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Research paper

Optimisation of Combustion and Radiometric Behaviour of Magnesium-Teflon-Viton (MTV) IR Decoy Flare Using Grey Relational Analysis

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Abstract: MTV compositions are the most preferred compositions for use in Infrared (IR) decoy flares due to their high energy output. To obtain high IR intensity and low burn rate, systematic combustion studies were done by replacing fuel (5-25 wt.%) and oxidiser (5-25 wt.%) and the addition of burn rate modifiers (1-5 wt. parts) to the baseline MTV compositions. A 100 g batch of each composition was prepared. IR intensity and burn time measurement were done in 3-5 μm using a Dual-band IR radiometer. GRA, a statistical analysis tool for optimisation, was employed to identify and optimise the process parameters to fulfil the objectives. A modified MTV composition was obtained with a 28.2% enhancement in the IR intensity and a 21% reduction in the burn rate as compared to the baseline MTV composition. DSC thermogram of the MTV composition helped in understanding the thermal behaviour. REAL thermochemical code was used to calculate the various equilibrium parameters for all the compositions. Spectral intensity versus wavelength data were obtained by SR-5000N Spectroradiometer. The computational data results matched with the experimental data.

Keywords: MTV, Grey Relational Analysis, GRA, optimisation, combustion behaviour, REAL calculation

1 Introduction

Magnesium (Mg)-Teflon®-Viton® (MTV) compositions are well known to provide very high IR radiation in the mid-IR region due to their vigorous reactions, which are highly exothermic and provide a very high flame temperature of the order of 2000 K [1]. The author has displayed various chemical and physical methods to modify the radiometric performance of MTV composition. Numerous works of MTV composition were carried out to modify the dynamic performance of the composition for various performance parameters, their processing, emission behaviour, *e.g.* [2-4], and for thermochemical and kinetics evaluation, *e.g.* [5-8]). Ju *et al.* [9] were added barium nitrate ($Ba(NO_3)_2$) to the MTV and silicon (Si)-based pyrotechnic composition to study the effect on the oxidation reaction course and the impact of oxidation content on the radiation combustion performance. Cesium dinitramide and ammonium dinitramide were mixed, respectively, with microcrystalline cellulose, and the mixture was burnt in a free atmosphere and temperature as well as VIS-, NIR- and IR spectra were obtained from the combustion process [10].

A study [11] was made to investigate the combustion behaviour and pressure dependence of burn rates of Si and aluminium (Al)-based systems in Teflon®/Viton® mixtures and compared them with reported MTV results. Wang *et al.* [12] have studied to see the effect of Al-Mg alloys with different Mg/Al ratios on the burning rate, combustion products, radiation intensity and specific radiation energy of Al-Mg/PTFE infrared compositions. The IR signature of an MTV flare in a jet engine nozzle was measured and compared with that of a jet engine IR signature [13]. The effects of iron (III) oxide (Fe_2O_3) nanoparticles and graphene on the heat of combustion, the burning rate and the infrared radiance of the pyrotechnic composition based on MTV were studied by Son *et al.* [14]. The use of EXPLO5 thermochemical software was shown to determine the theoretical amount of heat released, the specific volume of gaseous products, and the chemical composition of a mixture of potassium perchlorate ($KClO_4$)/ Mg-Al alloy/ polyurethane/ additives [15]. Investigation into the combustion and radiometric behaviour on the MTV composition by the addition of the burn rate modifiers was also studied [16]. The studies on the combustion and radiometric behaviour of MTV composition by partial replacement of Teflon® and Mg were studied [17, 18] to enhance the IR intensity and reduce the burn rate of the compositions.

MTV compositions have garnered significant attention due to their applications in decoy flares, pyrotechnics, and infrared countermeasure systems. Researchers have explored various approaches to improve their performance parameters, such as material modification, optimising the composition of Mg particles and binders to enhance IR emission, additive inclusion *i.e.* incorporating additives like oxidizers or metallic powders to control burn rates and processing techniques and investigating mechanical or chemical treatments to achieve homogeneity in mixtures. While these studies offer valuable insights, the methodologies often rely on experimental trial-and-error approaches or statistical tools like response surface methodology (RSM) and Taguchi methods.

Optimisation using the Taguchi method for obtaining the best process parameters to obtain the optimum compressive strength of MTV decoy flare pellets were studied [19, 20]. In both cases, optimisation of a single parameter was achieved using various process parameters such as mass, applied load, and dwell time. The application of multiple objective optimisation studies of IR decoy flare compositions was not found during the literature survey. Therefore, in the present study, the aim is to carry out optimisation studies of MTV compositions for multiple objectives. The combustion and radiometric behaviour of MTV compositions are dependent upon mainly two parameters: IR intensity and burn time. In general, the increase in burn rate of MTV composition leads to an increase in IR intensity and *vice-versa*. However, for the practical application of MTV compositions in IR flares, the requirement is to increase the IR intensity and decrease the burn rate. This is required as the flare is intended to give a high IR intensity and a high burn rate so that the flare can be in field of view of missile seeker for a longer time with a high IR intensity for effective decoying.

Studies have been focused on modifying the combustion and radiometric behaviour of MTV compositions for maximising IR intensity and minimising the burn rate, which represents critical performance goals. Achieving an optimal balance among these parameters is essential, as enhancing one often leads to adverse effects on the others. GRA is well-suited for the simultaneous optimisation of multiple parameters. The use of GRA in solving multiple attribute decision-making problems was shown by Kuo *et al.* [21]. The methodology for GRA and its application was also discussed [22]. GRA, an integral part of grey system theory, emerges as a promising optimisation tool for addressing such challenges due to its versatility and effectiveness in dealing with incomplete or uncertain information systems.

In the present study, with the aim of enhancing the IR intensity and reducing the burn rate, data were generated by modifications in MTV compositions by fuel and oxidiser replacement and the addition of burn rate modifiers. The replacement fuels, oxidisers and burn rate modifiers were chosen in such a manner that it modify the combustion reaction and provide selective emitters in the combustion zone to enhance the emissivity, thus enhancing the IR intensity. The objective of the optimisation calculations studies was to optimise the MTV composition by maximising the IR intensity and minimising the burn rate for MTV composition and, in doing so, find out the wt.% of each ingredient for optimum stoichiometry. However, looking at a large amount of data, it was very difficult to point out the optimum stoichiometry that would fulfil the experimental goal. Therefore, in this study, GRA is utilised to provide a robust framework for optimising the MTV composition, ensuring the required performance parameters. The findings contribute to the broader understanding of IR flare compositions and demonstrate the potential of GRA in tackling multi-objective optimisation challenges in pyrotechnic compositions. DSC of the MTV compositions was done to understand the combustion behaviour. REAL thermochemical code was used to determine the equilibrium species and temperature of the compositions [23]. The theoretical calculations were supported by the spectral plots obtained by firing the flare and measured using a spectroradiometer.

2 Methodology for Optimisation Using GRA

GRA quantifies the degree of correlation between multiple objectives using a dimensionless metric known as the Grey Relational Grade (GRG). In the present case, the objective is to simultaneously maximise IR intensity and minimise burn rate for each set of experiments carried out. The following GRA process was applied for the present optimisation studies:

– Step 1: Define the decision matrix:

The decision matrix given in Equation 1 contains experimental data where each row corresponds to a process parameter (*e.g.* replacement of fuel and oxidisier and addition of burn rate modifiers), and each column corresponds to a criterion (*e.g.* IR intensity and burn rate).

$$X = \begin{bmatrix} x_{11} & \cdots & x_{1n} \\ \vdots & \ddots & \vdots \\ x_{m1} & \cdots & x_{mn} \end{bmatrix} \tag{1}$$

– Step 2: Normalize the decision matrix:

Since IR intensity needs to be maximised and burn rate minimised, different normalisation formulas given in Equations 2 and 3 are used.

For IR intensity:

$$x'_{ij} = \frac{x_{ij} - min\,(x_j)}{max(x_j) - min(x_j)} \tag{2}$$

For burn rate:

$$x'_{ij} = \frac{max\,(x_j) - x_{ij}}{max(x_j) - min(x_j)} \tag{3}$$

The normalised matrix is as shown in Equation 4.

$$\mathrm{X}=\begin{bmatrix} \mathrm{x'}_{11} & \cdots & \mathrm{x}_{\prime 1\mathrm{n}} \\ \vdots & \ddots & \vdots \\ \mathrm{x'}_{\mathrm{m}1} & \cdots & \mathrm{x'}_{\mathrm{mn}} \end{bmatrix} \tag{4}$$

- Step 3: Determine the ideal values:
  The reference values x’$_0$ for each criterion are the optimal values given in Equation 5:

  x’$_0$ = [1 1 …. 1] for maximization and x’$_0$ = [0 0 …. 0] for minimization (5)

- Step 4: Calculate Grey Relational Coefficients (GRC):
  The GRC measures the closeness of each experimental run to the ideal solution and is as shown in Equation 6:

  $$\xi_{ij} = \frac{\Delta_{min} - \zeta\Delta_{max}}{\Delta_{ij} - \zeta\Delta_{max}} \tag{6}$$

  where = is the absolute difference between ideal and actual values, is the minimum difference across all experimental runs and criteria, is the maximum difference across all experimental runs and criteria and is the distinguishing coefficient, which is typically taken as 0.5 for the present study as IR intensity and burn rate have equivalent weights in terms of optimisation in their values.
- *Step 5: Calculate Grey Relational Grade (GRG):*
  The GRG is the average of the GRC values for each experimental run and is shown in Equation 7.

  $$GRG_i = \frac{1}{2}(\xi_{i,IR\ intensity} + \xi_{i,burn\ rate}) \tag{7}$$

- Step 6: Rank the experimental runs:
  Based on the the experimental runs are ranked. The composition

stoichiometry with the highest GRG is the best solution, for maximum IR intensity and minimum burn rate.

To achieve the objective of maximising IR intensity and minimising burn rate, three different sets of experimental runs were finalised. In the first experimental run, MTV composition was modified with fuels replacing Mg with 5-25 wt.%, keeping the contents of Teflon® and Viton® constant at 45 and 5 wt.%, respectively. In the second experimental run, Teflon® was replaced by oxidisers varying from 5-25 wt.%, keeping the contents of Mg and Viton® constant at 50% and 5%, respectively. In the third experimental run, 1-5 wt. parts of the burn rate modifiers were added to the baseline MTV composition. GRA was applied in each case to find the optimum combination of fuel wt.%, oxidiser wt.% and burn rate wt. parts added to the baseline MTV composition. In all cases, baseline MTV composition was: 50 wt.% Mg, 45 wt.% Teflon® and 5 wt.% Viton®.

3 Experiments

3.1 Materials

Spherical Mg powder having a particle size of <60 µm was used. Teflon® powder is a polytetrafluoroethylene polymer with a particle size of 450-550 µm. Viton® grade E-60C is a copolymer of vinylidene fluoride and hexafluoropropylene with a fluorine content of 66%. Magnalium, *i.e.* an alloy of Mg and Al (50:50), is a fine powder with an average particle size of <60 µm. Boron-Al-ligature (BAL) powder is manufactured in-house with an average particle size of 2-5 µm. BAL has a composition of boron >79%, Al >12% and Mg >1.5%. The melting point of BAL could not be determined due to its very high value. Si powder has a purity of >99% with an average particle size of <150 µm. Both 1,5-dinitronaphthalene (1,5-DNN, $C_{10}H_6N_2O_4$) and 4-phenylazophenol (4-PAP, $C_{12}H_{10}N_2O$) are fine powders having a particle size <10 µm. Sodium nitrate ($NaNO_3$) and $Ba(NO_3)_2$ are crystalline white powders with an average particle size of <60 µm and a purity of >99%. Cesium nitrate ($CsNO_3$) is a white to off-white crystalline high-purity powder with an average particle size of <60 µm and a purity of >99.5%. Manganese dioxide (MnO_2) powder has a purity of >99.5% with an average particle size of <44 µm, while bismuth trioxide (Bi_2O_3) is a light yellow coloured powder with a purity of >99% and an average particle size of <10 µm. Barium stearate ($C_{36}H_{70}O_4Ba$) is a fine powder with an average particle size of 7.4 µm, and calcium oxalate (C_2O_4Ca) is a fine powder with an average particle size of less than 120 µm. Both were procured from Loba Chemicals.

The Fe_2O_3 used is a nanopowder with an average particle size of less than 2 µm, with a purity of more than 98%. Bis(5-ethylcyclopenta-1,3-diene) iron (diethylferrocene, DEF, $C_{14}H_{18}Fe$) is a liquid with an iron content of 22-24%. 2,2-Bis(ethyldicyclopentadienyl iron) propane (catocene, $C_{27}Fe_2H_{32}$) is a brown-red sticky liquid with an iron content of 23.3-24.4%. While Fe_2O_3 was procured from Alfa Aesar, DEF and catocene were procured from Primodia Chemicals. All the other chemicals were procured from local suppliers of chemicals. The physical properties of chemicals are listed in Table 1.

Table 1. Physical properties of chemicals used in flare compositions

Chemicals / Formula	Molecular weight [g/mol]	Density [g/cm^3]	Melting point [°C]
Mg	24.3	1.74	648.8
Teflon® (C_2F_4)	100.0	2.14	328.0
Viton® ($C_{10}H_7F_{13}$)	374.1	1.85	82.0
Magnalium (Mg-Al)	26.5	2.12	600.0
BAL (B-Al)	12.9	2.47	–
Si	28.1	2.33	1412
1,5-DNN	218.2	1.61	219.0
4-PAP	198.2	1.10	152.0
$NaNO_3$	84.9	2.26	306.8
$Ba(NO_3)_2$	261.3	3.24	592.0
$CsNO_3$	194.9	3.68	414.0
MnO_2	86.9	5.03	535.0
Bi_2O_3	465.9	8.90	825.0
Barium stearate	704.3	1.14	120.0
Calcium oxalate	128.1	2.20	200.0
Fe_2O_3	159.7	5.24	1462.0
DEF	242.1	1.18	–
Catocene	468.2	1.27	–

3.2 MTV composition processing

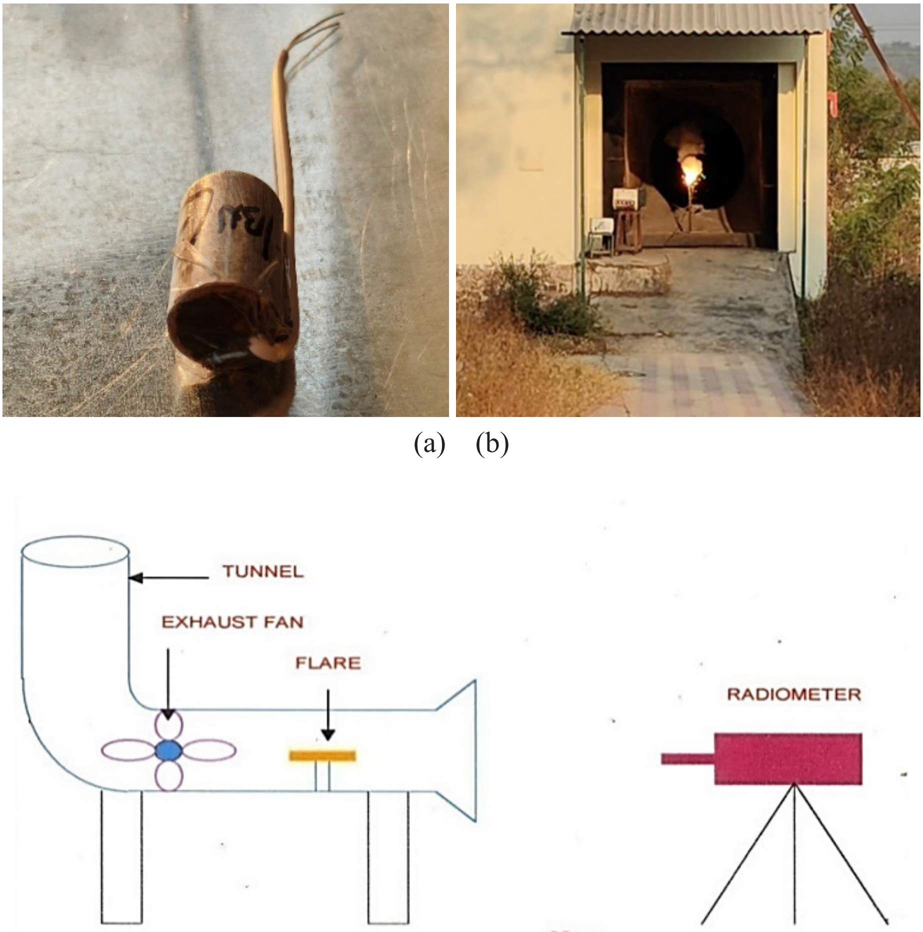
All the chemicals were tested in the laboratory for purity, particle size and melting point. 100 g of the baseline composition is prepared in a batch mixer. The required quantity of Viton® was previously mixed in acetone solvent for 24 h. Fuel and burn rate modifiers were added to Mg in a dry state and mixed. The Mg powder was then coated with the slurry of Viton®. The coated powder was then partially dried in atmospheric conditions. The partially dried Mg was granulated using an 8 BSS sieve. The granulated mixture was thereafter mixed with a pre-weighed quantity of Teflon® powder and the oxidiser in a batch mixer. After mixing, the mixture was passed through an 8BSS sieve three times to ensure homogeneous mixing. The final composition was air-dried in atmospheric conditions for 24 h before packing in antistatic bags. A total of 75 compositions, along with one baseline MTV composition, were prepared.

3.3 Instrumentation

Differential Scanning Calorimetry (DSC) was carried out using a Hyper DSC procured from Perkin Elmer, USA. The instrument is capable of operating within a temperature range of 30-450 °C. Calibration is done using pure indium. The Orion dual-band IR radiometer from CI Systems Israel was used to measure IR intensity and burn time for a single flare simultaneously in two channels. The radiometer has a field of view of 12° with the InGaAs detector working in 1.0-2.5 µm and the PV-MCT detector working in 2.5-5.5 µm wavebands. The plot of spectral intensity versus wavelength was obtained using an SR-5000N Spectro-radiometer from CI Systems Israel. The measurement was carried out in the range of 1.3-14.3 µm. However, the data presented here is for the waveband range of 1.3-5.0 µm. Both the radiometers were calibrated with a blackbody of temperature $T = 1000$ K (SR-2, CI Systems Israel, Cavity blackbody working up to 1200 °C).

3.4 Pelleting of MTV composition

The pelleting of MTV composition was done in a 20 mm diameter steel tube lined with a paper liner from the inside. The paper liner is fixed to the inner surface of the steel tube by using an epoxy resin-hardener mix. Each steel tube consists of the innermost layer of 10 g of the MTV composition, over which 0.5 g booster (50:50 mix of MTV composition and a fast-burning rate priming composition) was placed. The topmost layer consists of a 0.5 g priming composition (gunpowder). The layers of composition were consolidated using a calibrated hydraulic press at 6000 kg load for 15 s dwell time. A total of 6 steel tubes per composition were pressed. In total, 456 steel tubes were filled. Figures 1(a) to 1(c) shows the flare firing along with the radiometric firing setup.


(a) (b)

(c)

Figure 1. Flare assembled with squib (a), flare fired in the wind tunnel (b) and setup of radiometric measurement (c)

3.5 Radiometric measurement of MTV compositions

The radiometric measurement was done by placing the flares over a firing stand in a wind tunnel. A dual-band IR radiometer measuring 3-5 µm was used for recording data during the radiometric firing and kept at a distance of 40 m from the wind tunnel. Each steel tube was placed inside a wind tunnel fitted with an exhaust fan, which provides a wind velocity of 5-10 m/s, that is used to drive away the hot gases that may interfere with the radiometric measurement. An electrical pulse was used to initiate the squib, and IR intensity versus burn time data were recorded once the flare fired. The burn rate was calculated by dividing the height of the flare pellet by the burn time measured by the radiometer.

4 Results and Discussions

4.1 Formation of decision matrix

The Decision matrix was generated for maximising IR intensity and minimising burn rate for fuel replacement and is shown in Equation 8. The process parameters in this study are fuel replacement, oxidiser replacement and the addition of a burn rate modifier.

$$X=\begin{bmatrix} X'_{\text{IR Baseline MTV,IR intensity}} & \cdots & X_{,\text{Baseline MTV,burn rate}} \\ \vdots & \ddots & \vdots \\ X'_{\text{4}-\text{PAP,IR intensity}} & \cdots & X'_{\text{4}-\text{PAP,burn rate}} \end{bmatrix} \tag{8}$$

A similar decision matrix is made for maximising IR intensity and minimising burn rate for oxidiser replacement and is shown in Equation 9.

$$X=\begin{bmatrix} X'_{\text{IR Baseline MTV,IR intensity}} & \cdots & X_{,\text{Baseline MTV,burn rate}} \\ \vdots & \ddots & \vdots \\ X'_{\text{Bismuth Trixode,IR intensity}} & \cdots & X'_{\text{Bismuth Trixode,burn rate}} \end{bmatrix} \tag{9}$$

Another decision matrix is made for maximising IR intensity and minimising burn rate for the addition of burn rate modifiers, which is shown in Equation 10.

$$X=\begin{bmatrix} X'_{\text{IR Baseline MTV,IR intensity}} & \cdots & X_{,\text{Baseline MTV,burn rate}} \\ \vdots & \ddots & \vdots \\ X'_{\text{Catocene,IR intensity}} & \cdots & X'_{\text{Catocene,burn rate}} \end{bmatrix} \tag{10}$$

Table 2 shows the experimental runs 1-76, with the fuel, oxidiser and burn rate modifier added to the baseline MTV composition. Each experimental run's data for IR intensity and burn rate is the average value of 6 firings, of which three firings each are done at +76 and –46 °C conditioning for 3 h.

Table 2. Details of composition stoichiometry for the experimental runs

Experimental runs	Action	Oxidiser [wt.%]	Fuel [wt.%]	Burn rate modifier [wt. parts]	Mg [wt.%]	Teflon® [wt.%]	Viton® [wt.%]
1	Baseline composition	–	-	–	50	45	5
2-6	Fuel replacement		Magnalium: 5-25		45-25		
7-11			BAL: 5-25				
12-16			Si: 5-25				
17-21			1,5-DNN: 5-25				
22-26			4-PAP: 5-25				
27-31	Oxidiser replacement	$NaNO_3$: 5-25	–		50	40-20	
32-36		$Ba(NO_3)_2$: 5-25					
36-41		$CsNO_3$: 5-25					
41-46		MnO_2: 5-25					
46-51		Bi_2O_3: 5-25					
51-56	Burn rate modifier addition	–		Barium Stearate: 1-5		45	
55-61				Calcium Oxalate: 1-5			
61-66				Fe_2O_3: 1-5			
66-71				DEF: 1-5			
72-76				Catocene: 1-5			

4.2 GRA of MTV compositions with replacement of Mg

GRA was applied to the set of experimental runs in which Magnesium was replaced by fuels such as magnalium (runs 2-6), BAL (runs 7-11), Si (runs 12-16), 1,5-DNN (runs 17-21) and 4-PAP (runs 22-26). Baseline MTV composition was considered an experimental run 1. The calculated values of GRC, GRG and ranking of the experimental runs are given in Table 3. Applying GRA to the experimental runs to replace Mg in the baseline composition gives the optimum experimental run 2. The GRG for the experimental run 2 was calculated to be 0.6819. MTV composition having 45 wt.% Mg, 5 wt.% magnalium, 45 wt.% Teflon®, and 5 wt.% Viton® was found to maximise the IR intensity and minimise the burn rate.

Table 3. GRA for the set of experimental runs with the replacement of Mg

Run No.	IR intensity in 3-5 μm [W/sr]	Burn rate [mm/s]	Normalised IR intensity	Normalised burn rate	GRC IR intensity	GRC burn rate	GRG	Rank
1	239.0	3.86	0.75	0.000	0.667	0.333	0.4999	26
2	***289.6***	***3.48***	***1.00***	***0.126***	***1.000***	***0.364***	***0.6819***	***1***
3	279.9	3.23	0.95	0.209	0.912	0.387	0.6498	3
4	276.5	3.37	0.94	0.162	0.885	0.374	0.6296	6
5	247.2	3.32	0.79	0.179	0.705	0.378	0.5415	18
6	272.1	2.94	0.91	0.305	0.853	0.418	0.6354	5
7	231.0	3.51	0.71	0.116	0.633	0.361	0.4972	25
8	246.8	3.37	0.79	0.162	0.703	0.374	0.5382	21
9	247.8	3.05	0.79	0.268	0.708	0.406	0.5568	16
10	251.6	2.94	0.81	0.305	0.727	0.418	0.5726	13
11	220.5	2.64	0.66	0.404	0.594	0.456	0.5252	23
12	276.1	3.54	0.93	0.106	0.882	0.359	0.6205	7
13	263.5	3.56	0.87	0.099	0.795	0.357	0.5759	12
14	251.4	3.52	0.81	0.113	0.726	0.360	0.5431	17
15	247.2	3.39	0.79	0.156	0.705	0.372	0.5383	20
16	229.9	3.21	0.70	0.215	0.629	0.389	0.5090	24
17	196.0	2.01	0.54	0.613	0.519	0.563	0.5414	19
18	140.9	1.62	0.26	0.742	0.405	0.659	0.5321	22
19	156.2	1.34	0.34	0.834	0.431	0.751	0.5912	10
20	132.9	1.14	0.23	0.901	0.392	0.834	0.6133	8
21	95.7	0.84	0.04	1.000	0.343	1.000	0.6714	2
22	234.3	2.19	0.73	0.553	0.647	0.528	0.5873	11
23	203.1	1.83	0.57	0.672	0.539	0.604	0.5715	14
24	125.9	1.36	0.19	0.828	0.382	0.744	0.5629	15
25	108.4	1.07	0.10	0.924	0.358	0.868	0.6130	9
26	87.3	0.94	0.00	0.967	0.333	0.938	0.6356	4

4.3 GRA of MTV compositions with replacement of Teflon®

GRA was applied to the set of experimental runs in which Teflon® was replaced by oxidisers such as $NaNO_3$ (runs 27-31), $Ba(NO_3)_2$ (runs 32-36), $CsNO_3$ (runs 37-41), MnO_2 (runs 42-46) and Bi_2O_3 (runs 47-51). Baseline MTV composition was also included in the GRA of the compositions and taken as an experimental run 1. The calculated values of GRC, GRG and ranking of the experimental runs are given in Table 4. Applying GRA to the experimental runs for the replacement of Teflon® in the baseline composition gives the optimum experimental run 32. The GRG for the experimental run 32 was calculated to be 0.8065. MTV composition having 50 wt.% Mg, 5 wt.% $Ba(NO_3)_2$, 40 wt.% Teflon®, and 5 wt.% Viton® was found to maximise the IR intensity and minimise the burn rate.

Table 4. GRA of the set of experimental runs with the replacement of Teflon®

Run No.	IR intensity in 3-5 µm [W/sr]	Burn rate [mm/s]	Normalised IR intensity	Normalised burn rate	GRC IR intensity	GRC burn rate	GRG	Rank
1	239.0	3.86	0.29	0.658	0.356	0.413	0.5033	22
27	245.8	3.25	0.35	0.859	0.433	0.779	0.6063	11
28	253.4	4.06	0.41	0.592	0.458	0.551	0.5046	21
29	272.2	4.80	0.57	0.349	0.536	0.434	0.4850	23
30	257.3	5.68	0.44	0.059	0.473	0.347	0.4098	25
31	239.1	5.86	0.29	0.000	0.413	0.333	0.3732	26
32	***324.0***	***3.78***	***1.00***	***0.684***	***1.000***	***0.613***	***0.8065***	***1***
33	294.0	3.95	0.75	0.628	0.666	0.574	0.6197	10
34	291.0	4.42	0.72	0.474	0.644	0.487	0.5657	13
35	281.0	4.73	0.64	0.372	0.582	0.443	0.5123	20
36	274.0	5.31	0.58	0.181	0.544	0.379	0.4617	24
37	316.4	3.84	0.94	0.664	0.887	0.598	0.7428	2
38	313.7	4.52	0.91	0.441	0.853	0.472	0.6625	6
39	303.8	4.97	0.83	0.293	0.747	0.414	0.5808	12
40	299.6	5.17	0.80	0.227	0.710	0.393	0.5514	14
41	293.7	5.44	0.75	0.138	0.664	0.367	0.5153	18
42	289.4	3.57	0.71	0.753	0.633	0.670	0.6514	8
43	306.1	4.05	0.85	0.595	0.769	0.553	0.6611	7
44	318.6	4.28	0.95	0.520	0.917	0.510	0.7136	3
45	284.1	4.40	0.67	0.480	0.600	0.490	0.5450	16
46	277.7	4.57	0.61	0.424	0.563	0.465	0.5141	19
47	293.6	3.57	0.75	0.753	0.663	0.670	0.6662	5
48	286.1	3.69	0.68	0.714	0.612	0.636	0.6239	9
49	225.3	3.45	0.17	0.793	0.377	0.707	0.5420	17
50	207.0	3.32	0.02	0.836	0.338	0.752	0.5453	15
51	204.5	2.82	0.00	1.000	0.333	1.000	0.6667	4

4.4 GRA of MTV compositions with the addition of burn rate modifiers

GRA was applied to the set of experimental runs in which burn rate modifiers were added to the baseline MTV composition. Burn rate modifiers used were barium stearate (runs 52-56), calcium oxalate (runs 57-61), Fe_2O_3 (runs 62-66), DEF (runs 67-71) and catocene (runs 72-76). The experimental run 1 was for the baseline MTV composition. The calculated values of GRC, GRG and ranking of the experimental runs are given in Table 5. Applying GRA to the experimental runs for the addition of burn rate modifiers in the baseline composition gives the optimum experimental run 72. The GRG for the experimental run 72, was found to be 0.7323. MTV composition having 50 wt.% Mg, 45 wt.% Teflon®, 5 wt.% Viton® and 1 wt. part catocene was found to maximise the IR intensity and minimise the burn rate.

Table 5. GRA of the set of experimental runs with the addition of burn rate modifiers

Run No.	IR intensity in 3-5 µm [W/sr]	Burn rate [mm/s]	Normalised IR intensity	Normalised burn rate	GRC IR intensity	GRC burn rate	GRG	Rank
1	239.0	3.86	0.35	0.000	0.435	0.333	0.3840	26
52	253.2	3.30	0.48	0.318	0.489	0.423	0.4558	22
53	258.8	2.80	0.53	0.602	0.514	0.557	0.5354	18
54	254.8	2.50	0.49	0.773	0.496	0.688	0.5915	9
55	202.8	2.30	0.03	0.886	0.339	0.815	0.5769	12
56	200.0	2.10	0.00	1.000	0.333	1.000	0.6667	3
57	311.6	3.40	1.00	0.261	1.000	0.404	0.7018	2
58	296.5	3.10	0.86	0.432	0.787	0.468	0.6276	7
59	297.8	3.10	0.88	0.432	0.802	0.468	0.6349	6
60	259.0	2.80	0.53	0.602	0.515	0.557	0.5359	17
61	219.0	2.60	0.17	0.716	0.376	0.638	0.5068	20
62	268.7	3.10	0.62	0.432	0.565	0.468	0.5167	19
63	298.8	3.40	0.89	0.261	0.813	0.404	0.6085	8
64	288.6	3.50	0.79	0.205	0.708	0.386	0.5470	14
65	290.5	3.70	0.81	0.091	0.726	0.355	0.5402	15
66	294.4	3.70	0.85	0.091	0.764	0.355	0.5596	13
67	247.4	3.40	0.42	0.261	0.465	0.404	0.4343	25
68	286.7	3.50	0.78	0.205	0.691	0.386	0.5387	16
69	249.6	3.40	0.44	0.261	0.474	0.404	0.4387	23
70	238.4	3.20	0.34	0.375	0.433	0.444	0.4385	24
71	246.5	3.10	0.42	0.432	0.462	0.468	0.4648	21
72	***311.4***	***3.10***	***1.00***	***0.432***	***0.996***	***0.468***	***0.7323***	***1***
73	276.7	2.80	0.69	0.602	0.615	0.557	0.5861	10
74	270.3	2.70	0.63	0.659	0.575	0.595	0.5846	11
75	250.3	2.30	0.45	0.886	0.477	0.815	0.6457	5
76	229.3	2.20	0.26	0.943	0.404	0.898	0.6510	4

4.5 Finalisation of composition ingredients and stoichiometry

To find out the optimum process parameter by replacing fuel, oxidiser and burn rate modifiers to the baseline MTV composition, the best options (rank: 1) from these experimental runs were tabulated in a new decision matrix to apply GRA. The experimental run 1, for the baseline MTV composition, was also included in the GRA. The calculated values of GRC, GRG and ranking of the experimental runs are given in Table 6. Of all the experimental runs, the effect of a process parameter, *i.e.* the burn rate modifier catocene (1 wt. part) was found to have the maximum effect on the baseline MTV composition for maximising the IR intensity and minimising the burn rate and the GRA for the experimental run 72 was found to be 0.886.

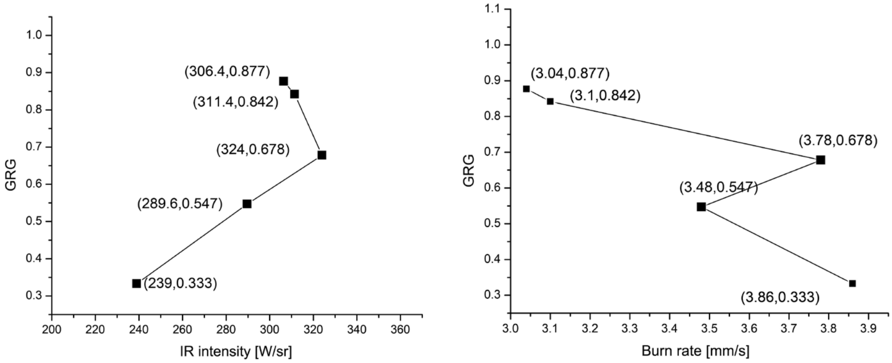
Table 6. GRA for optimising the modified MTV composition

Run No.	IR intensity in 3-5 µm [W/sr]	Burn rate [mm/s]	Normalised IR intensity	Normalised burn rate	GRC IR intensity	GRC burn rate	GRG	Rank
1	239.0	3.86	0.000	0.000	0.333	0.333	0.333	4
2	289.6	3.48	0.595	0.500	0.553	0.500	0.526	3
32	324.0	3.78	1.000	0.105	1.000	0.358	0.679	2
72	***311.4***	***3.10***	***0.852***	***1.000***	***0.771***	***1.000***	***0.886***	***1***

4.6 Proposed modified MTV composition

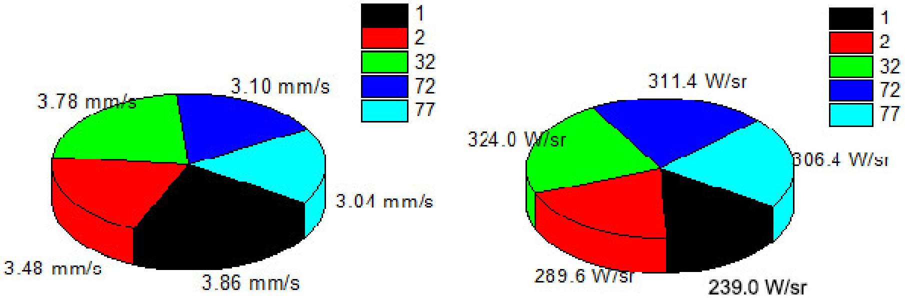
With the analysis of the experimental data using GRA, the effect of replacement of Mg and Teflon® and the addition of burn rate modifier to the MTV composition was successfully completed for optimisation of IR intensity and burn rate. Based on the GRG for the experimental runs 1-76, a new combination of ingredients can be proposed to have the maximum effect on the baseline MTV composition for maximising the IR intensity and minimising the burn rate. This composition was proposed by taking into account the optimum wt.% of Magnalium from Experimental run 77 was proposed for the experimental run with 45 wt.% Mg, 5 wt.% magnalium, 40 wt.% Teflon®, 5 wt.% $Ba(NO_3)_2$, 5 wt.% Viton® and 1 wt. part catocene. A 100 g batch of the proposed MTV composition has been prepared. Steel tubes with the diameter of 20 mm were filled with the modified MTV composition, and three flares were fired after conditioning for 3 h at +76 and –46°C. The IR intensity and burn rate data were obtained by firing the flares and measuring with the radiometer.

To verify that the modified MTV composition is the best option for maximising the IR intensity and minimising the burn rate, the GRA of the experimental runs 1, 2, 32 and 72 were repeated with the experimental run 77. The GRA for the experimental run, 77, was 0.877. The calculated values of GRC, GRG and ranking of the experimental runs are given in Table 7. The GRA shows that the GRG value for the modified MTV composition is the highest, showing that composition stoichiometry for experimental run 77 yields the optimum solution for maximising IR intensity and minimising burn rate. The plots of GRG versus burn rate are shown in Figure 2(a), and that for GRG versus IR intensity for the experimental runs are shown in Figure 2(b). Figure 3(a) shows the distribution of burn rate, and Figure 3(b) shows the distribution of IR intensity for experimental runs. Figures 4(a) and 4(b) show the plot of IR intensity versus burn time in 1.8-2.6 and 3-5 µm, respectively, for the experimental runs.



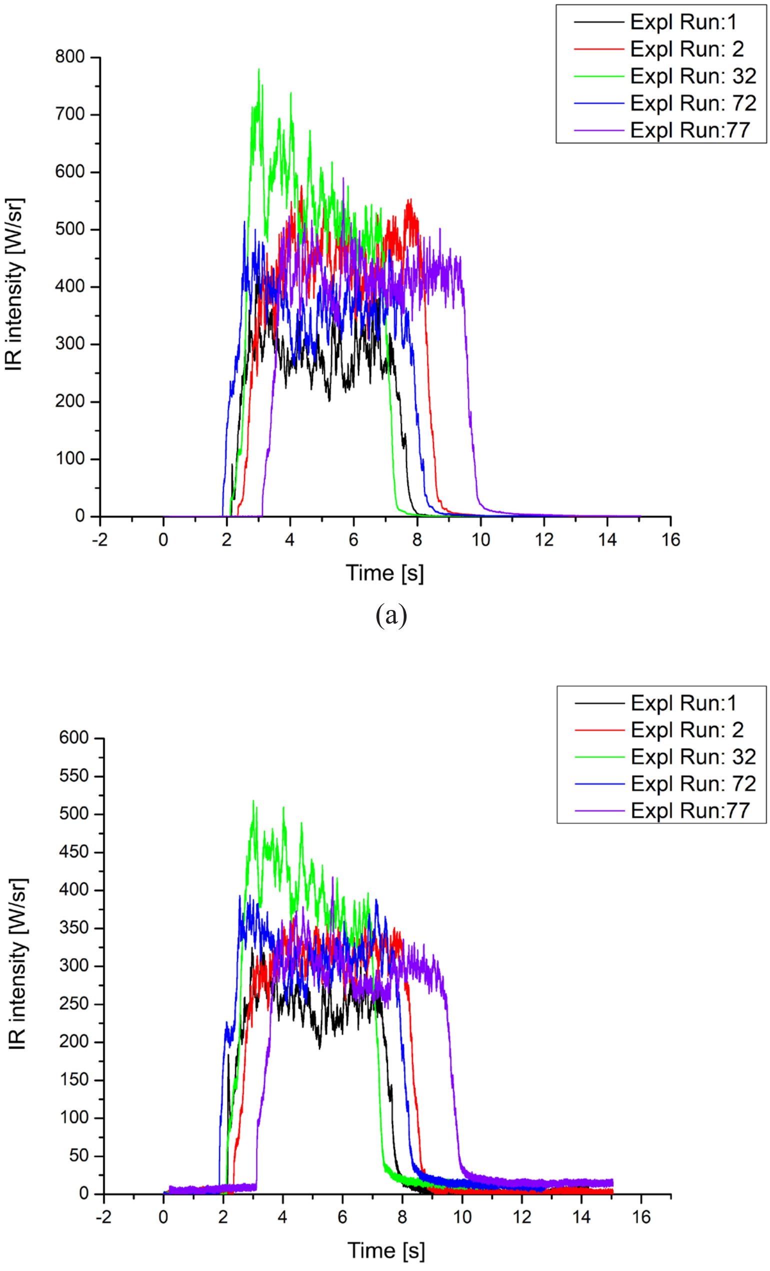
(a) (b)

Figure 2. Variation of GRG with IR intensity (a) and burn rate (b)



(a) (b)

Figure 3. Experimental runs with the distribution of burn rate (a) and IR intensity (b)



(a)

(b)

Figure 4. Plots of IR intensity versus burn time in 1.8-2.6 (a) and 3-5 μm (b)

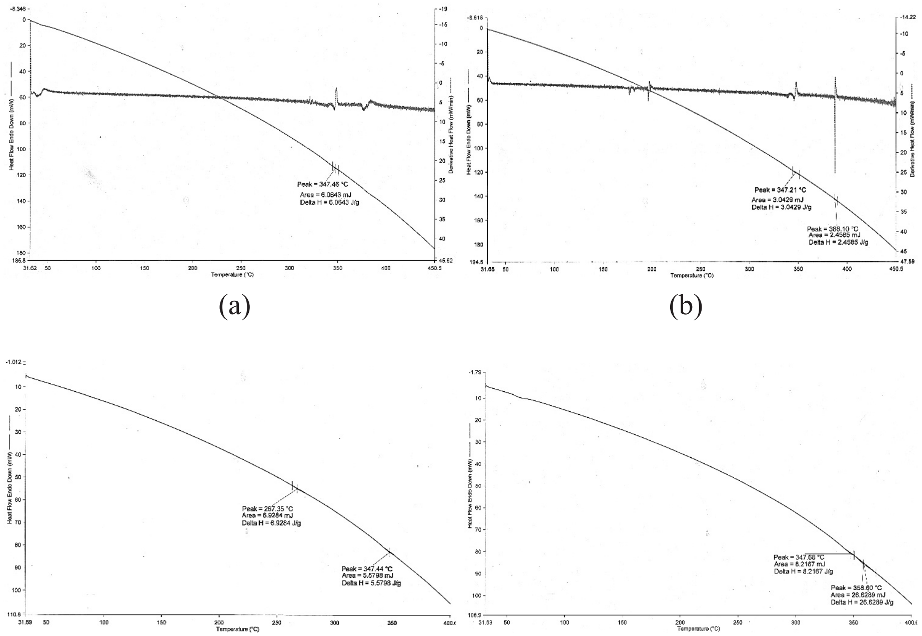
Table 7. GRA of the modified MTV composition

Run No.	IR intensity in 3-5 µm [W/sr]	Burn rate [mm/s]	Normalised IR intensity	Normalised burn rate	GRC IR intensity	GRC burn rate	GRG	Rank
1	239.0	3.86	0.000	0.000	0.333	0.333	0.333	5
2	289.6	3.48	0.595	0.463	0.553	0.482	0.518	4
32	324.0	3.78	1.000	0.098	1.000	0.357	0.678	3
72	311.4	3.10	0.852	0.927	0.771	0.872	0.822	2
77	***306.4***	***3.04***	***0.793***	***1.000***	***0.707***	***1.000***	***0.854***	***1***

5 DSC of MTV Compositions

DSC was employed to characterise the thermal behaviour of MTV compositions, enabling precise identification of phase transitions, thermal decomposition, and other heat-related phenomena critical to their pyrotechnic performance. Approximately 1 mg of each composition was placed in an Al pan within a furnace, with an empty Al pan serving as a reference in a separate furnace. Nitrogen gas was continuously purged into both furnaces at a controlled flow rate of 50 mL/min to maintain an inert atmosphere, preventing oxidative reactions that could obscure the intrinsic thermal properties of the MTV mixture. The DSC instrument dynamically adjusted the heat input to maintain the sample and reference at nearly identical temperatures, measuring the differential heat flow with high sensitivity. The temperature was ramped from 30 to 450 °C at a heating rate of 10 °C/min, a range and rate selected to capture key decomposition events of Teflon® (~450 °C onset), catocene (~350-360) °C and Viton® (~300-400 °C) while minimising Mg ignition under inert conditions.

The DSC was carried out for the MTV compositions in which Mg was replaced with Magnalium (experimental run 2), Teflon® was replaced with $Ba(NO_3)_2$ (experimental run 32), the addition of catocene (experimental run 72), and the baseline MTV composition (experimental run 1). The DSC thermograms are given in Figures 5(a) to 5(d).



(a) (b)

(c) (d)

Figure 5. DSC thermograms of MTV compositions: baseline MTV (a) and compositions with magnalium (b), $Ba(NO_3)_2$ (c) and catocene (d)

DSC for the baseline MTV composition shows only one endothermic peak at a temperature of ~347 °C with a ΔH value of +6.06 J/g. The peak at ~347 °C suggests the melting of Teflon®.

DSC for MTV composition with magnalium shows two endothermic peaks at temperatures of ~347 and 388 °C, with ΔH values of +3.04 and +2.46 J/g, respectively. The first peak at ~347 °C suggests Teflon®'s melting, and the second peak at ~388 °C suggests the likely thermal decomposition of Teflon® to release fluorine for further reaction with the fuels present in the MTV composition. Due to the interaction amongst components, the melting peak of Teflon® has shifted as pure Teflon® melts at 327 °C.

DSC carried out for MTV composition with $Ba(NO_3)_2$ shows two endothermic peaks at temperatures of ~267.3 and ~348 °C with ΔH values of +6.9 and +5.5 J/g, respectively. The low-temperature endotherm peak at ~267.3 °C might relate to the initial degradation of the Viton® binder within the MTV composition. This temperature is consistent with its known thermal decomposition. The second endotherm peak at ~348 °C might be due to the breakdown of Teflon® due to bond dissociation, which might lead to additional reactions involving Mg and

$Ba(NO_3)_2$. $Ba(NO_3)_2$ can also start decomposing, potentially releasing oxygen and contributing to an exothermic reaction in further stages.

DSC carried out for baseline MTV composition with catocene shows two endothermic peaks at temperatures of ~347.6 and ~358.6 °C with ΔH values of +8.2 and 26.6 J/g, respectively. Pure Teflon® has a degradation onset typically above 327 °C and major decomposition around 350 °C. The first endotherm indicates the melting or thermal degradation of Teflon®. The second endotherm indicates the thermal degradation of catocene itself due to its reaction with other components at elevated temperatures, generating a sharp endotherm as energy is absorbed during degradation.

The DSC technique was chosen for its ability to detect subtle endothermic and exothermic transitions, quantify reaction enthalpies, and provide reproducible thermal profiles essential for understanding the stability and energy release of MTV compositions. DSC is an appropriate technique for observing the thermal decomposition behaviour of MTV samples, rather than for analysing their combustion processes, due to its fundamental principles and limitations. DSC measures the heat flow associated with phase transitions and chemical reactions as a function of temperature under controlled conditions, typically in an inert atmosphere like nitrogen or argon. This makes it well-suited for studying the endothermic or exothermic events during thermal decomposition, such as the breakdown of polymeric components (*e.g.* Teflon® or Viton®) or the release of volatile species from MTV samples, providing insights into their thermal stability and reaction energetics.

6 REAL Thermochemical Calculation for Combustion of MTV Compositions

The REAL code is one of several thermochemical codes (*e.g.* CHEETAH, ICT, NASA-CEA) employed to analyse pyrotechnic and explosive materials. It calculates equilibrium states by minimising Gibbs free energy, making it suitable for studying MTV formulations, which are widely used in infrared decoy flares due to their high energy release and spectral emission properties.

Thermochemical computation was done for the modified MTV compositions and the baseline MTV composition using the REAL code [22]. Input data given to the code consists of the mass fraction of the chemical ingredients used in each of the MTV compositions, along with the initial equilibrium parameters, which in this case were $H = 0$ J/g and pressure $P = 0.1$ MPa. REAL code computes the equilibrium combustion species by assuming that the continuity, energy and momentum equations are one-dimensional, combustion is complete and adiabatic, the velocity of the combustion zone is zero, and there is homogeneous mixing of the species in the combustion zone. The enthalpy of formation of the chemical ingredients was also an input parameter, determined using the ICT thermochemical database [24-26]. The output values from the code consist of the mass fraction of species in the combustion zone and the temperature, which is presented in Table 8.

Table 8. REAL thermochemical calculation for the experimental runs

Composition	Unit	Experimental runs				
		1	2	32	72	77
Mg	[wt.%]	50	45	50	50	45
Magnalium	[wt.%]	0	5	0	0	5
Teflon®	[wt.%]	45	45	40	45	40
$Ba(NO_3)_2$	[wt.%]	0	0	5	0	5
Viton®	[wt.%]	5	5	5	5	5
Catocene	[wt. part]	0	0	0	1	1
Combustion temperature	[°C]	2061	2048	1991	2051	1965
Species						
Mg	[mass fraction]	0.229	0.221	0.263	0.229	0.252
MgF		0.119	0.111	0.094	0.115	0.082
MgF_2		0.370	0.341	0.239	0.349	0.201
HF		0.004	0.004	0.003	0.006	0.003
$C_{(c)}$		0.124	0.124	0.098	0.130	0.105
$MgF_{2(c)}$		0.150	0.144	0.229	0.165	0.232
CO		-	-	0.032	-	0.031
H_2		-	-	-	-	0.001
N_2		-	-	-	-	0.005
$Fe_3C_{(c)}$		-	-	-	-	0.001
AlF		-	0.034	-	-	0.037
AlF_2		-	0.005	-	-	0.003
AlF_3		-	0.012	-	-	0.007
BaF		-	-	0.004	-	0.004
BaF_2		-	-	0.028	-	0.027
Fe			-	-	0.002	0.001

The REAL computation shows that the modified MTV composition has the lowest combustion temperature and highest species of selective emitters as compared to the other MTV compositions. The selective emitters such as CO, AlF, AlF_2, AlF_3, BaF and BaF_2 contribute to the IR intensity in the mid-IR waveband, thereby generating higher IR intensity for the modified MTV composition. The MTV composition with $Ba(NO_3)_2$ shows the highest amounts of CO generated, while the condensed carbon ($C_{(c)}$) is the lowest. This helps the composition to maintain a higher IR intensity in the mid-waveband than that in the lower waveband, and therefore, the IR Intensity ratio (ratio of IR intensity in the lower waveband to the mid-waveband) is the lowest for the MTV composition with $Ba(NO_3)_2$. The lower combustion temperature of the modified MTV composition helps to shift the wavelength of maximum radiation to the mid-IR waveband. CO is absent in the compositions in which the combustion temperature is higher, and it is produced (0.031) in the modified MTV composition as well as the MTV composition with $Ba(NO_3)_2$ (0.032). A maximum amount of gaseous MgF_2 (0.370) is produced in the combustion zone by baseline MTV composition. With modification in the MTV composition, the mass fraction of gaseous MgF_2 decreases drastically in both the modified MTV composition (0.201) and the MTV composition with $Ba(NO_3)_2$ (0.239).

In general, the partial replacement of fuel, oxidizer and the addition of burn rate modifiers in MTV composition shows a decrease in the equilibrium temperature of the combustion zone, due to which IR intensity in 3-5 μm waveband is found to increase for the MTV compositions. The various species concentrations along with equilibrium temperature, were found using the REAL code.

7 Spectral Intensity *versus* Wavelength using Spectroradiometer

The plot of spectral intensity versus wavelength for both the baseline MTV and modified MTV compositions was obtained by firing compositions filled in 20 mm diameter steel tubes in a wind tunnel and measured using a spectroradiometer. The spectral plots are shown in Figure 6. The plot shows that the spectral intensity recorded in the wavelength spectrum of the modified MTV composition is higher than that of the baseline MTV composition, specifically in the higher waveband of 3-5 μm.

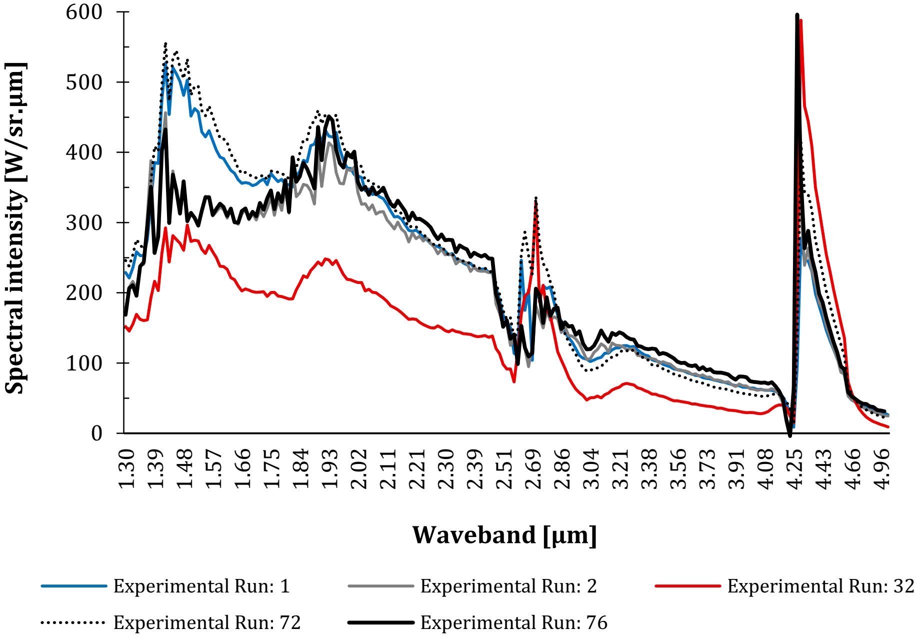


Figure 6. Spectral intensity versus wavelength of MTV compositions

The highest spectral intensity recorded for the modified MTV composition in the 3-5 µm waveband can be attributed to the higher amounts of selective emitters that contribute to the 3-5 µm waveband. All the MTV compositions except the baseline MTV composition emit CO, which acts as a selective emitter. CO is generated in the modified MTV composition, which has a band strength of 250.36 $cm^2 \cdot atm^{-1}$ at 4.67 µm [27]. The lower equilibrium temperature is another factor for the higher spectral emission of the modified MTV compositions in higher wavebands. The higher temperature of the baseline MTV composition causes a shift of the wavelength of maximum radiation to the lower waveband. The condensed species, such as $C_{(c)}$, is lower in the modified MTV composition. The $C_{(c)}$ acts as a grey body, and upon heating, the carbon contributes more to the IR intensities in the lower IR waveband. The amount of free Mg is higher in the modified MTV compositions as compared to the baseline MTV composition. In the secondary reaction, the free Mg reacts with the oxygen from the atmosphere, contributing to higher heat release to form MgO. Therefore, the modified MTV compositions have an overall higher spectral intensity in the waveband spectrum than the baseline MTV composition.

8 Conclusions

- Grey Relational Analysis (GRA) was successfully applied to maximise IR intensity and minimise burn rate in the present study. The process parameters in this study are the replacement of fuel, oxidiser and the addition of burn rate modifiers. Amongst the fuel replacements, MTV with 5 wt.% Magnalium was found to be most optimum, while for oxidiser replacement, MTV with 5 wt.% $Ba(NO_3)_2$ was found to be the most optimum. MTV with 1 wt. part catocene was found to be most optimum for maximising the IR intensity and minimising burn rate for the addition of burn rate modifiers. The ranking was made for each process parameter by evaluating the highest GRG. Amongst all the process parameters identified, the effect of the addition of catocene 1 wt. part (experimental run72) has the maximum effect on maximising IR intensity and minimising burn rate.
- A new composition stoichiometry (experimental run 77) with 45 wt.% Mg, 5wt. % magnalium, 40 wt.% Teflon®, 5 wt.% $Ba(NO_3)_2$, 5 wt.% Viton® and 1 wt. part catocene could be identified using GRA, and the modified MTV composition was proposed to maximise the IR intensity and minimise the burn rate. To prove the hypotheses, trials were carried out for experimental run 77, and GRA was applied on experimental runs, and the results showed that the GRG for experimental run 77 yielded the maximum IR intensity and minimum burn rate. The modified MTV composition yielded a 28.2% enhancement in the IR intensity in the 3-5 μm waveband and a 21% reduction in the burn rate in comparison to the baseline MTV composition. Experimental data from radiometric measurement, REAL calculation and spectral measurement proved the GRA hypotheses. GRA, therefore, not only proved useful in optimising the modified MTV composition but also led to a new optimised MTV composition, and the computational results were validated with the experimental data. Such composition optimisation would be useful in composition selection for use in IR decoy flares for more effective decoying of incoming threats to the aircraft/helicopter.
- The study successfully demonstrated that GRA is a reliable and efficient technique for multi-objective optimisation and optimisation of such multi-objective process parameters. Although simultaneous optimisation of only two objectives was shown in this study, the GRA approach serves as a robust foundation for the simultaneous optimisation of multiple objectives for multiple process parameters for similar pyrotechnic formulations. This is left as future work.

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Authorship contribution statement

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Hitesh Kumar:	foundations, methods, performing the experimental part
Sunil Jain:	foundations, other contribution to the publication
Shaibal Banerjee:	foundations, methods, other contribution to the publication

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