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IMPROVEMENT OF THE PROPERTIES OF INSULATING POLYMERS USING ARAMID FIBER FOR SOLID ROCKET MOTOR INSULATION

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ABSTRACT

Development and characterization of asbestos-free rubbers for use as rocket motor insulators are presented. Such insulation is based on aramid fiber in the pulp form as reinforcement for Ethylene Propylene Diene Monomer (EPDM) in the liquid form.

Aramid fiber (Kevlar) in the pulp form has been used and characterized as EPDM filler material. This method permits manufacturing EPDM rocket motor insulation in which Kevlar pulps are dispersed and immobilized in the EPDM polymeric matrix. A detailed description and procedure for the mixing cycle is explained. The curing methodology was justified. Kevlar pulp/EPDM has been shown to exhibit better thermal properties than its asbestos containing counterpart. Its thermal characteristics however fall short of some of the best asbestos insulators.

Insulation material description, processing and manufacturing techniques are discussed along with thermal properties (effective thermal conductivity, effective specific heat capacity and effective thermal diffusivity). The physical and mechanical properties (density, hardness, tensile strength and elongation) of different compositions were obtained. The ablation resistance was measured. Thermo-gravimetric analyses versus Kevlar pulp content, as well as a differential scanning calorimetry are discussed. The effect of changing Kevlar pulp volume fraction was studied. Also a comparison of Kevlar pulp/EPDM's performance (both thermally and mechanically) with other rocket motor insulating materials was done.

Kevlar pulp filled EPDM has been shown to exhibit better thermal, mechanical, physical and ablative properties than its asbestos containing counterpart.

KEYWORDS

Composites for aerospace.

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NOMENCLATURE

AP	Ammonium Polyphosphate
C _p	Specific Heat, J/kg.°K
DSC	Differential Scanning Calorimetry
EPDM	Ethylene Propylene Diene Monomer
K	Thermal Conductivity, W/m .°K
KP	Kevlar Pulp
PCA	Peroxide Cross linking Agent
Phr	Part per hundred ratio
TGA	Thermo Gravimetric Analysis

INTRODUCTION

Solid rocket motors typically include an outer case or shell that houses solid propellant grains. The rocket motor case is conventionally manufactured from a rigid, yet durable, material such as steel or filament-wound composite. The propellant is housed within the case and is formulated from a composition designed to undergo combustion and thereby produces the requisite thrust for attaining rocket motor propulsion. Internal insulation in a solid rocket motor is a layer of heat-barrier material placed between the internal surface of the case and the propellant. [1-3] The primary function of internal insulation is to prevent the rocket motor case from reaching temperatures that may endanger its structural integrity. Typically, the insulation is bonded to the inner surface of the case and is generally fabricated from a composition capable of withstanding the high temperature gases produced when the propellant grains burn. The combustion of solid rocket propellant generates extreme conditions within the case of the rocket motor. For example, temperatures inside the rocket motor case typically reach 2,760° C (5,000° F.), and interior pressures may exceed 1500 psi (10.35 MPa). These factors combine to create a high degree of turbulence within the rocket motor case. In addition, particles are typically entrained in the gases produced during propellant combustion. Under the turbulent environment, these entrained particles can erode the rocket motor insulation. If the insulating layer and liner are pierced during rocket motor operation, the casing is susceptible to melting or degradation, which can result in failure of the rocket motor. Thus, it is crucial that insulation compositions withstand the extreme conditions experienced during propellant combustion and protect the case from the burning propellant. [4-5]

JUSTIFICATION OF THE WORK

Characterization of reinforced rubbers for use as rocket motor insulators is presented, One such insulation is aramid fiber (Kevlar) in the pulp form (KP) [6] filled Ethylene Propylene Diene Monomer (EPDM) in the liquid form which is peroxide cured (PCA) and contains ammonium polyphosphate flame retardant agent (AP). Different formulations of Kevlar pulp with EPDM polymer were prepared to investigate the physical, mechanical and thermal properties. The difference in the formulations is the Kevlar pulp phr content (10-30 phr). The formulations were mixed using C.B. Bra bender mixer equipped with two sigma blades to attain uniform

dispersion of the Kevlar pulp. The formulations were cured under a press ($T = 170$ °C, $P = 28$ tons). Sheets with dimensions (300 x 150 x 3 mm, 300 x 150 x 1 mm) were made for investigation of the physical, mechanical, thermal and ablative properties.

PHYSICAL AND MECHANICAL PROPERTIES

According to ASTM the physical and mechanical properties were measured. A list of physical and mechanical properties is shown in Table (1) and Figures (1), (2).

THERMAL PROPERTIES

The determination of the thermal diffusivities of the cured thermal insulation compositions was done by the Nanoflash technique. [7] Using this method, the front side of a plane – parallel sample with a well defined thickness is heated by a short light or laser pulse. The resulting temperature rise on the back surface is measured versus time using an infrared detector. Analyzing the measured detector signal with appropriate mathematical models yields information on various thermophysical properties of the material. This satisfies the requirements for measurements of thermal diffusivities according to ASTM E 1461. Fig 3 shows the main components of the nanoflash instrument.

The determination of the specific heat capacities was done by doing differential scanning calorimetry (DSC). [8] The DSC Heat Capacity analysis calculates the actual specific heat capacity at any temperature in the DSC scan. The measurement is made by heating a test specimen at a fixed rate over a designated temperature range, where the specimen is held in thermal equilibrium before and after dynamic heating. The heat flow obtained from the specimen is recorded as a function of the actual sample temperature. This heat flow, normalized to the specimen mass and heating rate, is directly proportional to the specimen's specific heat capacity. To obtain specific heat capacity analysis we need three DSC graphs: a sample, a baseline, and a reference. The baseline data is used for baseline subtraction from the reference and sample data. Typically sapphire is used as the reference material.

With using sapphire calibrant as a reference and do DSC for base line (without sample). DSC of the material samples was done using samples around 8 mg. We obtained the resultant curves which relate the heat flow for every material composition with temperature for base line, sapphire calibrant, 10, 30 phr KP, compositions. These are shown in Fig 4, 5, 6, 7 respectively where only DSC curves for 10, 30 KP content compositions were selected for representation.

To calculate the thermal conductivity K of the cured thermal insulation compositions, the formula $K = \rho \cdot A \cdot C_p$ is used where ρ is the density of the material, A is the thermal diffusivity of the material and C_p is the specific heat capacity of the material. A list of thermal properties is shown in Table (2) and Fig. (8), (9).

THERMAL DEGRADATION

Another important aspect of the characterization of the material compositions includes defining the primary reactions in the decomposition of the material. [9] TGA (Thermo gravimetric) analysis is a very useful tool in establishing the primary reactions that occur. The data are useful in obtaining decomposition temperatures of the whole material composition. TGA of the insulation samples was done using samples of around 12 mg in a temperature range from 25 °C to 1000 °C with a heating rate 40 °C/min. The resultant curves which relate the weight % for every insulation composition with temperature for 10, 30 phr KP compositions are shown in Fig. 10, 11 respectively where only TGA curves for 10, 30 KP content compositions were selected for representation.

The TGA tests indicate for all compositions that an initial decomposition temperature for EPDM (matrix) occurs around 420 °C and the final decomposition is at 547 °C where EPDM decomposes to carbonaceous residue of free carbon. These provide a net effect of strong carbon based char which is highly erosion resistant. Also the tests indicate that an initial decomposition temperature for ammonium polyphosphate (flame retardant agent) occurs around 548 °C and the final decomposition is at 692 °C. In addition TGA for the individual constituents (KP, AP and EPDM) was done alone. The TGA curve for KP alone is shown in Fig. 12.

The only stable ingredient above 1000 °C is Kevlar pulp which is stable up to 1450 °C, in addition to the carbon based char remains from decomposition of EPDM. Not all 100 % of Kevlar pulp will be stable up to 1450 °C but up to 80 % of it decomposes which represents all the atoms inside the structure of Kevlar except carbon atoms. These 20 % represent carbon based char which is highly erosion resistant.

As a conclusion from TGA curves the only stable material above 1000 °C will be the carbon based char remains from decomposition of EPDM and KP. So as Kevlar pulp phr increases in the insulation composition, the insulation efficiency increases with respect to decomposition.

ABLATION TEST

Another important aspect of the characterization of the material compositions includes measurement of ablation rate according to ASTM-E-285-80. [10] The ablation test was done by preparing a sample of the insulation material (30 phr KP) with 3 mm thickness, length 20 cm and width 20 cm and subjected to a high temperature torch. The sample characteristics before and after the test are shown in Table 3.

The resultant ablation rate is outstanding for rocket motor insulation. This is due to the KP content which itself has very high ablation resistance and stability up to 1450 °C. The temperature in the back of the ablation test sample (79 °C) indicates that the insulation which contains Kevlar pulp is outstanding thermal insulation material.

CONCLUSION

Increasing the Kevlar pulp phr (part per hundred ratio) content inside EPDM will improve the performance of EPDM as a solid rocket motor insulation with respect to tensile strength, ablation resistance, thermal diffusivity and thermal conductivity. However this will not give high performance with respect to decomposition resistance.

FUTURE WORK

It is intended to use hybrid reinforcements for elastomers where we complement the insufficient decomposition resistance of KP filled elastomers by the high temperature stable material, chopped carbon fiber (CCF) which is stable up to 3200°C. The characteristics of chopped carbon fiber and Kevlar pulp, using the hybrid (CCF, Kevlar pulp) may have improved performance in ablation resistance and decomposition temperatures due to using CCF and thermal properties due to using Kevlar pulp.

Table (1): Physical and mechanical testing results for different phr contents of KP

Property Composition	Tensile Strength (MPa)	Elongation (%)	Density (g/cm³)	Hardness (shore A)
EPDM + 10 phr KP + 60 phr AP + 5 phr PCA	5.13	31.4	1.156	87.8
EPDM + 15 phr KP + 60 phr AP + 5 phr PCA	6.08	26.9	1.163	89.9
EPDM + 20 phr KP + 60 phr AP + 5 phr PCA	7.19	21.3	1.171	91.1
EPDM + 25 phr KP + 60 phr AP + 5 phr PCA	8.24	15.6	1.179	92.3
EPDM + 30 phr KP + 60 phr AP + 5 phr PCA	9.35	11.7	1.185	93.4

Table (2): Thermal testing results for different phr contents of KP

Property Composition	Thermal diffusivity (mm²/s)	Specific heat capacity (J/kg.C)	Thermal conductivity (W/m.C)
EPDM + 10 phr KP + 60 phr AP + 5 phr PCA	0.111	1834	0.235
EPDM + 15 phr KP + 60 phr AP + 5 phr PCA	0.103	1801	0.216
EPDM + 20 phr KP + 60 phr AP + 5 phr PCA	0.096	1778	0.2
EPDM + 25 phr KP + 60 phr AP + 5 phr PCA	0.088	1756	0.182
EPDM + 30 phr KP + 60 phr AP + 5 phr PCA	0.081	1741	0.167

Table 3: Ablation rate test results

property	before	after
Thickness of the sample (mm)	3	2.62
weight of the sample (gm)	36	34.2
Time (seconds)	60	
Temperature in front of sample (°C)	2010	
Temperature in back of sample (°C)	79	
Resultant ablation rate (mm/sec.)	0.01	
Regular ablation rate (mm/sec.)	0.09-0.2	

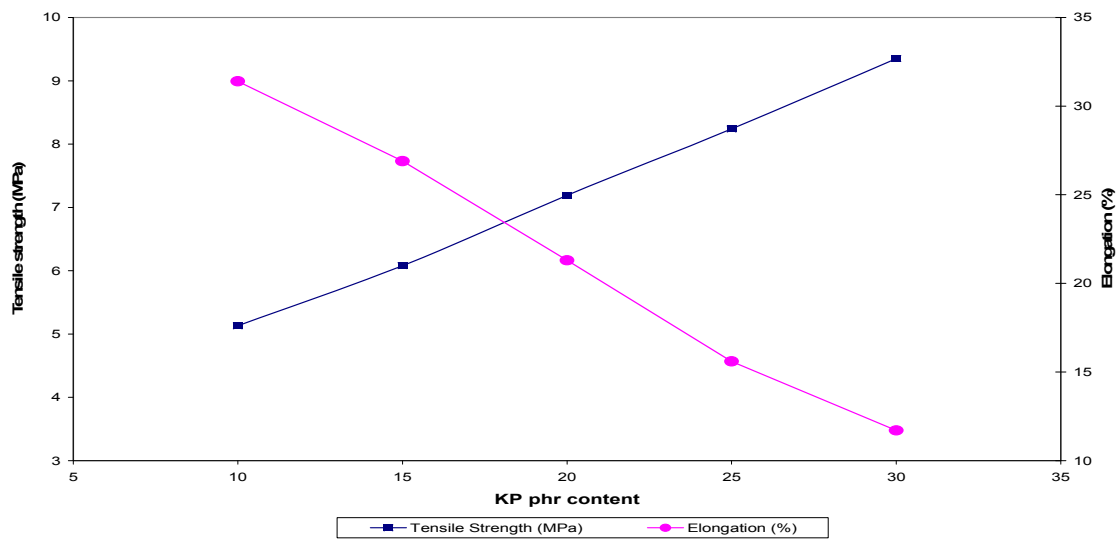


Fig (1): Tensile strength & Elongation of the material as a function of KP phr content

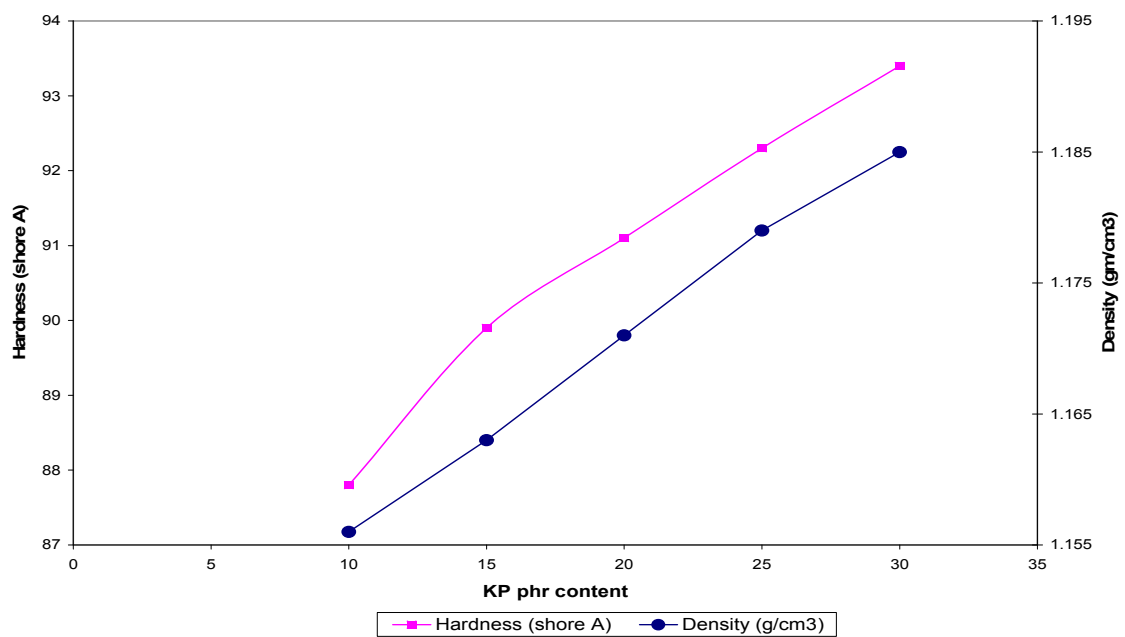


Fig (2): Hardness & Density of the material as a function of KP phr content

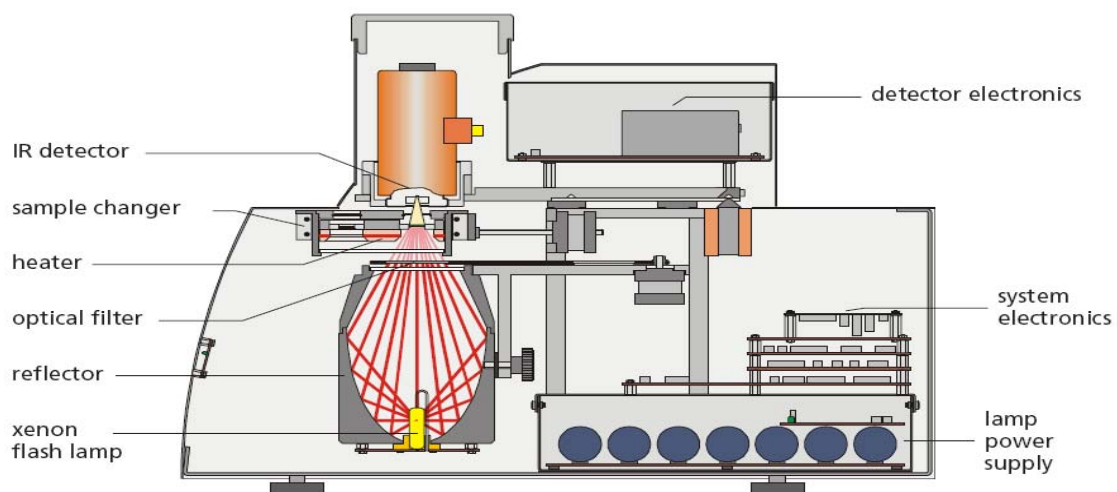


Fig (3): The nanoflash instrument main measurement components

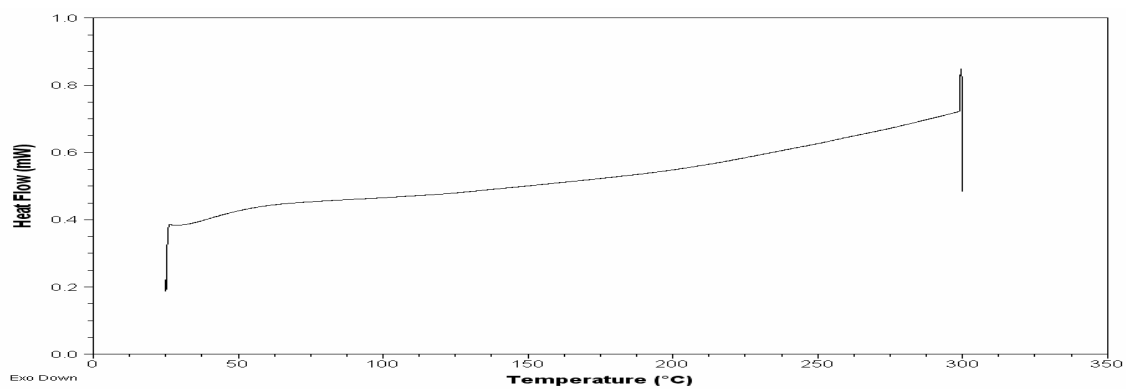


Fig (4): DSC curve for base line (without sample)

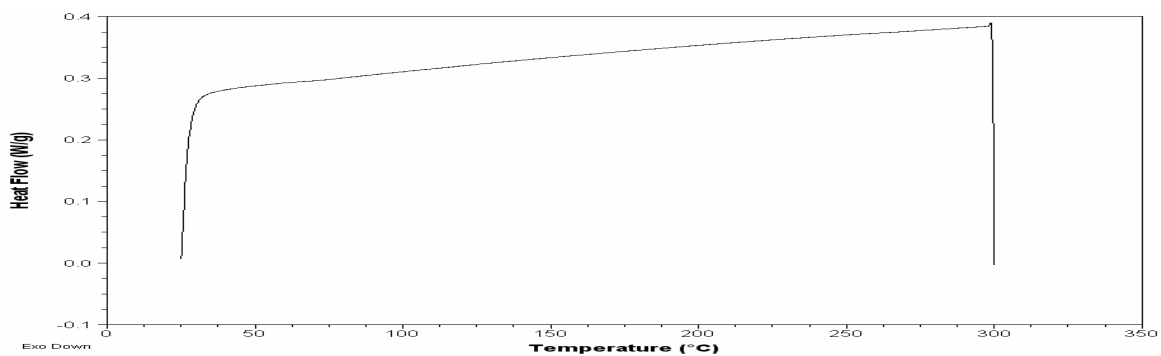


Fig (5): DSC curve for sapphire calibrant

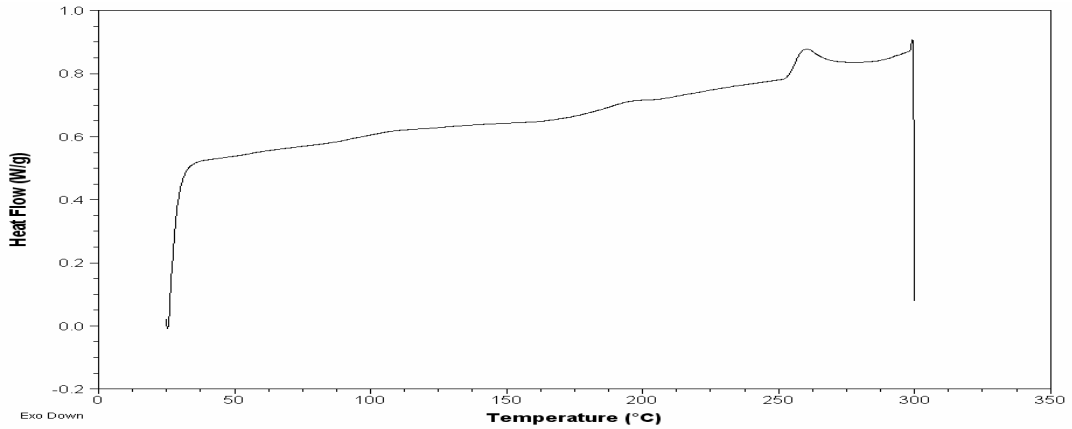


Fig (6): DSC curve for insulation composition having 10 phr KP content

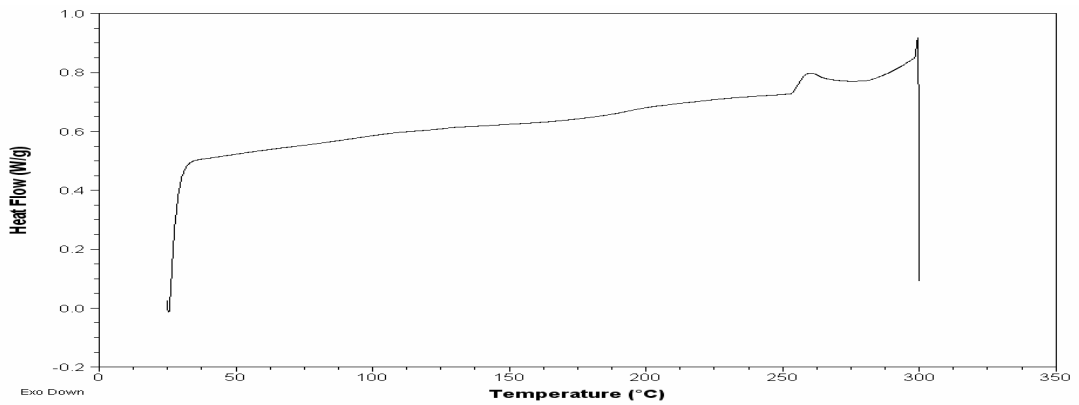


Fig (7): DSC curve for insulation composition having 30 phr KP content

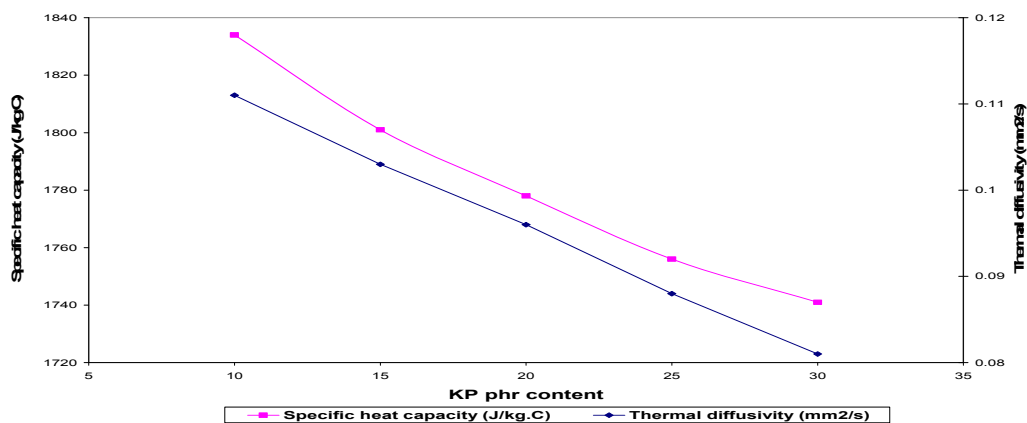


Fig (8): Specific heat capacity & thermal diffusivity of the material as a function of KP phr content

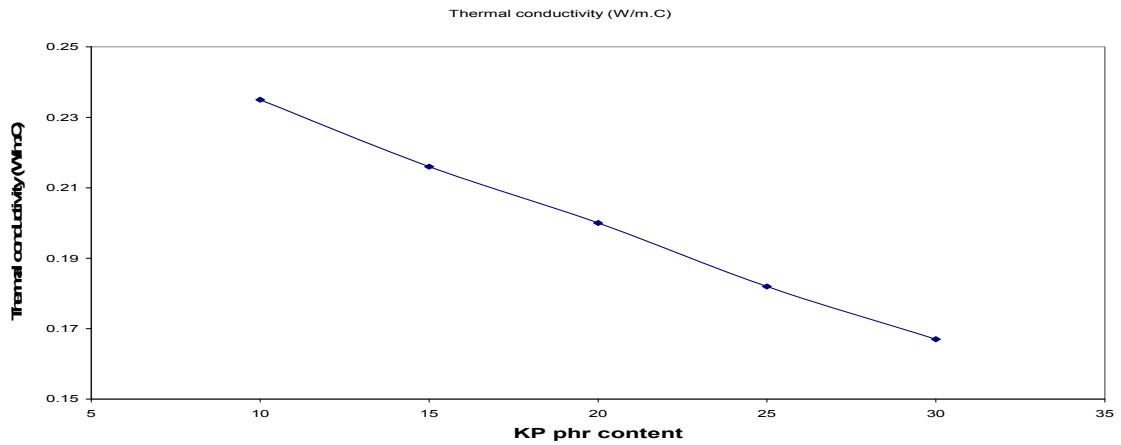


Fig (9): Thermal conductivity of the material as a function of kp phr content

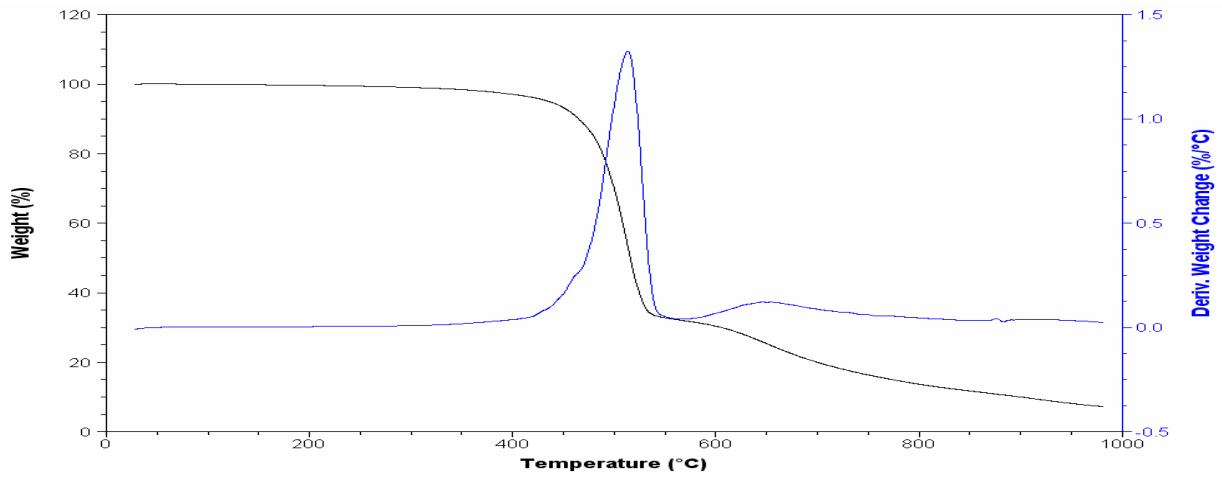


Fig (10): TGA curve for insulation composition having 10 phr KP content

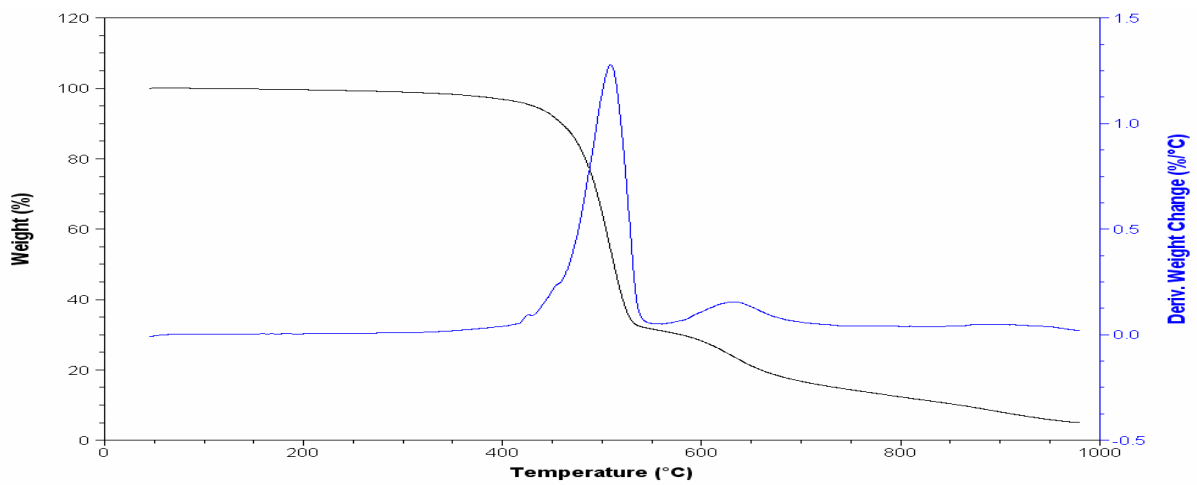


Fig (11): TGA curve for insulation composition having 30 phr KP content

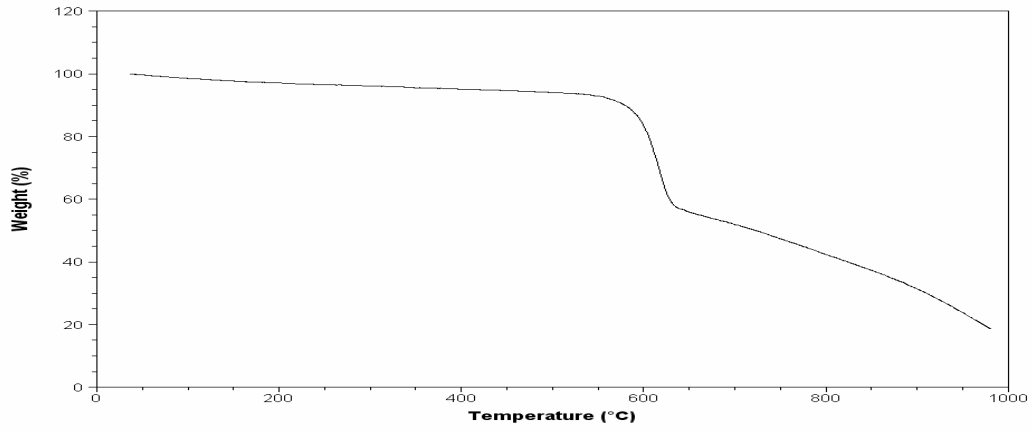


Fig (12): TGA curve for KP alone

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REFERENCES

- [1] C.M. Bhuvanewari, M.S. Sureshkumer, S.D. Kakade, and Monaj Gupta. "Ethylene – propylene rubber as a futuristic elastomer for insulation of solid rocket motor". Defence science journal, Vol 56, No.3 July 2006 pp.309-320.
- [2] Guillot, David. G. Novel, US Patent no 6787586, Sept. 7, 2004 'EPDM rocket motor insulation.
- [3] Roberto Isopi, Eros Pittarelli, Antonio Sebasta. "Development of low density insulators for space motors". SNIA Viscosa, Space and defence Division Via Sicilia 162, rome, Italy, 1979.
- [4] R. E. Morgan, A. S. Prince, and S. A. Selvidge, and J. Phelps, C. L. Martin, T. W. Lawrence. "Non – asbestos insulation testing using a plasma torch". July 2000.
- [5] Wang Zheng, Yongqiang Hu, *Solid Rocket Motor, 1st ed.*, Astronautics industry Press, Beijing, 1993, pp. 229-236.
- [6] Dupont advanced fibers systems, Technical guide for KEVLAR, USA, 2005.
- [7] Guillot, David. G. Novel, US Patent no 6893597, May 17, 2005 'Method of insulating a case of a solid propellant rocket motor'.
- [8] Guillot, David. G. Novel, US Patent no 5498649, March 12, 1996 'Low density thermoplastic elastomeric insulation for rocket motors'.
- [9] Guillot, David. G. Novel, US Patent no 5399599, March 21, 1995 'Thermoplastic elastomeric internal insulation for rocket motors for low temperature applications.
- [10] A. J. Kinloch and R. J. Young, Behavior of polymers, Elsevier Science Publishers, Ltd., London, 1983.