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Study on Magnesium-based Pyrotechnic Composition as a Priming Charge

A.S. Redkar, V.A. Mujumdar and S.N. Singh
High Energy Materials Research Laboratory, Pune-411 021

ABSTRACT

A new pyrotechnic composition containing Mg/KNO_3/phenolic resin has been formulated and studied in detail for its sensitivity, mechanical and thermal properties, moisture and environmental effects and performance in a closed vessel. The data generated reveal that this composition shows superior performance, better mechanical properties and less susceptibility to moisture as compared to gunpowder. In addition, performance of the composition under extreme hot (45 °C) and cold (–26 °C) environmental conditions is not affected at all. Differential thermal analyser results indicate that phenolic resin plays a vital role in reducing the ignition temperature of Mg/KNO_3 system.

1. INTRODUCTION

Gunpowder is commonly used as a priming composition and igniter in pyrotechnics and double base propellants, respectively. However, extensive work carried out by several scientists on gunpowder primarily containing KNO_3, charcoal and sulphur indicates that the combustion phenomenon is greatly influenced by the type and origin of charcoal, the particle size, the moisture content and the temperature[1-3]. Besides, sulphur present in the gunpowder accelerates the corrosion of *Mg* powder in metal-based pyrotechnic compositions. As a result, the composition reliability[4] is adversely affected. In view of these drawbacks and also the inferior energy output of the gunpowder, metal-based pyrotechnic compositions generally containing Mg/KNO_3 and binder system, have been reportedly used as priming and igniter compositions[5]. These compositions show better performance as compared to gunpowder due to high exothermic reaction. Literature survey on metal-based priming and igniter compositions has revealed that a few binders such as plasticised ethyl cellulose[6] (PEC), laminac[7], polyvinyl alcohol and polyvinyl acetate[8], etc. of varying percentage (1-10 per cent), have been incorporated in *Mg*, B/KNO_3 systems to achieve better homogeneity and improved mechanical and thermal properties. Further, most of the igniter compositions based on this system are nearer to the stoichiometric ratio. Keeping these facts in view, it was considered of immense interest to formulate a new pyrotechnic composition based on Mg/KNO_3 system with phenolic resin having ingredient percentage of 38/51/11, which is a stoichiometrically balanced composition, and evaluate in detail its physicochemical properties in addition to performance. This paper reports the results of various experiments conducted on this composition. Further, an attempt has also been made to understand the role of phenolic resin binder in combustion phenomenon by means of differential thermal analyser (DTA).

2. EXPERIMENTAL METHODS

The composition Mg/KNO_3/phenolic resin (38/51/11) was prepared by taking *Mg* powder of high purity (> 98 per cent) and particle size + 63 μm/ –150 μm and dried KNO_3, particle size –125 μm. The phenolic resin having softening point 68 - 74 °C was procured from the trade. Magnesium powder (38 g) was first coated with resin lacquer which was obtained by dissolving the phenolic resin powder (11 g) in methyl ethyl ketone (20 ml). Subsequently, the coated *Mg*

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powder after removal of solvent was mixed with dried KNO_3 (51 g) and then sieved through 600 μm sieve to get a homogeneous mixture. The composition was subjected to the evaluation tests.

3. EVALUATION

3.1 Measurement of Sensitivity

3.1.1 Impact Sensitivity

Impact sensitivity of the composition was measured by fall hammer method using 2 kg drop weight and 20 mg of sample. The height given in Table 1 refers to 50 per cent probability of explosion of the compositions.

3.1.2 Friction Sensitivity

Friction sensitivity of the composition was measured by Julius Peter apparatus using 10 mg of the sample. The values given in Table 1 refer to the minimum weight under which three samples did not ignite.

Table 1. Data on sensitivity of *Mg*-based composition and GP-40

Composition	Impact 50% exptl height (cm)	Friction non-ignition (kg)	Spark non-ignition (J)
Mg/KNO₃/phenolic resin	97.5	36*	3.5*
Gunpowder (*KNO₃/C/S*)	54.0	36*	3.5*

* Tested up to the maximum limit of the apparatus.

3.1.3 Spark Sensitivity

Sensitivity to spark was measured[9] by taking 10 mg of sample between two electrodes placed at a distance of 2 - 2.5 mm. The energy of spark was varied from 15 mJ to 5 J. The values given in Table 1 refer to the optimum energy at which two samples did not ignite.

3.2 Thermal Characteristics

3.2.1 Deflagration Temperature

Deflagration temperature was obtained on 5 mg sample by gradually raising the temperature at the rate of 5 °C/min. The temperature at which the sample ignited was recorded (Table 2).

3.2.2 Ignition Temperature

Ignition temperature was recorded by using DTA (laboratory made). DTA curves were recorded in the presence of static air with 5 mg sample in alumina crucibles at heating rate of 10 °C/min (Table 2).

3.2.3 Calorimetric Value Measurement

Calorimetric value of the composition was determined by Parr Bomb calorimeter of 300 cc volume at 1 atm pressure. A quantity of 1 g of the sample was ignited and total heat output was measured (Table 2).

3.3 *P*-max Measurement

Pressure-time output of *Mg*-based composition as well as gunpowder (GP-40) was determined in a closed vessel of 100 cc volume at loading densities 0.02, 0.04, 0.06, 0.08 and 0.10 g/cc. Compositions were placed in a cloth bag and ignited with an electric squib. The results of *P*-max at ambient temperature are shown in Fig. 1.

3.4 Crushing Strength

Crushing strength of the composition was measured by Instron machine using pressed pellets of dimension 20 mm × 20 mm. The values reported in Table 3 are the average of five pellets.

3.5 Moisture Absorption

Moisture absorption was determined by exposing the composition (2 g each) at 64, 75, 84 and 96 per cent

Table 2. Data on thermal characteristics of *Mg*-based composition and GP-40

Composition	Deflagration temperature (°C)	Cal-val (cal/g)	Gas evolved (ml/g)	Exotherm onset temp (°C)
Mg/KNO₃/phenolic resin	> 360	1784	138	452
Gunpowder (*KNO₃/C/S*)	> 360	889	252	424

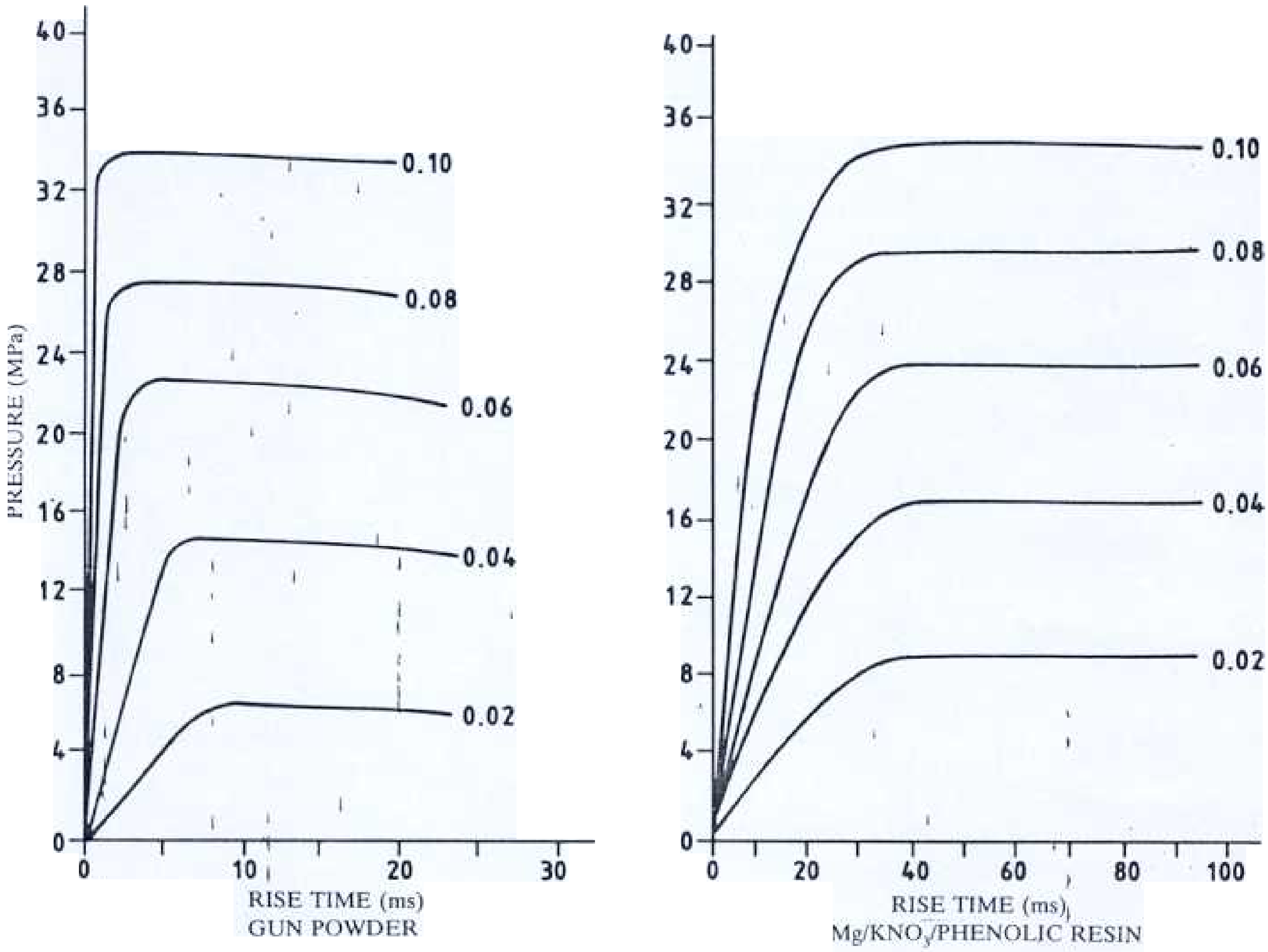

Figure 1. CV firing at different loading densities.

Table 3. Data on crushing strength after exposure to different relative humidities

Composition	Time (hr)	Crushing strength (kg/cm^2) Relative humidity (%) 52	64	75	84	96
Mg/KNO_3/Phenolic resin	0	564	–	–	–	–
	24	552	472	401	387	361
	48	560	455	377	386	350
	72	550	441	369	376	325
	96	540	379	367	370	320
Gun powder ($KNO_3/C/S$)	0	532				
	24	520	364	348	284	229
	48	525	356	337	215	168
	72	525	345	302	112	136
	96	515	340	300	55	45

relative humidity, for a period of 24, 48, 72 and 96 hr, respectively. The increase in weight due to moisture absorption was recorded (Fig. 2).

3.6 Environmental Tests

ISAT(B) tests for the composition were conducted for 4, 13, 36 and 52 weeks as per standard procedure[10] to assess the functioning as well as shelf-life. The loose composition (1 g) placed in monotubular propellant (3.5 g) and properly sealed was taken for conducting the tests. The *P*-max was recorded by CV firing after completing the test in a stipulated period.

3.7 Ignitability

Pyrotechnic compositions were first pressed in steel tubes having 30 mm length and 20 mm diameter in two increments. Magnesium-based composition as a priming having quantities 0.3 g and 0.5 g, respectively were pressed on top of the main composition under 7 ton dead load. The ignitability was tested with an electric squib.

4. RESULTS AND DISCUSSION

Results of sensitivity to impact, friction and spark are given in Table 1. It is seen that Mg/KNO_3/phenolic resin-based composition is less sensitive to impact than gunpowder but values for friction and spark sensitivities are comparable. This may be attributed to having more or less similar order of oxygen balance as obtained in gunpowder and also the elastic nature of the phenolic resin.

Data on thermal characteristics (Table 2) indicate that Mg/KNO_3/phenolic resin-based composition possesses twice cal-val but less gas volume as compared to gunpowder; the composition is relatively more energetic due to high exothermic reaction between *Mg* and oxidiser. Further, it is also seen that the ignition temperature of the composition is slightly higher than that of gunpowder.

Data on closed vessel at ambient temperature as shown in Fig. 1 indicate that *P*-max increases in *Mg*-based composition as well as gunpowder with

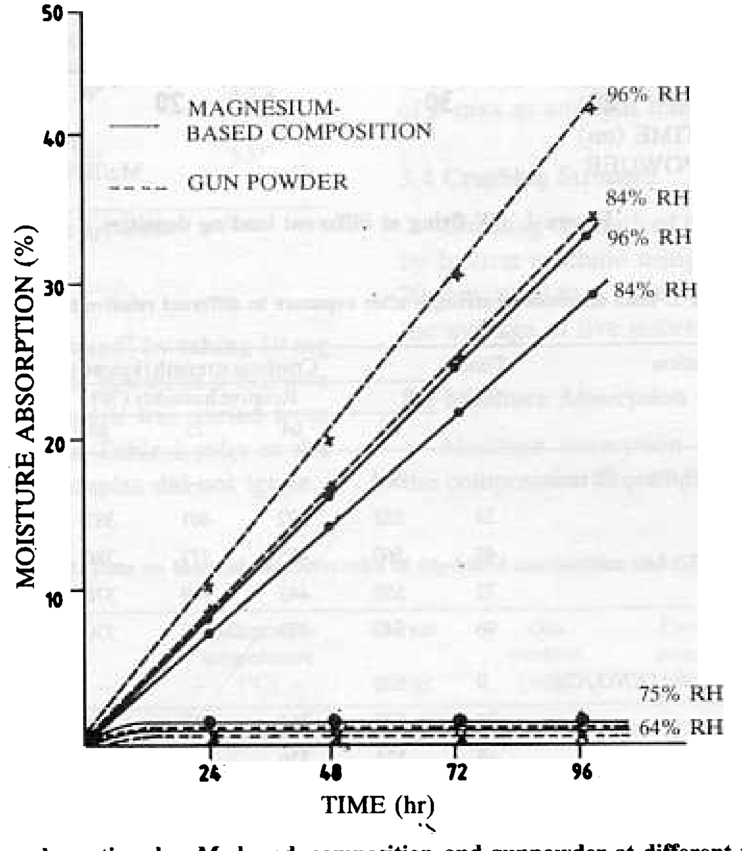

Figure 2. Moisture absorption by *Mg*-based composition and gunpowder at different relative humidities.

increase in loading density. Increase in *P*-max in the composition is 20-25 per cent more than in gunpowder because of high heat evolution in the chemical reaction of the composition. It is also seen that although there is no change in *t*-max (time to attain *P*-max) appreciably with increased loading density for both compositions, *t*-max for *Mg*-based composition is 5-6 times more than that for gunpowder because of delay in ignition of the composition. Secondly, the results of hot and cold temperature conditioning (Table 4) show that *t*-max is least affected under different temperature conditions.

Table 4. Data on CV firings for *Mg*-based composition and GP-40

Composition	Loading density (g/cc)	Temperature condition: Ambient (27 °C) *P*-max (MPa)	*t*-max (ms)	Hot (+55 °C) *P*-max (MPa)	*t*-max (ms)	Cold (-20 °C) *P*-max (MPa)	*t*-max (ms)
Mg/KNO_3/phenolic resin	0.04	17.0	39.6	18.00	35.00	18.00	36.40
	0.06	24.0	35.0	24.00	33.23	23.36	33.75
Gunpowder ($KNO_3/C/S$)	0.04	14.2	4.8	14.07	4.78	13.70	6.60
	0.06	20.4	4.4	20.38	4.90	20.00	6.20

Data on moisture absorption (Fig. 2) indicate that the composition as well as gunpowder (GP-40) are not much affected by moisture up to 75 per cent RH, but above 80 per cent RH there is a significant moisture absorption in both compositions. Secondly, the composition is less susceptible to moisture as compared to gunpowder. Further, it is also seen from Table 3 that the crushing strength of *Mg*-based composition is comparable with that of gunpowder up to 75 per cent RH but it gets severely reduced beyond 84 per cent RH for gunpowder, while in case of *Mg*-based composition the crushing strength decreases marginally. In addition, the results of the composition exposed to 96 per cent RH as shown in Table 5 indicate that cal-val reduces drastically whereas ignition temperature increases appreciably due to ingress of moisture. This means that the reactivity of the composition is extensively affected by moisture. This has been further supported by CV firing results, which reveal that pressure output decreases as compared to *P*-max at ambient temperature. The data on ISAT(B) test (Table 6) reveal

Table 5. Data on cal-val and CV firings after exposure to 96% RH

Composition	Exotherm by DTA (°C)	Cal-val (cal/g)	CV firing at 0.05 g/cc *P*-max (MPa)	*t*-max (ms)
Mg/KNO_3/phenolic resin	476	1309	11.80	126.00
Gunpowder ($KNO_3/C/S$)		690	12.14	6.44

Table 6. Data on CV firing of *Mg*-based composition after ISAT(B) test with double base propellant

Propellant charge weight: 3.5 g; *Mg*-based composition weight: 1 g

Period of exposure (week)	Ambient *P*-max (MPa)	Ambient *t*-max (ms)	Hot* *P*-max (MPa)	Hot* *t*-max (ms)	Cold* *P*-max (MPa)	Cold* *t*-max (ms)
0	12.30	225.50	13.40	218.00	12.90	220.0
4	12.00	230.00	12.55	219.10	11.30	244.8
13	12.25	222.10	12.40	220.00	11.30	240.0
26	12.00	228.05	12.78	220.00	12.45	247.0
39	12.50	224.00	12.46	224.00	12.89	242.0
52	12.25	220.00	12.50	212.00	12.00	243.0

* Cartridges were conditioned to +45 °C and -26 °C after completion of ISAT(B) test.

that the performance of the composition is not at all affected in different environmental conditions.

Data on ignitability of the *Mg*-based composition used as priming with pyrotechnic compositions (Table 7)

Table 7. Ignition behaviour of *Mg*-based composition and GP-40

Composition	Priming quantity (g)	Main pyrotechnic composition $Mg/Sr(NO_3)_2/NaNO_3$/PVC/polyester (g)	Results
Mg/KNO_3/phenolic resin	0.3	15	Ignited
	0.5	15	Ignited
Gunpowder ($KNO_3/C/S$)	0.3	15	Ignited
	0.5	15	Ignited

indicate that the optimum quantity for ignition of main pyrotechnic composition is 0.3 g, similar to gunpowder. However, 0.2 g *Mg*-based composition in pressed form is difficult to ignite with an electric squib.

Mg/KNO_3/phenolic resin-based composition was further studied by DTA to investigate the role of binder in the combustion phenomenon. The DTA curves for phenolic resin and KNO_3, are shown in Fig. 3 and those for phenolic resin/KNO_3, Mg/KNO_3 and Mg/KNO_3/phenolic resin are shown in Fig. 4. From Fig. 4, it is evident that the ignition of Mg/KNO_3 takes place at 495 °C whereas the ignition of Mg/KNO_3/phenolic resin compositions occurs at 452 °C. This may be attributed to the exothermic reaction between binder and oxidiser before *Mg*-oxidiser reaction, thereby causing reduction in ignition temperature. This mechanism is further supported by the DTA curve of phenolic resin with oxidiser, which gives the onset exotherm temperature at 380 °C, involving chemical reaction between free methylene group obtained from thermal degradation[11] of phenolic resin and oxidiser.

5. CONCLUSION

The results obtained indicate that phenolic resin-based composition is an energetic pyrotechnic composition which can be used as a priming for pyrotechnic compositions for better ignition reliability in place of gunpowder.

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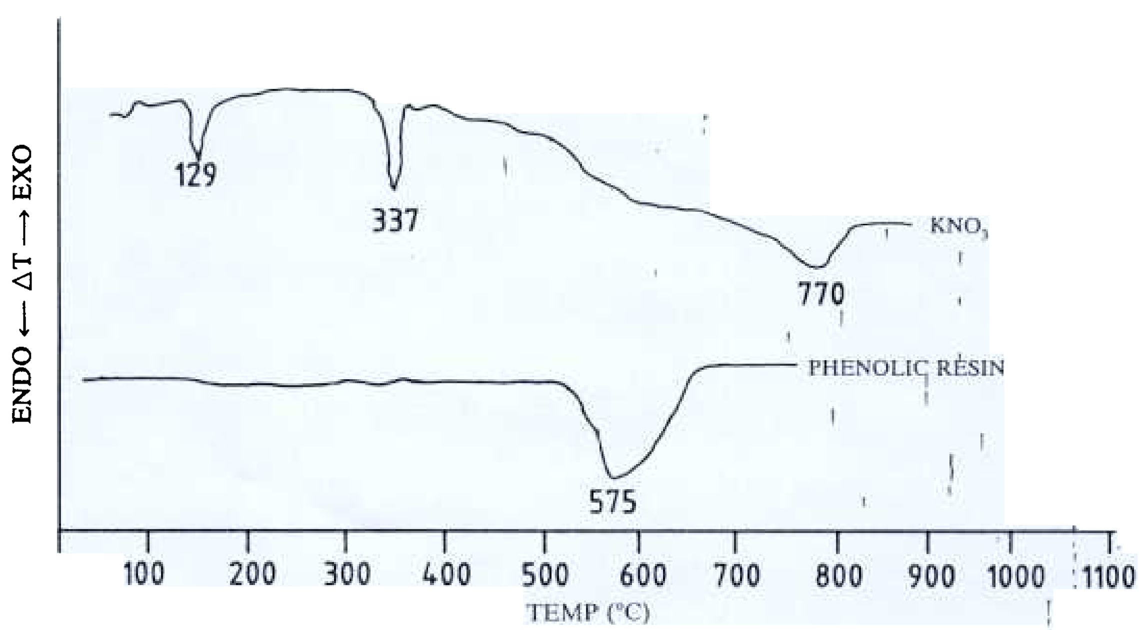

Figure 3. DTA curves for phenolic resin and KNO_3.

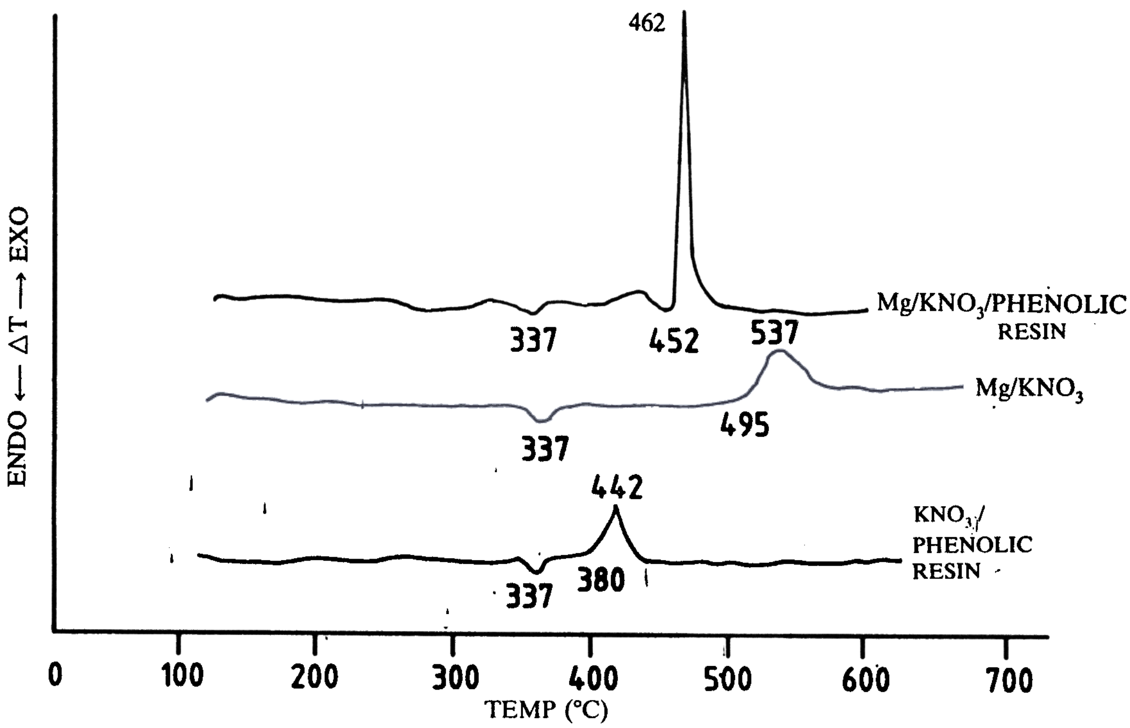

Figure 4. DTA curves for binary and ternary systems.

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