

Effect of Adding Fibers and Nano Particles on the Thermal and Ablative Properties of EPDM Rubber Thermal Insulators for Solid Rocket Motor Application

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Abstract— The rocket motor is one of the main types of jet propulsion engines, and the solid rocket motors is one of the chemical propulsion system where the fuel and oxidizer are both incorporated in a single solid, called the propellant grain. The combustion chamber contains the propellant grain and the thermal insulation. The primary function of the thermal insulation which is defined as a layer of heat barrier placed between the internal surface of the case and the propellant grain is to protect the case from reaching temperatures that may endanger its structure integrity. The present study is to develop and characterize rubber thermal insulators for high temperature application for solid rocket motors and to improve the surface life of the insulants. The insulators are based on aramid fiber in the pulp form (Kevlar), Carbon mono fibers and Nano carbon black as fillers for TRILENE liquid polymers which is a family of viscous, low molecular weight ethylene-propylene diene terpolymers, with ammonium polyphosphate as flame retardant and an organic peroxide as crosslinking agent. Different formulations based on these fillers were prepared. The fillers are dispan in the EPDM polymeric matrix to obtain a homogenous master batch for curing. The thermal and ablative properties of insulations were measured. The effect of changing aramid fiber in the pulp form, carbon mono fiber and/or Nano carbon black volume fractions was studied. Prediction of thermal conduction of multiphase thermal insulation composite materials was carried using parallel, Series, Maxwell Eucken and Effective medium theory models. Comparison with the measured values allows determining the model which estimates best the thermal conductivity of composite insulation material. For application for solid rocket motor insulation, Reinforcement of EPDM with KP improves the performance of the material with respect to thermal properties (thermal conductivity) while not improving well the performance with respect to ablation resistance. Using hybrid reinforcement content (KP + CMF+ NCB) inside EPDM improves the performance of EPDM with respect to thermal properties, ablation resistance and decomposition resistance. The best volume fraction which gives the best performance of the insulation material is 10 Phr KP + 5 Phr CMF + 5 Phr NCB. A new type of insulation material using the hybrid reinforcements was developed for the first time.

Keywords— Thermal insulator, Fibers, Nanoparticles, Fillers, Solid rocket motor

I. INTRODUCTION

The rocket motor is one of the main types of jet propulsion engines, and the solid rocket motors is one of the chemical propulsion system it considered as the simplest form of

chemical propulsion where the fuel and oxidizer are both incorporated in a single solid, called the propellant grain, which located inside the combustion chamber (motor case). Figure (1) shows the main components of the solid rocket motor [1-2].

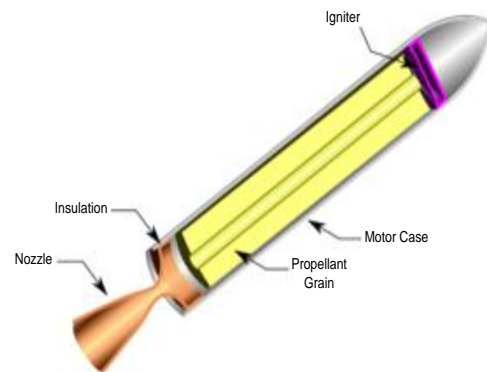
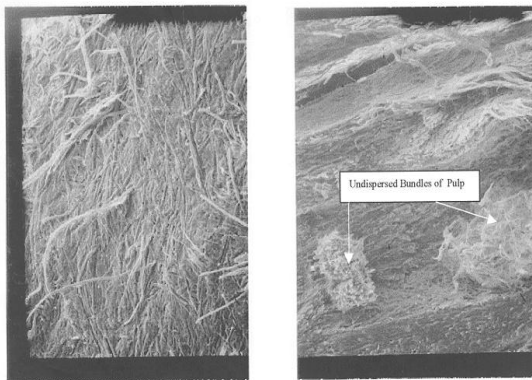


Fig.(1)Longitudinal cross section of solid rocket motor

The combustion chamber contain the propellant grain and the thermal insulation, in general when the propellant ignited it release huge amount of gases and heat energy .the temperature inside the combustion chamber can reaches to 2400-3700 °C in a very short time ,these high temperature may destroy the rocket motor case [3].the primary function of the thermal insulation which is defined as a layer of heat barrier placed between the internal surface of the case and the propellant grain is to protect the case from reaching temperatures that may endanger its structure integrity[4].Insulators usually consist either of thermosetting or elastomers plastics it identified by a hybrid name (fiber/matrix)[5].

The present study is to develop and characterize rubber thermal insulators for high temperature application for solid rocket motors and to improve the surface life of the insulants. The insulators are based on aramid fiber in the pulp form (Kevlar), Carbon mono fibers and Nano carbon black as fillers for TRILENE liquid polymers which is a family of viscous, low molecular weight ethylene-propylene diene terpolymers, with ammonium polyphosphate as flame retardant and an organic peroxide as crosslinking agent.

TRILENE liquid polymers (EPDM) has been established as a standard for solid propellant rocket motors insulation due to its superior ablation, characteristics, excellent physical properties and process ability [6]. TRILENE liquid polymers (EPDM) is good to adjust the elastic properties of the rubbery insulators and to perform a good dispersion of the fillers in the matrix better than the solid EPDM to produce a homogenous insulators [7]. Figure (2) shows us aramid fibers in trilene mixed in bra bender mixer and aramid fibers in solid EPDM (Nordel 4640) mixed using two roll mill.



Aramid Fibers in Trilene Aramid Fibers in Nordel

Fig.(2) Aramid fibers in trilene mixed in bra bender mixer and aramid fibers in solid EPDM (Nordel 4640) mixed using two roll mill.

Polyphosphate flame retardant which is used in this study is coated one with a silicone resin film which does not come to viscous in storage by absorbing moisture and does not coagulate and disperse uniformly in the matrix. Also the resin film does not crack during stirring and its dispersed condition is held stable this coating layer can increase the surface life of the insulants as it will not absorb moisture during storage [8]. Ammonium phosphate flame retardant when starts to decompose it gives polymeric phosphoric acid and ammonia. The polyphosphoric acid reacts with hydroxyl or other groups of a synergist to a nonstable phosphate ester. In the next step the dehydration of the phosphate ester follows. On the surface carbon foam is built up against the heat source (charring). The carbon barrier acts as an insulation layer, preventing further decomposition of the material [9].

Kevlar was found to be the most material having low thermal conductivity and good ablation properties as a result it has been used as reinforcement material for most thermal insulations which are used in the field of the solid rocket motors. Also combining Kevlar with other fibers or powders can enhance the ablation rate of the insulant [10-14]. Using Carbon mono fibers mixed with EPDM is especially useful for placement in nozzle or case between internal surface and the grain propellant [15-18]. By adding Nano carbon black we can decrease the spaces founded between the filler and the polymer resulting in decreasing the decomposition temperature and the ablation rate which fulfill the requirement of the solid rocket motors [19-21].

II. MATERIALS AND METHODS

- A. Ethylene propylene diene monomer (EPDM) as Trilene source...Lion copolymer geismar,Llc
- B. Kevlar, poly para phenylene terephthalamide, source...Dupont company.
- C. Carbon mono fibers, , source...Dupont company.
- D. Nano carbon black (ISAF N220) ,source... Beilum carbon chemical limited.
- E. Organic Peroxide Crosslinking Agent, Varox, [2,5 dimethyl 2,5 di(T-butyl peroxy)hexane],source...Dupont company.
- F. Ammonium polyphosphate flame retardant agent (Phos-Chek P/30), source...BK Giulini GmbH.

The formulations were composed of 100 Phr EPDM with 60Phr Ammonium polyphosphate flame retardant and 5 Phr peroxide crosslinking agent with different amounts of reinforcement's fillers.

The insulators were mixed in a C. W. Bra bender mixer to attain uniform dispersion of the reinforcement within EPDM. and cured under a press ($T = 175\text{ }^{\circ}\text{C}$, $P = 10\text{ bar}$). Curing molds were cleaned after each sample and Teflon based release agent was applied between the mold parts to produce samples of identical dimensions (250 x 150 mm).

III. EXPERIMENTAL WORK

The thermal conductivity K of the cured thermal insulation compositions were measured using the comparative thermal conductivity instrument where the thermal conductivity of the unknown specimen is determined by comparing this property to the known thermal conductivity of a reference material. The reference material is chosen to match, as closely as possible, the expected thermal conductance of the unknown sample. Another important aspect of the characterization of the material compositions includes defining the primary reactions in the decomposition of the material. DSC instrument was used for measuring the specific heat capacity while the TGA instrument was used to detect the initial and final temperatures of every insulant. TMA measures material deformation changes under controlled conditions of force, atmosphere, time and temperature it was used for determining the thermal expansion coefficient. Ablation rate was measured according to ASTM-E-285-80. The ablation test was done by preparing a sample of the insulation material with 3 mm thickness, length 20 cm and width 20 cm. Then it bonded to a steel piece with the same dimensions with the special adhesive Epon 828 and Epicure. A thermocouple is then fixed in the back of steel sheet and the insulation material is exposed to a high temperature torch ($2100\text{ }^{\circ}\text{C}$). The sample characteristics before and after the test are recorded.

Table (1) prepared samples formulations (PHR)

Comp. Samples	TRILENE 67	AP PHOSCHC K P30	PCA	KP	CM F	NC B
S2	100	60	5	20	-	-
S6	100	60	5	-	20	-
S10	100	60	5	-	-	20
S11	100	60	5	-	-	10
S12	100	60	5	-	-	5
S13	100	60	5	15	5	-
S14	100	60	5	10	10	-
S15	100	60	5	5	15	-
S16	100	60	5	15	-	5
S17	100	60	5	10	-	10
S18	100	60	5	5	-	15
S19	100	60	5	-	15	5
S20	100	60	5	-	10	10
S21	100	60	5	-	5	15
S22	100	60	5	10	5	5
S23	100	60	5	5	10	5
S24	100	60	5	5	5	10

IV. RESULTS AND DISCUSSIONS

It is obvious that increasing KP Phr content in the material will result in decreasing the thermal conductivity of the material, This is due to the lower thermal conductivity of KP (0.04 W/m.k) compared to EPDM (0.36 W/m°C). This goes with the requirement of the insulation material to have a very low thermal conductivity. But increasing Phr content of CMF or NCB in the material will result in increasing the thermal conductivity of the material due to the high thermal conductivity of CMF(6.4 W/m°C) and NCB(10 W/m°C). for the KP with CMF and KP with NCB hybrid samples and it appears that decreasing KP Phr content in the material and increasing the CMF Phr content or the NCB Phr content in the material will result in increasing its thermal conductivity.

This is due to the lower thermal conductivity of KP (0.04 W/m.K) as compared to CMF and NCB which have high thermal conductivity (6.4 W/m.K) and (10 W/m.K).for the CMF and NCB hybrid samples it appears that decreasing CMF Phr content in the material and increasing the NCB Phr content in the material will result in increasing its thermal conductivity but by a small amount This is due to the that the thermal conductivity of NCB (10 W/m.K) is higher than that of CMF(6.4 W/m.K). The KP and CMF and NCB mixture samples appears that decreasing KP Phr content and increasing CMF and NCB Phr content in the material will result in increasing its thermal conductivity. Figure (3) show us the samples of thermal conductivity test and figure (4) show the result of thermal conductivity test.



Fig.(3) Samples of Thermal Conductivity Test

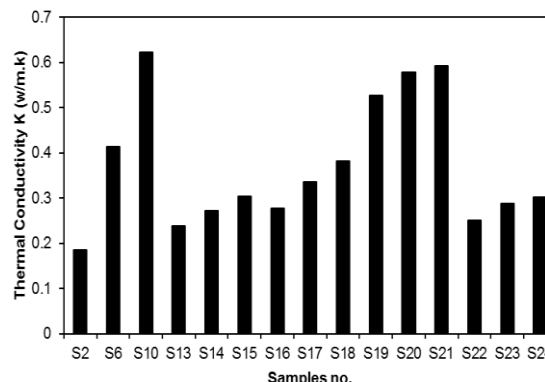


Fig.(4) Result of Thermal Conductivity Test

For TGA analysis for the matrix (EPDM) alone was done. It appeared that the initial decomposition temperature for EPDM is 401 °C and the final decomposition temperature for EPDM is 496 °C. Also For TGA analysis of the flame retardant alone the tests indicate that an initial decomposition temperature for ammonium polyphosphate (flame retardant agent) occurs around 545 °C and the final decomposition is at 670 °C. These temperatures are lower than those obtained from TGA of the whole insulation because fillers work as active shield for EPDM and AP against decomposition. Figure (5) show us the resultant TGA curves of EPDM and AP alone. As For the 20 phr KP, CMF and NCB samples The TGA tests indicate for all compositions that an initial decomposition temperature for EPDM (matrix) occurs around 471 °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 576 °C and the final decomposition is at 763 °C. The only stable ingredient above 1000 °C is KP, CMF and NCB which is stable up to 1450, 3000 and 3500 °C, in addition to the carbon based char remains from decomposition of EPDM. So the remaining weight for the 20 phr KP, CMF and NCB sample just before 1000 °C is 7.028 %, 13.075 % and 15.65 % of the total insulation weight. Figure (6) show the resultant TGA curves as a function of 20 phr KP, CMF and NCB. for the KP with CMF and KP with NCB hybrid samples it appears that as the phr of CMF or NCB increases and the phr of KP decreases inside EPDM the insulation efficiency increases with respect to decomposition. This is clear where the remaining weight for sample (13) insulation composition

just before 1000 °C is 9.624 % of the total insulation weight while for sample (15) insulation composition is 12 % of the total insulation weight as shown in figure (7). This is due to the CMF which is very stable up to 3000°. Also for the remaining weight for sample (16) insulation composition just before 1000 °C is 11.08 % of the total insulation weight while for sample (18) insulation composition is 13.16 % of the total insulation weight as shown in figure (7). This is due to NCB which is highly stable up to 3500°. for the CMF and NCB hybrid samples it appears that as the phr of NCB increases and the phr of CMF decrease inside EPDM the insulation efficiency increases with respect to decomposition. This is clear where the remaining weight for sample (19) insulation composition just before 1000 °C is 13.8 % of the total insulation weight while for sample (15) insulation composition is 16.35 % of the total insulation weight as shown in figure (7). This is due to the CMF + NCB which are highly stable up to 3500°. the KP and CMF and NCB mixture samples it appear that as the phr of CMF and NCB increases and the phr of KP decreases inside EPDM the insulation efficiency increases with respect to decomposition. This is clear where the remaining weight for sample (22) insulation composition just before 1000 °C is 11 % of the total insulation weight while for sample (24) insulation composition is 16 % of the total insulation weight as shown in figure (7). This is due to the CMF and the NCB which are highly stable up to 3000°C and 3500°C.

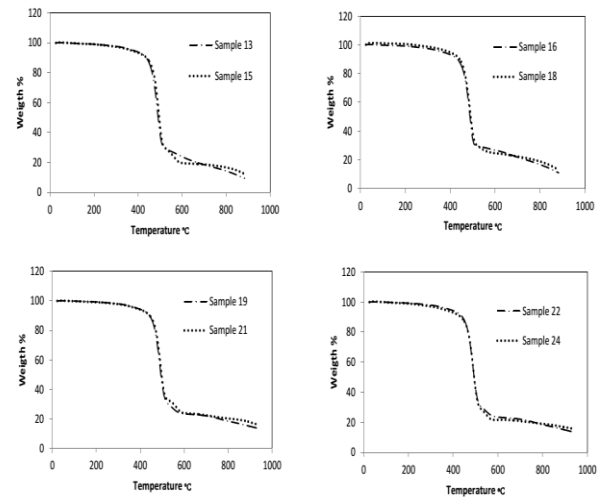


Fig.(7) Resultant TGA curves of different EPDM composites samples

For the KP sample the heat capacity is 2827 J/Kg. °C and for the CMF sample the heat capacity is 2482 J/Kg. °C and for the NCB sample the heat capacity is 2342 J/Kg. °C. So for the KP and CMF hybrid samples and the KP and NCB hybrid samples it appears that decreasing KP Phr content in the material and increasing the CMF or the NCB Phr content in the material will result in decreasing the heat capacity. This is due to the lower specific heat capacity of CMF (660 J/kg. °C) and NCB (600 J/kg. °C) compared to KP (1450 J/kg. °C) and EPDM (2177 J/kg. °C). For the CMF and NCB sample the heat capacity is 2342 J/Kg. °C So for the CMF and NCB hybrid samples it appears that decreasing CMF Phr content in the material and increasing the NCB Phr content in the material will result in decreasing its specific heat capacity. This is due to that the specific heat capacity of CMF is (660 J/kg. °C) compared to NCB (600 J/kg. °C). for the KP and CMF and NCB mixture samples it appears that decreasing KP Phr content and increasing CMF and NCB Phr content in the material will result in decreasing its heat capacity. This is due to the high specific heat capacity of KP (1450 J/kg. °C) and EPDM (2177 J/kg. °C) compared to the lower specific heat capacity of CMF (660 J/kg. °C) and NCB(600 J/kg. °C). Figure (8) show us the samples of specific heat capacity test.

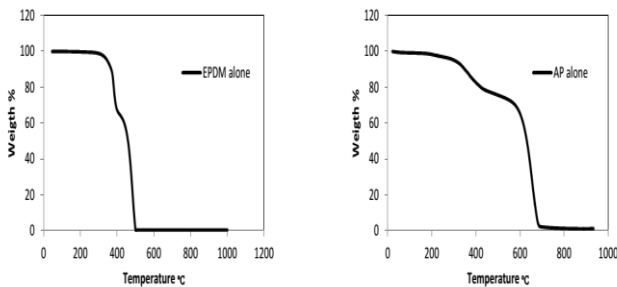


Fig.(5) Resultant TGA curves of EPDM and AP alone

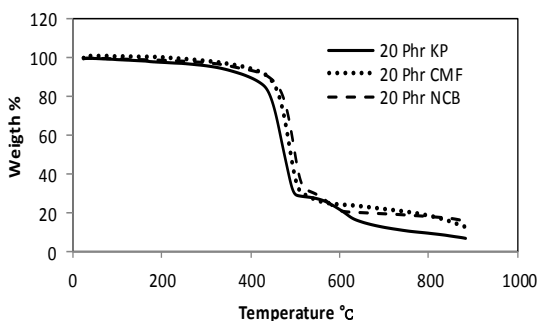


Fig.(6) Resultant TGA curves as a function of 20 phr KP, CMF and NCB

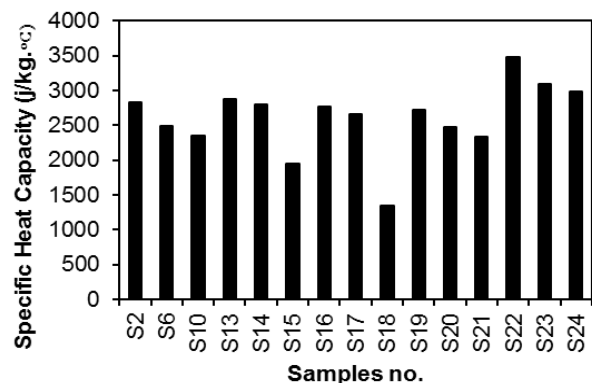


Fig.(8) Samples of Specific Heat Capacity test

For TMA analysis the KP sample show that the coefficient of thermal expansion is $-824.5 \mu\text{m}/\text{m}.\text{°C}$ and for the CMF sample the coefficient of thermal expansion is $-1207 \mu\text{m}/\text{m}.\text{°C}$ and for the NCB sample the coefficient of thermal expansion is $-909.8 \mu\text{m}/\text{m}.\text{°C}$. So for the KP and CMF hybrid samples it appears that decreasing KP Phr content in the material and increasing the CMF or NCB Phr content in the material will result in increasing the coefficient of thermal expansion. For the CMF and NCB hybrid samples it appears that decreasing CMF Phr content in the material and increasing the NCB Phr content in the material will result in decreasing the coefficient of thermal expansion. for the KP and CMF and NCB hybrid samples it appears that decreasing KP Phr content in the material and increasing the CMF and NCB Phr content in the material will result in increasing the coefficient of thermal expansion. Figure (9) show us the samples of TMA test.

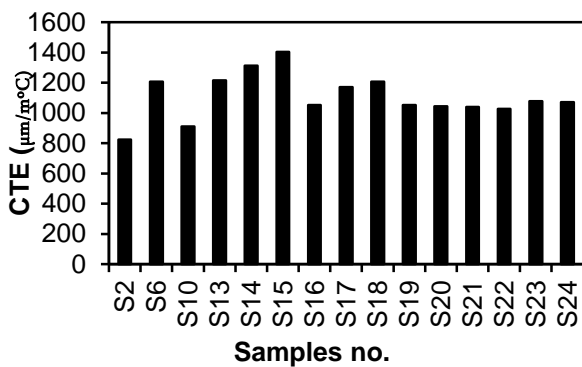


Fig.(9) Samples of TMA test

Ablation resistance for sample 2 (20phr KP content) appeared that the mixing using C. W. Bra bender permits homogeneous dispersion of Kevlar pulp in the EPDM polymeric matrix. This homogenous distribution of KP inside EPDM improves the performance of EPDM with respect to the thermal resistance. For ablation resistance of sample (2) the resultant ablation rate (0.016 mm/sec) this is due to the KP content which itself has high ablation resistance and stability up to 1450°C . The temperature at the back of the ablation test sample after 60 seconds (60°C) indicates that the insulation which contains KP is outstanding thermal insulation material. This satisfies the requirement of the insulation material to have very low heat conduction. For ablation resistance of samples (6) and (10), the resultant ablation rate (0.012 mm/sec) which corresponds to CMF with 20 phr content and (0.011 mm/sec) which corresponds to NCB with 20 phr content. This is due to the CMF and NCB content which itself have very high ablation resistance and stability up to 3000°C and 3500°C . The temperature at the back of the ablation test sample after 60 seconds (120°C) indicates that the insulation containing CMF is a conductive material. also for NCB The temperature at the back of the ablation test sample after 60 seconds (160°C). This goes against the requirement of the insulation material to have very low heat conduction. For ablation resistance of samples (14) and (17), the resultant ablation rate (0.014 mm/sec) and (0.013 mm/sec) which corresponds to 10 phr KP and 10 phr CMF (sample 14) and 10 phr KP and 10 phr NCB (sample 17) is outstanding for rocket

motor insulation. The temperature at the back of the ablation test sample after 60 seconds (80°C) and (87°C) indicates that the insulation which contains (KP + CMF) or (KP + NCB) is outstanding thermal insulation material. This satisfies the requirement of the insulation material to have very low heat conduction. For ablation resistance of sample (20), The resultant ablation rate (0.01 mm/sec) which corresponds to CMF with 10 phr and NCB with 10 phr content. This is due to the CMF and NCB content which have high ablation resistance and stability up to 3000°C and 3500°C . The temperature at the back of the ablation test sample after 60 seconds (140°C) indicates that the insulation which contains (CMF + NCB) is a conductive material. This goes against the requirement of the insulation material to have very low heat conduction. For ablation resistance of sample (22), The resultant ablation rate (0.012 mm/sec) which corresponds to KP with 10 phr and CMF with 5 phr and NCB with 5 phr content are outstanding for rocket motor insulation. This is due to the KP content which itself has high ablation resistance and stability up to 1450°C and the CMF and NCB which have a very high ablation resistance and stability up to 3000 and 3500°C . The temperature at the back of the ablation test sample after 60 seconds (70°C) indicates that the insulation which contains (KP + CMF + NCB) is outstanding thermal insulation material. This satisfies the requirement of the insulation material to have very low heat conduction. Figure(10) show us the samples of the ablation test before and after test.

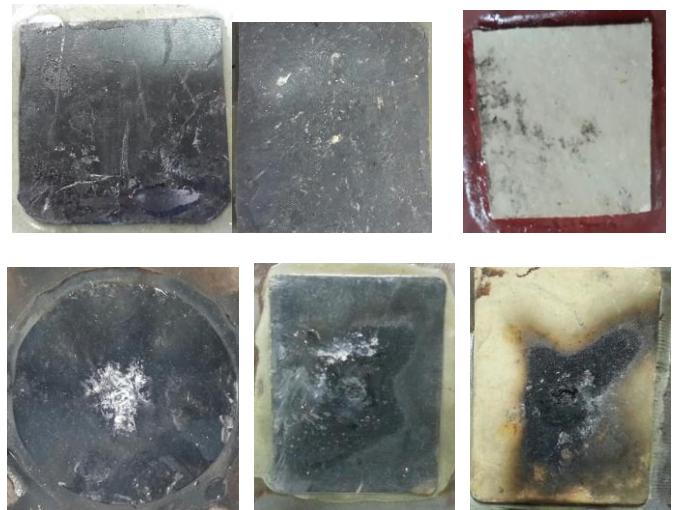


Fig.(10) Samples of the Ablation test before and after test

V. THERMAL CONDUCTION MODELING

The thermal conductivity of any material can be calculated using the relation in formula (1)

$K = \rho \cdot A \cdot C_p$ (1) Where K, ρ , A and C_p are the thermal conductivity, density, thermal diffusivity and heat capacity of the material respectively.

Modeling the effective thermal conductivity of heterogeneous or composite materials is of interest in many heat transfer applications. Many researchers provide reviews of relevant modelling approaches. A substantial number of effective thermal conductivity models has been proposed, some of which have been intended for highly specific applications.

effective thermal conductivity models found in the literature are based on one or more of four basic structural models; specifically, the Series (2), Parallel (3), Maxwell–Eucken (4) and Effective Medium Theory (EMT) (5) models[22-25].

$$K = \frac{1}{(v_f/k_f) + (v_m/k_m)} \quad (2)$$

$$K = k_f v_f + k_m v_m \quad (3)$$

$$K = k_m \frac{k_f + 2k_m + 2v_f(k_f - k_m)}{k_f + 2k_m - v_f(k_f - k_m)} \quad (4)$$

$$v_f \frac{k_f - K}{k_f + 2K} + v_m \frac{k_m - K}{k_m + 2K} = 0 \quad (5)$$

Where K is the effective thermal conductivity of the whole composite material, km and kf are the thermal conductivities of the matrix (EPDM) and fiber (KP, CMF and/or NCB) respectively, vf and vm are the volume fractions of the fiber (KP, Al and/or Si) and matrix (EPDM) respectively. Figure (11) shows the Models based on the structure of the homogenous phase and dispersed phase

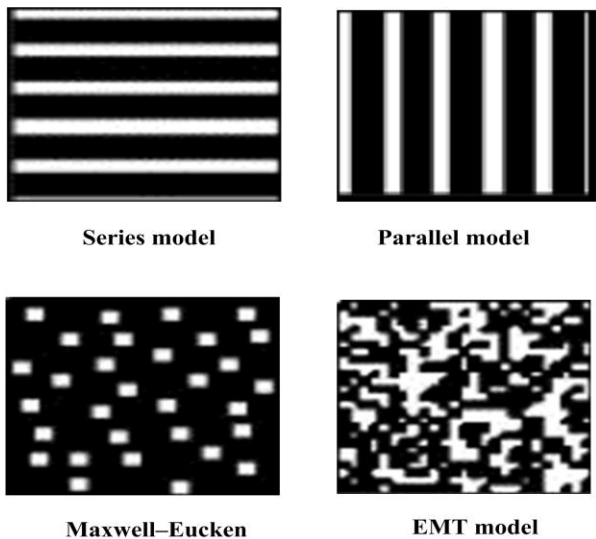


Fig.(11) Models based on the structure of the homogenous phase and dispersed phase

For using KP or CMF or NCB as a filler or using hybrid of them we have the thermal conductivity of EPDM which is 0.36 w/m.k. and for KP, CMF and NCB is 0.04, 6.4 and 10 w/m.k. So by substitution into the models equations we can get the effective thermal conductivity calculated values of all models. The results compared to the experimental values. Table (2) and Figure (12) show the calculated and measured thermal conductivities of the samples.

Table (2) Calculated and Measured Thermal Conductivities

Models \ Samples	Series	Parallel	Maxwell Eucken	Effective Medium Theory	Exp. Results
S2	0.154	0.306	0.289	0.285	0.184
S6	0.427	1.366	0.537	0.597	0.414
S10	0.429	1.965	0.550	0.629	0.623
S13	0.414	0.572	0.466	0.475	0.238
S14	0.423	0.836	0.509	0.539	0.279
S15	0.426	1.101	0.527	0.574	0.303
S16	0.420	0.721	0.495	0.517	0.277
S17	0.426	1.136	0.528	0.577	0.335
S18	0.428	1.551	0.543	0.609	0.382
S19	0.427	1.516	0.542	0.607	0.526
S20	0.428	1.666	0.545	0.615	0.577
S21	0.433	1.816	0.548	0.623	0.591
S22	0.425	0.986	0.520	0.561	0.250
S23	0.426	1.251	0.534	0.588	0.288
S24	0.427	1.401	0.539	0.600	0.302

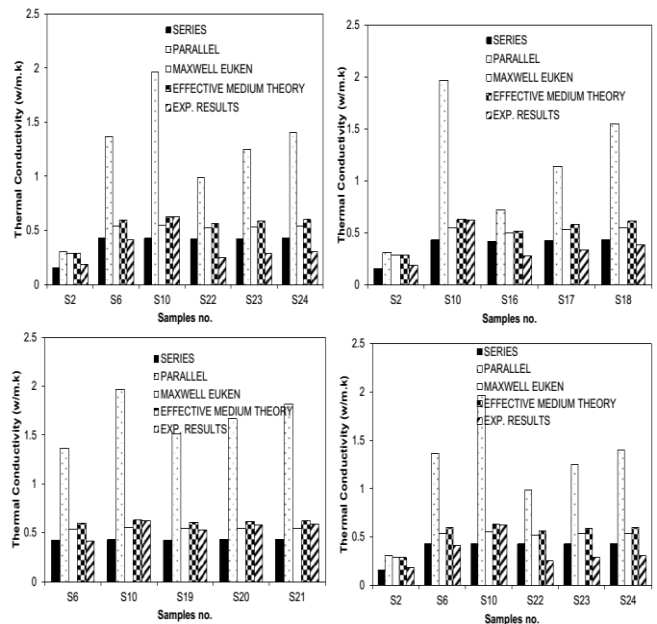


Fig.(12) Calculated and Measured Thermal Conductivities of the Samples.

From figure (12) the model that gives result closet to the experimental measurement for using KP with CMF or NCB or using a mixture of them as a filler is the Series model which assumes layers of the components aligned perpendicular to the heat flow. For KP and CMF the difference is within 28.8 % of the measured value. Also for KP and NCB the difference is within 10.7 % of the measured value. For the mixture of the KP, CMF and NCB the difference is within 29.2 % of the measured value. But for the samples containing hybrid of CMF and NCB the model that gives result closet to the experimental measurement is the Maxwell Eucken the difference is within 7.2 % of the measured value.

As a result, the thermal conductivity of KP and CMF, KP and NCB, and KP with CMF and NCB reinforced EPDM can be estimated by the following formulas. Figure (13) shows the estimated and the experimental results.

$$K = 1.282 \frac{1}{(v_f/k_f) + (v_m/k_m)} \quad (6)$$

$$K = 1.107 \frac{1}{(v_f/k_f) + (v_m/k_m)} \quad (7)$$

$$K = 1.292 \frac{1}{(v_f/k_f) + (v_m/k_m)} \quad (8)$$

For CMF and NCB reinforced EPDM the thermal conductivity can be estimated by the following formula

$$K = 1.072k_m \frac{k_f + 2k_m + 2v_f(k_f - k_m)}{k_f + 2k_m - v_f(k_f - k_m)} \quad (9)$$

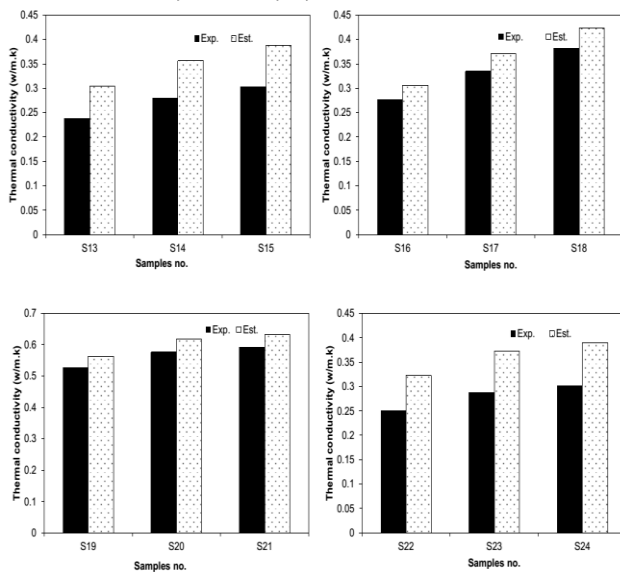


Fig.(13) Estimated and Experimental Thermal Conductivities results

VI. CONCLUSION

Best formulation which gives the least thermal conductivity was achieved by the sample containing 20 Phr KP (sample S2), while the highest specific heat capacity was achieved by the sample containing 10 phr KP + 5 phr CMF + 5 phr NCB (sample S22), the least ablation rate was achieved by the sample containing 10 Phr CMF + 10 Phr NCB (sample S20), also the least outer case temperature was achieved by the sample containing 20 phr KP (sample S2).

Reinforcement of EPDM with KP improved the performance of the material with respect to thermal properties (thermal conductivity) while not improving well the performance with respect to ablation resistance. Reinforcement of EPDM with CMF or NCB improved the performance of the material with decomposition properties and ablative properties while not improving the performance with respect to thermal conductivity.

Using hybrid reinforcement content (KP + CMF + NCB) inside EPDM improves the performance of EPDM with respect to thermal properties, ablation resistance and decomposition resistance. The best volume fraction which gives the best performance of the insulation material is 10 Phr KP + 5 Phr CMF + 5 Phr NCB which fulfil all the requirement of thermal insulation for solid rocket motors. The best model to estimate the thermal conductivity of KP with CMF or NCB formulations material is the series model which assumes layers of the components aligned perpendicular to the heat flow.

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