2.5	3	4	5	7	10	12
ATH %	0.5	1	1.5	2	2.5	3	4	5	7	10	13	16

3. 2 Thermogravimetric Analysis (TGA)
Thermal analysis for different formula of PPDMS thermal protection coating mixtures was conducted using (TGA - 50) analysis as previously mentioned. The degradation for each sample was measured at three rates (5,10, 15, 20 °C/min). Figs. (3-1) to (3-7) illustrate the TGA curves for different samples.

PDMS exhibits mainly a 4-step degradation behavior, involving different sections It was shown that PDMS thermally decomposes to cyclic oligomers through Si–O bond scission in a chain-folded cyclic conformation energetically favored by overlapping of empty silicon d-orbitals with orbitals of oxygen and carbon atoms.

Degradation for PDMS samples starts at 340°C and ends at 950°C for (PDMS-S1 to PDMS- S5). gradual improvement was noticed on adding the mixture flame retardant (ATO, ATO) the maximum improvement was noticed by sample (PDMS –S6) as observed in Fig. (3-6).

remarkable improvement was noticed on adding (Mica) 40 % as indicate by sample (PDMS-S7) which has a high thermal stability Fig. (3-7).

Fig. (3 -3) TG Curves of PDMS-S3 at Different Heating Rates
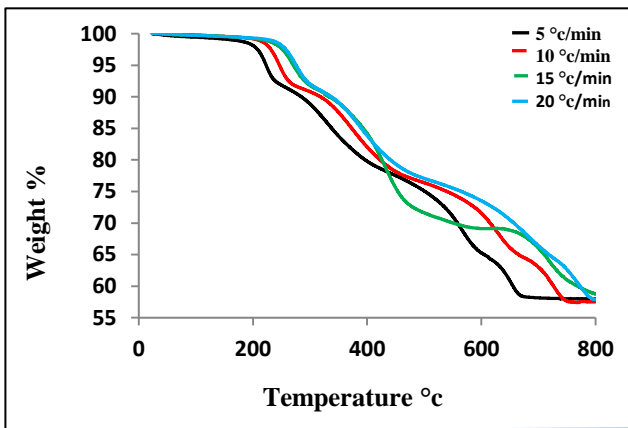

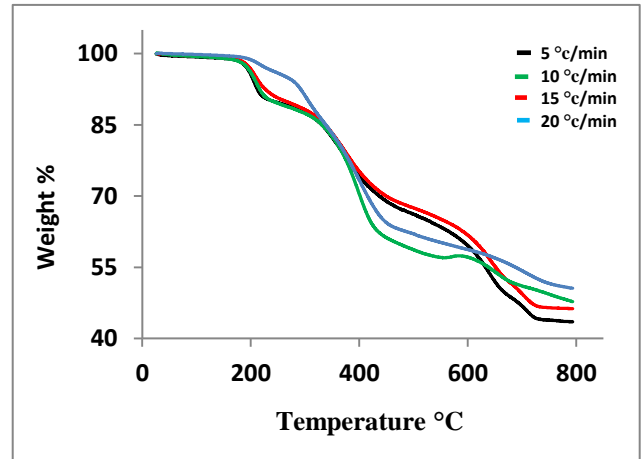Fig. (3 -1) TG Curves of P PDMS –S1 at Different Heating Rates

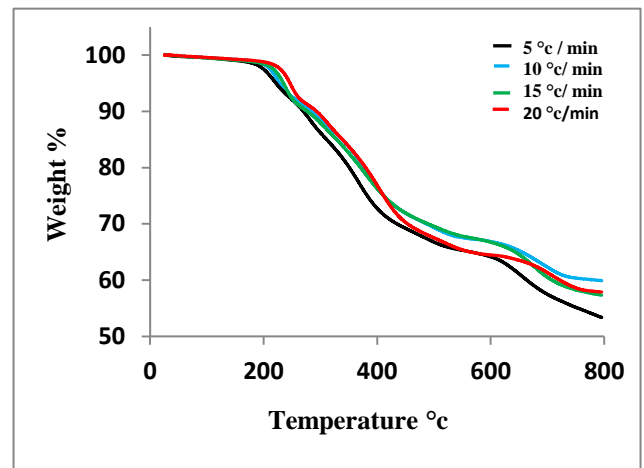Fig. (3 -4) TG Curves of PDMS –S4 at Different Heating Rates

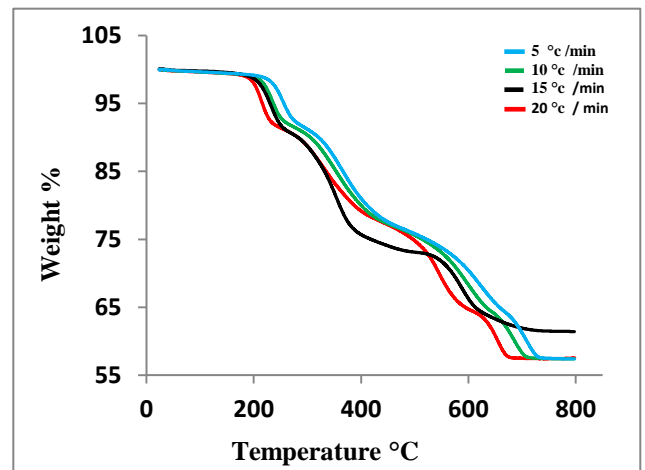Fig. (3 -2) TG Curves of PDMS-S2 at Different Heating Rates

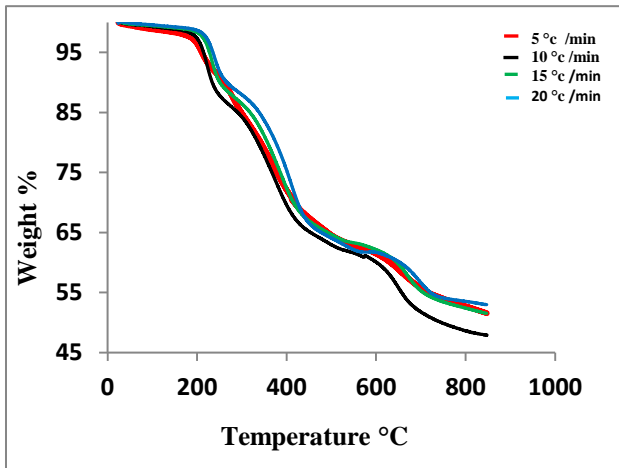Fig. (3 -5) TG Curves of PDMS –S5 at Different Heating Rate

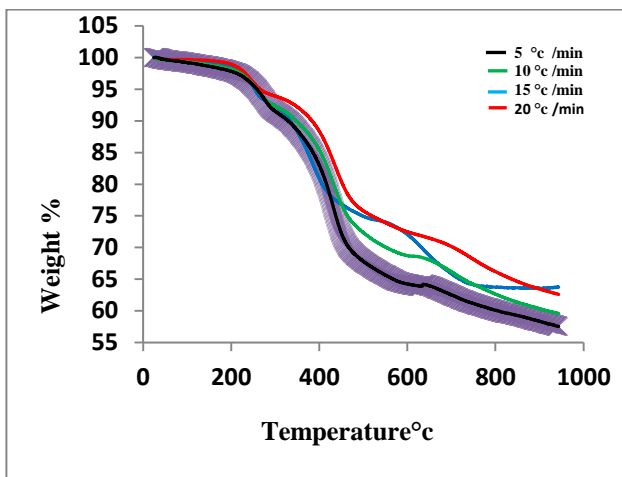Fig. (3 -6) TG Curves of PDMS –S6 at Different
Heating Rate

Fig. (3 -7) TG Curves of PDMS –S7 at Different
Heating Rates

3.3. Modeling of degradation kinetics
3.3.1 Kinetics of Thermal Degradation of PDMS Samples

In this section, the activation energies of degradation of PDMS samples were evaluated using Flynn-Wall-Ozawa and Kissinger-AKahira-Sunose Method. At first, a brief introduction to the kinetic methods is given here-in-after to define the degree of degradation (Conversion) ($\mathcal{X}$).

Where ($\mathcal{X}$) is given by equation (3.1) [13].

$$x = \frac{w_0 - w}{w_0 - wf} \qquad \dots\dots\dots\dots\dots (3.1)$$

Where W_0, W, and W_f are the initial, instantaneous, and final weight of the sample during the degradation process, respectively.

For non-isothermal degradation, the degradation rate (d $\mathcal{X}$/dt), can be generally described as:

$$\frac{dx}{dt} = K(T) f(x) \qquad \dots\dots\dots\dots\dots (3.2)$$

f (x): is a general differential function of degradation, depending on the degradation reaction mechanism.

x: Conversion (**is** a dimensionless quantity).

d x /dt**:** Rate of Conversion (Sec^{-1}).

k (T): is the temperature-dependent rate constant

which often has Arrhenius-type dependence:

$$k(T) = A\exp\left(\frac{-Ea}{RT}\right) \qquad \dots\dots\dots\dots (3.3)$$

Where:

A**:** Is pre-exponential factor (having a unit of inverse time).

Ea=E**:** Apparent activation energy (KJ/mole).

R**:** Gas constant.

T**:** Absolute temperature.

Using equation (3.2) and introducing the linear heating rate $\beta = dT/dt$, equation (3.3) can be rewritten as:

$$\frac{dx}{dt} = \beta \frac{dx}{dt} = A \exp\left(\frac{-Ea}{RT}\right) F(X) \qquad \dots\dots\dots\dots (3.4)$$

Equation (3.4) assumes that the three parameters (Ea, A, f($\mathcal{X}$) describe a chemical or physical change, irrespective of its complexity. Starting from equation (3.4) various kinetic evaluation methods have been developed [14].

If the activation energy depends on $\mathcal{x}$, the use of various iso-conversional methods could lead to various activation energies for a given degree of degradation. In this study, two iso-conventional methods were used to evaluate the activation energy, namely Flynn-Wall-Ozawa and Kissinger-AKahira-Sunose.

Flynn-Wall-Ozawa method, Plots of $\log \beta$ versus 1/T in this method were used to calculate the activation energy by measuring the slope of a straight line [15].

Result illustrated in table (3-3) and fig (3-8) to fig. (3-13). A plot of lnβ versus 1/T the straight line of slope (-1.052Ea/R) obtained from thermograms for several heating rates. The slope can be used to evaluate the apparent activation energy, as shown by equation (3.5).

$$\ln\beta = \ln\frac{AE}{R\,g(x)} - 5.331 - 1.052\frac{E}{RT} \qquad \dots\dots (3.5)$$

Kissinger-AKahira-Sunose method, the Eq. (1) can be shown as follows:

$$\frac{d(x)}{f(x)} = \frac{A}{\beta} * \exp\left(-\frac{E}{RT}\right) dT \qquad \dots\dots\dots (3.6)$$

Which is integrated with the initial condition of x =0 At T = t$_o$

Following expression:

$$g(x) = \int_0^x \frac{dx}{f(x)} \frac{A}{\beta} \int_{T_0}^T \exp\left(-\frac{E}{RT}\right) dT = \frac{AE}{\beta R} P\left(\frac{E}{RT}\right) \qquad \dots\dots\dots\dots (3.7)$$

Since, essentially the technique assumes that the A, f (x) and E are independent of T, while A and E are independent of x. Result illustrated in table (3-4) and fig (3-14) to fig. (3-18).

The KAS method is based on the Coats-Redfern approximation [16].

$$\ln \frac{\beta}{T^2} = \ln \frac{AR}{Eg(x)} - \frac{E}{RT} \qquad \ldots \ldots (3.8)$$

Thus, the plot Ln β/ T^2 vs. $1/T$ for a constant value of x should be a Straight line whose slope can be used to evaluate the apparent activation Energy [17].

3.3.2 Pre-Exponential Factor Calculation

From equation (3.2) by putting f (x)=(1-x) we get: -

$$\frac{d(x)}{d(t)} = A\, e^{-\frac{E}{RT}} (1 - X) \qquad \ldots\ldots\ldots (3.9)$$

Where: (1-x) is the dimensionless amount of reactive polysiloxanes remaining.

$$\frac{d(x)}{(1-x)} = Ae^{-E/RT} * d(t) * \frac{d(T)}{d(T)} \qquad \ldots\ldots (3.10)$$

Where: - $\qquad \frac{dt}{dT} = 6 \qquad$ for β=10 °c/min

Then Equation (3.10)

$$\frac{dx}{(1-x)} = 6A\, e^{-E/RT}dT \qquad \ldots\ldots\ldots (3.11)$$

By integration both sides of equation (3.10) we get: -

$$\int_0^{0.1} \frac{dx}{(1-x)} = 6A \int_{T0}^{T1} e^{-E/RT}dT \ldots\ldots\ldots (3.12)$$

we repeated the integration for conversion (0.1,0.2,0.3,0.4,) and different T. we can get the value of Pre-Exponential Factor (A) [18].
For different PDMS coating samples by using MATLAB program and the results are illustrated in table (3-2).
MATLAB program shown as follows:

```
Clc
T = [283 325 430 565 650]; % Put values of T0, T1, T2,
T3 and T4
X = [0 0.1 0.2 0.3 0.4]; % Put values of X
E = 29.25;      % Put value of E
R = 8.314;
m = length(T) - 1;
for i = 1:m
   fun1 = @(x) exp(-E./(R*x));
   P(i) = integral (fun1, T(i), T(i+1));
   fun2 = @(x) 1. /(1-x);
   Q(i) = integral (fun2, X(i), X(i+1));
End
```

```
S = ([m sum(P); sum(P) sum(P.*P)]) \[sum(Q);
sum(P.*Q)];
plot (P, Q);
A = S (2)/6
```

Table (3-2): Value of Pre-Exponential Factor for Different Samples

Conversion x	0.1	0.2	0.3	0.4	E_a	A_{app} (s^{-1})
		Temp °C			Kj/mole	
S1 Rate β=10 °C\min	325	430	565	650	29.25	0.3898 x10^5
S2 Rate β=10 °C\min	330	455	570	----	55.65	0.2802 x10^4
S3 Rate β=10 °C\min	325	428	620	720	63.56	0.5714 x10^6
S4 Rate β=10 °C\min	315	422	577	675	71.03	0.2082 x10^5
S5 Rate β=10 °C\min	230	395	450	615	72.54	0.4543 x10^6
S6 Rate β=10 °C\min	325	375	410	510	73.12	0.7143 x10^5
S7 Rate β=10 °C\min	305	380	450	725	73.66	0.2351 x10^5
S8 Rate β=10 °C\min	256	375	422	475	85.49	0.6157 x10^5
S9 Rate β=10 °C\min	275	399	550	650	98.25	0.6242 x10^5
S10 Rate β=10 °C\min	240	375	475	658	99.14	0.4851 x10^5
S11 Rate β=10 °C\min	285	345	424	610	103.74	0.4343 x10^5

Table (3-3): Activation Energy for Thermal Degradation of different PDMS Samples as Calculated by Flynn-Wall-Ozawa Method

Conv	S1		S2	
X	Ea	R^2	Ea	R^2
0.1	33.93	0.9093	76.28	0.9561
0.2	26.37	0.9869	67.30	0.9751
0.3	19.16	0.9001	40.655	0.9761
0.35	22.74	0.9442	38.37	0.9623
0.4	33.23	0.9909	-----	-------
0.45	40.10	0.9781	------	------
Ea$_{total}$	29.25		55.65	(KJ/mole)

Conv	S3		S4	
X	Ea	R^2	Ea	R^2
0.1	61.09	0.9503	117.28	0.8043
0.2	71.68	0.854	64.86	0.991
0.3	77.27	0.9446	61.94	0.9049
0.35	88.34	0.991	71.59	0.9807
0.4	69.96	0.8457	40.08	0.9408
0.45	------	------	157.19	0.677
Ea$_{total}$	73.66		85.49	(KJ/mole)

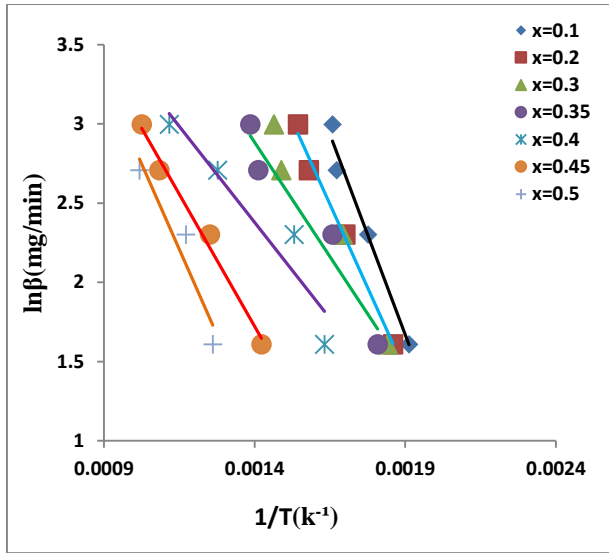| Conv	S5		S6	
X	Ea	R^2	Ea	R^2
0.1	111.86	0.8058	136.83	0.9968
0.2	44.52	0.9957	132.12	0.9995
0.3	89.15	0.940	215.56	0.9408
0.35	40.12	0.8827	67.33	0.8797
0.4	310.36	0.7875	43.54	0.945
0.45	26.42	0.9633		
Ea$_{total}$	103.74		119.08	(KJ/mole)

Fig. (3-8) Plot of lnβ versus 1/T for PDMS S1 (FWO)
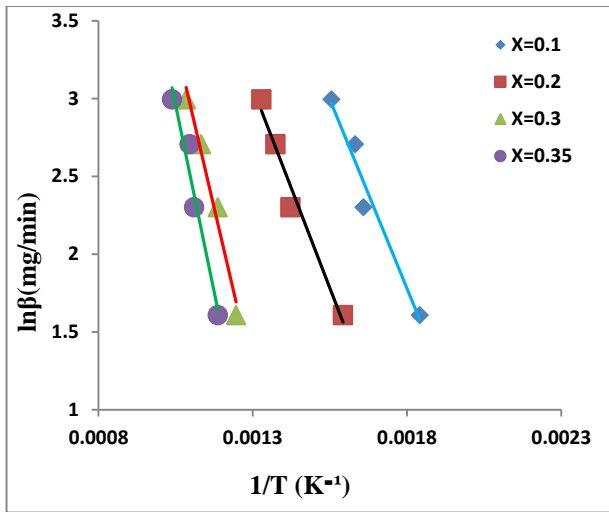

Fig. (3-9) Plot of lnβ versus 1/T for PDMS S2 (FWO)

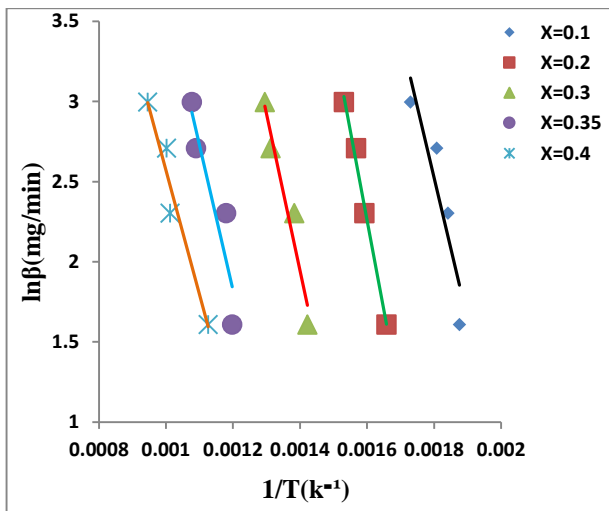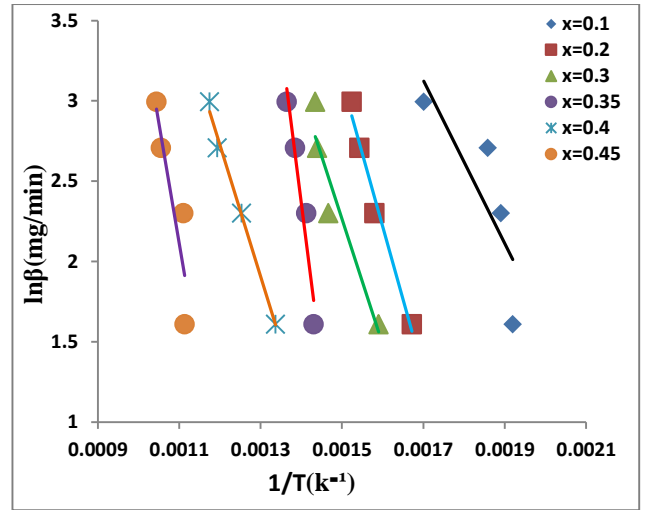Fig. (3-10) Plot of lnβ versus 1/T for PDMS S3 (FWO)
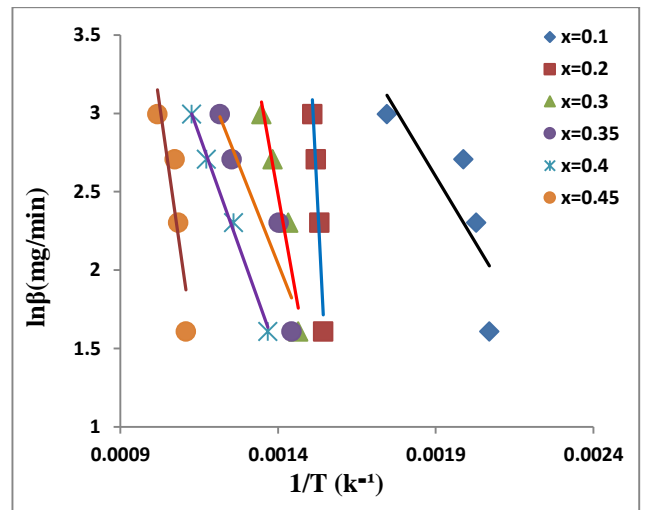

Fig. (3-11) Plot of lnβ versus 1/T for PDMS S4 (FWO)
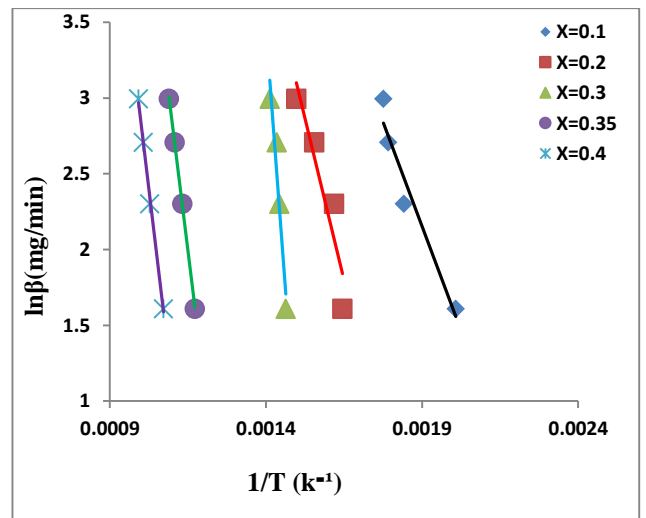

Fig. (3-12) Plot of lnβ versus 1/T for PDMS S5 (FWO)

Fig. (3-13) Plot of lnβ versus 1/T for PDMS S6 (FWO)

Table (3-4): Activation Energy for Thermal Degradation of different PDMS Samples as Calculated by Kissinger-AKahira-Sunose method

samples	Activation Energy (E$_a$) (KJ/mole)			
S1	85.43	50.21	---------	54.06
S2	112.96	76.55	62.65	61.85
S3	76.97	58.17	98.58	142.62
S4	93.57	112.82	---------	260.76
S5	123.38	168.26	---------	194.28
S6	136.0	140.65	---------	241.75

Table (3-5): Peak Temperature for Thermogravimetric Analysis (TGA) PDMS Mixture as Calculated by Kissinger-AKahira-Sunose Method

samples	Peak Temperature (^{0}k)			
	P1	P2	P3	P4
S1/β5	459.36	609.1	-------	843.53
S1/β10	467.68	623.47	-------	873.8
S1/β15	480.14	669.56	-------	925.96
S1/β20	485.14	671.11	-------	973.4
S2/β5	487.35	609.43	818.16	926.99
S2/β10	505.48	631.36	869.32	931.39
S2/β15	520.91	633.43	--------	956.23
S2/β20	528.7	664.82	--------	974.91
S3/β5	494.33	603.97	837.47	923.76
S3/β10	501.23	623.05	852.89	933.52
S3/β15	519.11	631.03	-------	939.8
S3/β20	525.16	668.84	908.2	978.9
S4/β5	493.39	641.62	--------	928.98
S4/β10	498.45	649.93	--------	946.38
S4/β15	508.52	656.55	--------	967.22
S4/β20	513.81	668.84	--------	976.63
S5/β5	470.03	643.9	--------	916.21
S5/β10	475.25	662.8	--------	949.4
S5/β15	482.05	663.55	--------	950.16
S5/β20	488.9	676.4	--------	952.43
S6/β5	490.37	649.18	--------	904.8
S6/β10	509.74	656.26	--------	907.7
S6/β15	523.92	662.87	--------	908.65
S6/β20	517.59	678.32	--------	914.41

Fig. (3-14) Plot of lnβ/T^2 versus 1/T for PDMS S1 (KAS)

Fig. (3-15) Plot of lnβ/T^2 versus 1/T for PDMS S2 (KAS)

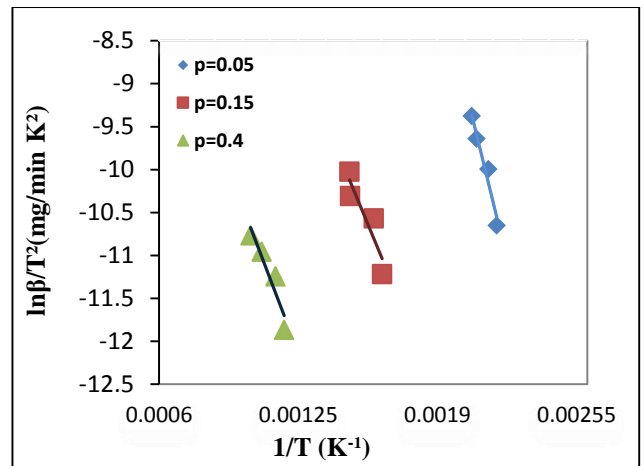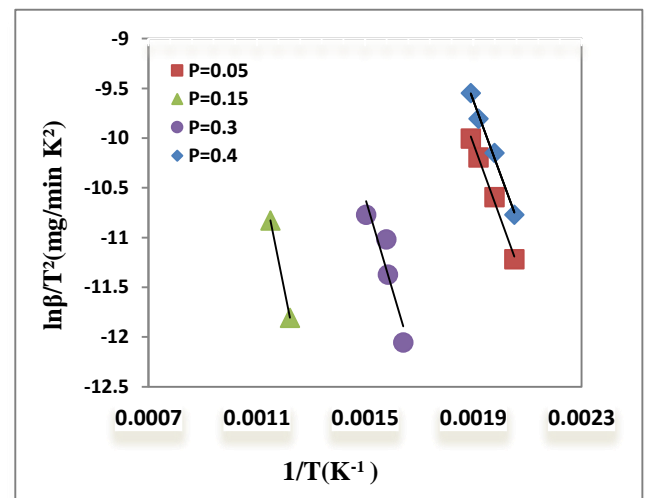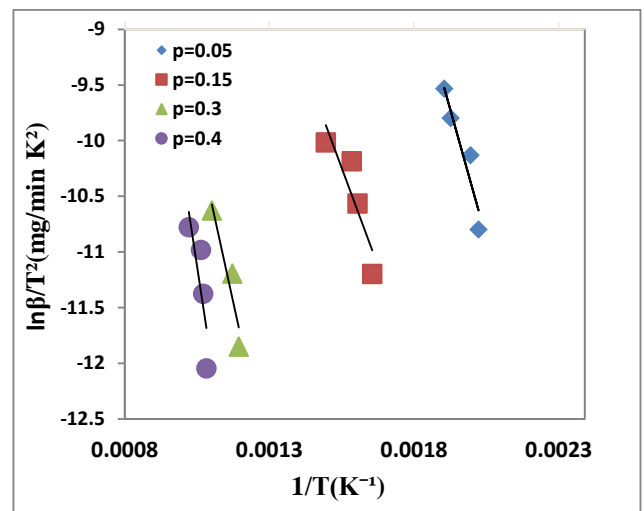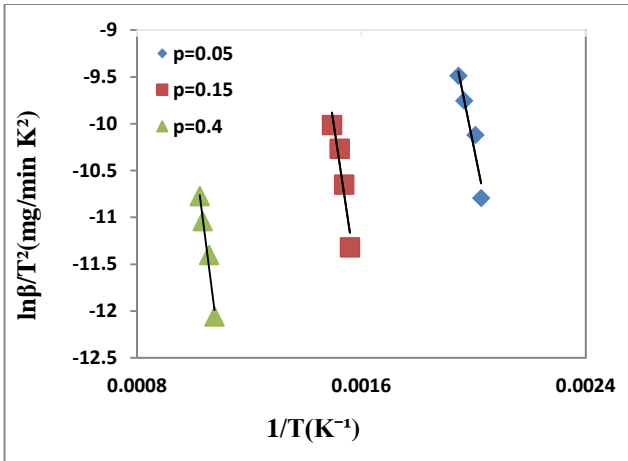Fig. (3-15) Plot of lnβ/T^2 versus 1/T for PDMS S3(KAS)

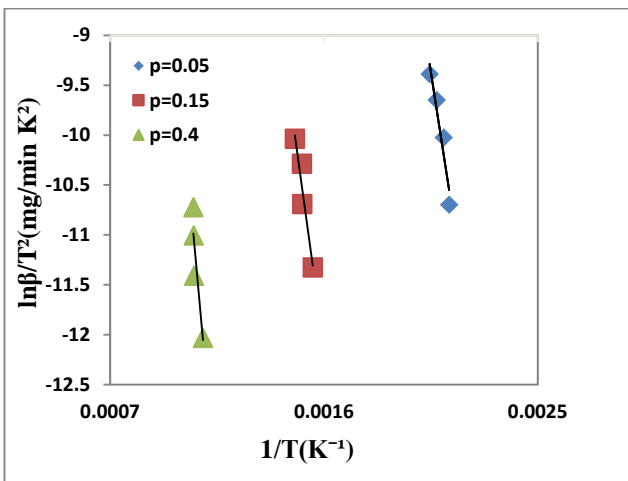Fig. (3-16) Plot of $\ln\beta/T^2$ versus $1/T$ for PDMS S4(KAS)

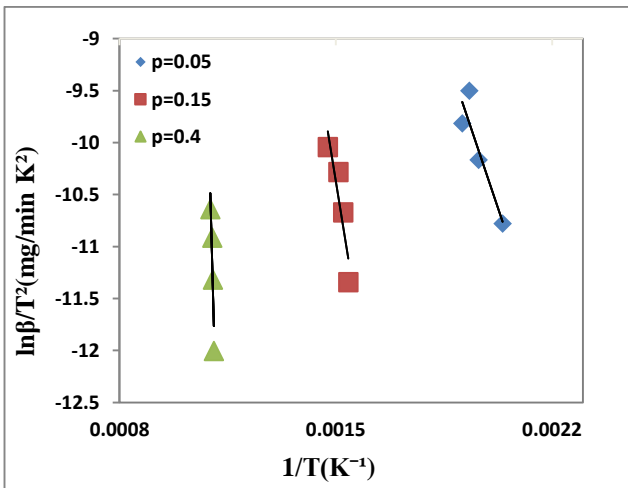Fig. (3-17) Plot of $\ln\beta/T^2$ versus $1/T$ for PDMS S5(KAS)

Fig. (3-18) Plot of $\ln\beta/T^2$ versus $1/T$ for PDMS S6(KAS)

3.3 Flammability properties of PDMS samples

Flammability properties such as Limiting Oxygen index (LOI %) as well as UL94V Test are measured.

3. 4.1 Limiting Oxygen Index (LOI %)

We obviously found that (LOI %) decreased by the addition of mixture of (mica) %, up to a maximum

respectively of (40) %, illustrated by Fig. (3-19). The results are tabulated in Table (3-6).

Table (3-6): Limiting Oxygen Index (LOI %) of PDMS

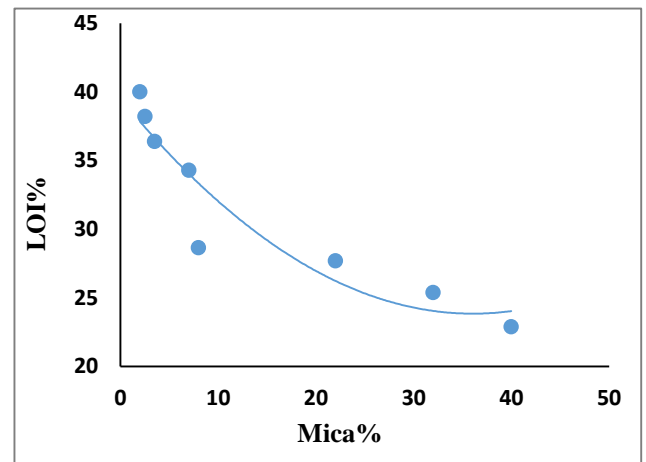| Sample code	1	2	3	4	5	6	7	9
LOI%	40	38.2	36.4	34.3	28.6	27.7	25.4	22.9
Burning Period (mm)	10	14	18	20	29	31	33	35
Length Burnt (mm)	21	22	23	25	37	40	43	46
Response + or -	+	+	+	+	+	+	+	+
Mica%	2	2.5	3.5	7	8	22	32	40

Fig (3 - 19) Limiting Oxygen Index (LOI %)

3.4.2 UL94V Test of PDMS

It observed that the value of UL94V test vary from V-1 to V-0, which indicate that the flammability properties of PDMS samples increased by addition of flame retardant mixture (ATO+ATH) and the results tabulated in Table (3-7).

Table (3-7): UL94V of PDMS Samples

Sample code	Rating UL94V	Flame Dropping	Thickness (mm)	Total Flaming time	Maximal Flaming time
1	V-2	Yes	3.2	254.4	56.5
2	V-2	Yes	3.2	252.5	28.3
3	V-1	No	3.2	120	18.5
4	V-0	No	3.2	52.2	11.8
5	V-0	No	3.2	50.4	11.1
6	V-0	No	3.2	31.5	10.2
7	V-0	No	3.2	30.2	9.3
8	V-0	No	3.2	28.1	8.4
9	V-0	No	3.2	25.2	7.5

4 CONLUSION

In this study the conclusion can be summarized as follows:

1. The degradation of PDMS takes place in 4-steps.
2. Degradation for PDMS samples starts at 340°C and ends at 950°C for (PDMS-S1 to PDMS- S5). improvement gradually noticed on adding the mixture flame retardant (ATO, ATO) %, for samples (PDMS-S6).
3. The highest resistance to thermal degradation is notice on adding (Mica) 40 % for (PDMS-S7) indicate a high thermal stability at high temperature.
4. An increasing in activation energy from (29.25 to 119.08) Kj/mole was noticed on adding (Mica%) using Flynn-Wall-Ozawa method.
5. The thermal stability of the PDMS samples, were enhanced by addition of flame retardant mixture (ATO+ATH) % as revealed by UL94V to V0.
6. Flammability improvement achieved by addition of flame retardant mixture (Mica) % which decreased the value of Limiting Oxygen Index (LOI %) of PDMS from (40 to 22.9) %.
7. PDMS as a sprayable thermal insulation for metallic case of aircrafts is become a simpler coating application than using adhesive thermal insulation application, because of it is high thermal, flammability stability and can applied in difficult surfaces.

5 LIST OF ABRIVIATION

KAS: Kissinger-AKahira-Sunose method

Clc: MATLAB calculating symbol

PDMS: Polydimethyl siloxane

FWO: Flynn-Wall-Ozawa kinetic method

ATO: Antimony trioxide

ATH**:** Aluminum tri-hydrate

ASTM: American Society of Testing and Materials

AZC:Abo-Zabal Company for Specialty Chemicals

LOI %: Limiting Oxygen Index

TGA: Thermal Gravimetric Analysis

UL94V: Vertical flame test

R^2**:** Root mean square

S: Sample number

UV: Ultraviolet

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