Influence of Different Metal Additives on the Burning Mechanism of Electrically Controlled Solid Propellant

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Abstract: - The focus of this study is to determine the effect of various metal additives on the combustion of the electrically controlled solid propellants (ECSP). In this research, an attempt is made to synthesize ECSP with a baseline composition and then blend it with different metal additives such as aluminum (1%), titanium (1%), tungsten (5-15%), and magnesium (5%). This approach allows studying the effect of each metal additive on the combustion behaviour of the ECSP. ECSP samples used in this study are composed of lithium perchlorate (an oxidant), polyvinyl alcohol (a binder/fuel), and boric acid (a cross-linking agent) as the major components. The weight ratio of lithium perchlorate to water during propellant synthesis is 1:1.85, which is just above the solubility limit. Flame visualization is accomplished using a DSLR camera with a shutter speed of 24 frames per second, which captures the rapid changes in the flame structure. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) of these compositions revealed that the magnesium based ECSP has higher thermal stability as compare to any other ECSP compositions considered in this study. The decomposition temperature for the Mg based ECSP is always above 400 °C which is not particularly seen in any other composition so far. Also, when Mg is added to the baseline composition, the combustion ceases. This is because when the Mg powder is mixed with water, it reacts with the water releasing gases, making structure porous and reduces the electrical conductivity of the Mg-based ECSP sample.

Keywords – Electrically Controlled Solid Propellant, Metal additives, Burning characteristics, Thermal analysis

1 Introduction

Electrically controlled solid propellants (ECSPs) are a type of propellant that ignites only when an external electrical power source is applied and extinguished when the external electric power supply is cut off. While electrically controlled solid propellants (ECSP) have been the subject of research for several decades, there is still limited understanding of how different metal content in the composition of ECSP compares to conventional solid propellants. They have gained popularity in space applications because of their ability to provide stable, controllable, and versatile propulsion. ECSPs have many advantages over conventional solid and liquid propellants. One of the most significant advantages is their ability to provide precise and controllable thrust. Unlike conventional solid propellants, the combustion rate of ECSPs can be controlled and adjusted by varying the electrical current supplied to the propellant. ECSPs also offer higher performance and energy density than conventional liquid and solid propellants. They are relatively lightweight and do not require bulky tanks or pumps, which makes them an attractive option for space applications. Another advantage of ECSPs is their increased safety. Conventional solid and liquid propellants can be hazardous to handle and store, but ECSPs can be stored safely without the risk of accidental ignition.

ECSPs offer several advantages over traditional solid propellants, including their stability, precise ignition control, on/off capability, high specific impulse values, non-toxic and environmentally friendly nature, customizable properties, and potential for high performance. These benefits make ECSPs ideal for use in a variety of space applications, such as satellite propulsion and attitude control, as well as high-performance rocket motors. The electrically controlled nature of ECSPs also provides greater safety in handling and transportation and allows for more precise control over the combustion process, resulting in improved performance and reduced residue production. the low ignition energy requirements of ECSPs make them suitable for use in low-power systems, which is an important consideration for some space applications.

The research focus on the effect of adding metal additives (Al, Mg, Ti) to a baseline ECSP (lithium perchlorate and polyvinyl alcohol) is indeed important in understanding the physio-chemical mechanism of ECSP combustion. The addition of metal additives can significantly affect the burning behavior of ECSPs, including their combustion rate, specific impulse, and exhaust plume characteristics. These additives can act as combustion catalysts and increase the energy released during combustion. The research aims to understand the underlying physio-chemical mechanism of ECSP combustion, which is important for optimizing the performance of these propellants. By gaining a deeper understanding of the combustion behavior of ECSPs, researchers can develop new additives or modify existing ones to enhance their performance, efficiency, and safety. This can ultimately lead to the development of more advanced and effective propulsion systems for space exploration and other applications.

Sawka et al. (2013) researched on hydroxyl ammonium nitrate (HAN) based ECSP. The study demonstrated the potential of HAN-based ECSP for use in micro to macro propulsion technology. However, the self-sustaining combustion observed at high pressure is a drawback that needs to be addressed in future research. Self-sustaining combustion can lead to uncontrolled and potentially dangerous burning, which can negatively impact the performance and safety of the propulsion system. The ability to adjust the burning rate by changing the electrical power input is an important feature of ECSPs. This allows for precise control over the combustion process, which is critical for ensuring safe and efficient operation of the propulsion system. Future research should focus on addressing the self-sustaining combustion issue and further optimizing the performance of HAN-based ECSPs. This can involve exploring different additives or modifying the combustion process to enhance the safety and efficiency of these propellants.

Bao et al. (2022) conducted experiments on the impact of graphite as an additive in HAN-based ECSP. It is interesting to note that the addition of carbon in the form of graphite has both positive and negative effects on the properties of the propellant. On the one hand, the addition of graphite increases the thermal conductivity of the propellant. This is beneficial because it can improve heat transfer during combustion, which can lead to better control over the combustion process and improved performance of the propulsion system. Nevertheless, the addition of graphite reduces the adiabatic flame temperature of the propellant. This can have a negative impact on the performance of the propulsion system as it can lower the specific impulse and reduce the efficiency of the propulsion system.

Gnanaprakash et al. (2022) looked into the effect of tungsten as a metal additive in ECSP. It is interesting to note that the addition of tungsten had both positive and negative effects on the properties of the propellant. On the positive side, the addition of tungsten decreased the decomposition temperature of the propellant by 60℃. This can be beneficial because it can allow for lower temperature operation and potentially increase the safety and reliability of the propulsion system. However, the addition of tungsten also reduced the thermal stability of the ECSP. This can have a negative impact on the performance and safety of the propulsion system because it can lead to uncontrolled burning and potentially dangerous situations.

The study conducted by He et al. (2019) is on the effect of aluminum as a metal additive in ECSP. It is interesting to note that the LP-based ECSP showed higher thermal stability and better electrical control compared to HAN-based composition. The weight ratio of LiClO4/PVA/H2O was varied in the experiments, with ratios of 1/0.43/0.36, 1.0/0.67/0.42, and 1.0/1.0/0.5 tested. The aluminum powder was kept below 20% in the composition, likely to avoid any negative effects on the thermal stability of the propellant. Research reported by He et al. (2019) highlights the importance of carefully selecting the composition and metal additive in ECSPs to optimize their performance and ensure their safety and reliability. The results suggest that LP-based compositions with aluminum additives may be a promising option for future space propulsion technology.

Glascock et al. (2019, 2020) investigated the effects of metal additives (Al, Mg and Ti) on the burning behavior of HAN and PVA based ECSPs. They found that the addition of these metals increased the specific impulse of the propellant and improved its burning behavior, but also led to increased residue production. They also observed that the addition of magnesium led to the production of a green flame during combustion. Gobin et al. (2022) characterized ECSP using the polymer electrolytes with different percentage of polyethylene oxide (PEO), LP and ammonium perchlorate (AP). It is observed that the magnitude of the applied voltage is the dictating factor for the burning rate of the ECSP. It was further noticed that the decomposition was always initiated at the negatively charged cathode. The formation of the liquid decomposition layer when spread to the anode accelerates the decomposition of the perchlorate ions.

Hiatt et. al., (2016) discusses the results of laboratory experiments and basic research on the electrolytic characteristics of Electric Solid Propellant. The high performance electric propellant (HIPEP) formulation is used that consists of 85% wt. S-HAN-5 ionic liquid oxidizer, 15% wt. PVA binder, additives like 2% wt. boric acid for compositions with cross linking agents are used. They developed mathematical models to describe the electrical and thermal characteristics of the

ESP samples and used these models to gain insights into the underlying mechanisms governing the behaviour of ESP.

Baird et. al., (2017) presents a study on the thermochemistry of combustion in a composite material composed of polyvinyl alcohol (PVA) and HAN. Proposed the mechanism of PVA and HAN based combustion. The study provides important insights into the thermochemistry of combustion in PVA + HAN composite materials and highlights their potential for use in propulsion systems. The findings suggest that PVA + HAN composites have favourable combustion properties and could be a promising alternative to traditional propellants. They have reported about the effect of variation in the electrode area ratio, that dictates where the preferential burning of the ECSP (HAN + PVA based) would take place. Combustion always takes place at the smaller electrode irrespective of the polarity of the electrodes.

It is important to consider compatibility when developing ECSP, as certain combinations may lead to undesired outcomes related to incomplete combustion or hypergolic effects. Our findings suggest that the addition of Mg in the LP-based ECSP did not result in any combustion, due to non-formation of liquid layer at the surface of ECSP sample when DC power is supplied. The thermal analysis tells that the significant exothermic peaks for LP-Mg based ECSP appeared at higher temperatures as opposed to other metals considered in this work. Meanwhile, addition of Mg to the HAN-based ECSP produced hypergolic effects. Overall, the work performed here highlights the importance of compatibility testing when developing ECSPs.

2 Experimental Setup

2.1 Propellant preparation

The main components of the ECSP samples in the study are LP (oxidizer, 99% purity from Alfa Aesar Ltd.), polyvinyl alcohol (binder/fuel, molecular weight 146,000-186,000, degree of hydrolysis >99% from Sigma-Aldrich Ltd.), and boric acid (cross-linking agent). The study uses Mg (particle size 10 µm, from US Research Nanomaterials Inc.), Ti (particle size 800 nm, from US Research Nanomaterials Inc.), and Al (particle size 10 µm, from US Research Nanomaterials) as metal fuel additives. Different ECSP compositions were synthesized to study the effect of metal additives. The weight ratio of LP to water was kept at 1:1.85 (slightly higher than solubility limit). Three ECSP compositions were created with different metal additives: 1st composition with 5%

Mg, 2nd with 1% Ti, and 3rd with 1% Al. The ingredients were mixed using a planetary centrifugal mixer (Thinky ARE-310, Japan) for 45 minutes to homogenize them after LP and PVA were dissolved in water. The different compositions and metal additive content are listed in Table *1* of the study and Figure *1* shows the cured baseline composition.

Table 1: ECSP composition.

Ingredients	M0 (mass in g) Baseline	M1 (Al based, 1%)	M1 (Ti based, 1%)	M5 (Mg based, 5%)
Distilled water	5.388	5.3228	5.3228	5.063
LP	2.912	2.8772	2.8772	2.737
PVA	1.0	1.0	1.0	1.0
Metal	0.0	0.1	0.1	0.5
Glycerol	0.5	0.5	0.5	0.5
Boric acid	0.2	0.2	0.2	0.2

Figure 1: Cured ECSP sample (baseline composition).

2.2 Setup design and flame visualization setup

Experiments were done to qualitatively understand the behavior of the ECSP combustion with different metal additives. There are two arrangements made to conduct the experiments. One set of arrangement includes the molybdenum bottom electrode and the top electrode made of nichrome wire. Moreover, the effect of the polarization of the direct current (DC) voltage was checked. Figure 2 shows such setup with nichrome wire, and Fig. 3 shows a set up with mesh electrode. The second set of the arrangement involved a mesh electrode placed above the ECSP to allow for visualization of the combustion process. This setup was used to observe the flame structure and propagation. Both setups allowed for the measurement of the electrical conductivity and voltage across the ECSP during combustion, as well as the collection of data on the combustion rate and characteristics. The experiments were conducted with ECSPs containing different metal additives, including Al, Ti, and Mg, in order to investigate the effects of the additives on the combustion behavior of the propellant. The second set of arrangement aims at directing the thrust generated by the ECSP combustion using the mesh electrode. This task was accomplished by utilizing the mesh type electrode at one end and the solid electrode at the other end. These electrodes for second arrangement were made of Al, so that the mesh design and fabrication become feasible.

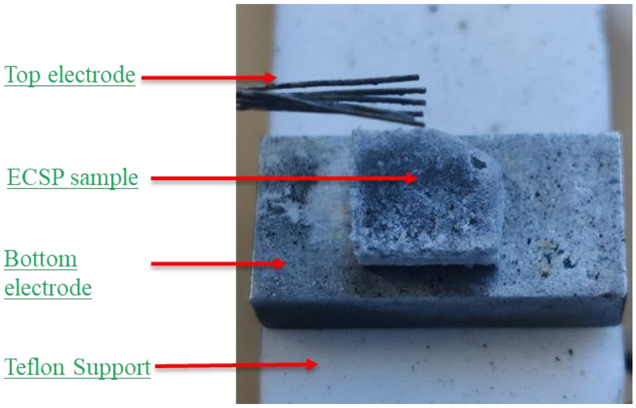


Figure 2: Setup with nichrome wire as one electrode.

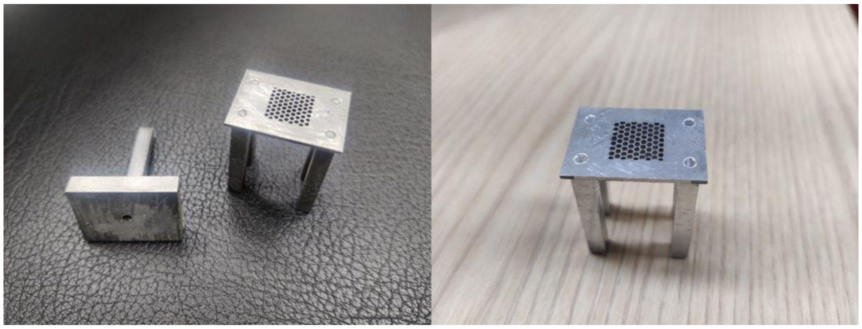


Figure 3: Mesh electrodes made of aluminium.

Experiments were conducted to visualize the flame of the metal based ECSP propellant under open atmospheric conditions. A nichrome wire is used as an electrode. The propellant was kept in the open atmosphere on top of the molybdenum electrode connected to the negative wire of the DC power source. The positive wire of the DC power source was connected to the nichrome wire which acted as the second electrode. A DSLR camera with 24 fps was used to capture the video of the ECSP combustion. During the experiments, it was observed that the flame of the metal-based ECSP propellant had a yellowish-green color, which indicates the presence of metal ions. The flame also had a bright and intense appearance, indicating high combustion efficiency. The video footage showed that the flame front propagated from the nichrome wire electrode towards the molybdenum electrode, indicating that the preferential burning occurred at the positive electrode. The change in polarity did not affect the propagation direction of the flame front. The experiments also showed that the higher voltage of 300 V resulted in a more intense and brighter flame compared to the lower voltage of 200 V. It is noticed that the burning happens at the electrode where the less area is in contact with the propellant. So, the flatness of the propellant surface is important. But it is seen that for all the ECSP either metallized or non-metallized, they have some level of porosity while maintaining the flatness of the surface was difficult. Mesh electrodes were fabricated to direct the thrust generated from the ECSP combustion to the desired direction. The mesh size is an important factor in this design, as it determines the size of the openings through which the product gases can escape. A smaller mesh size will restrict the gas flow due to the formation of the condensed phase layers, while a larger mesh size will allow for more uniform ejection of the product gases. This mesh design can be used to control the direction of the thrust.

2.3 Characterization method

Differential scanning calorimetry (DSC 3+, Mettler Toledo) and thermogravimetry (TGA 2, Mettler Toledo) were used for the thermal investigation of the ECSP samples in order to characterize them at various heating rates of 10 ℃ /min and 20 ℃ /min. Along with thermal analysis, surface morphology was observed using the scanning electron microscopy (SEM) imaging, and its elemental mapping was done using the energy dispersive spectrometry (EDS) analysis of the ECSP samples.

3 Results and Discussion

3.1 Flame visualization with nichrome wire as one electrode

Experiments were conducted to understand the effect of polarity on the preferential burning of the ECSP at the electrodes. It was observed that the burning initiates at the electrode where the area of the electrode is less. This is because the energy power density is high at the point where the area is less. The use of negative or positive polarity would not hinder burning of the ECSP if one of the electrode areas is less compare to the other electrode. This implies that the flatness of the ECSP sample surface is crucial to maintain the unidirectional combustion. Figure *4* shows the collage of images of ECSP combustion with one of the electrodes as nichrome wire. Images are sequentially stacked row-wise. Figure *5* shows the typical current and voltage data when the nichrome wire is used as one of the electrodes. The total power required to burn the ECSP sample remains fixed. Extra energy imparted to ECSP sample in terms of the excess voltage results in the increased burning rate. In the voltage and current graph, it is seen that when the nichrome wire comes in contact with the ECSP sample, the current rises and correspondingly voltage dips.

Figure 4: Collage of images row wise showing the ECSP combustion.

ECSP sample is kept in the molybdenum electrode, where this electrode is attached to the negative side of the DC supply. The nichrome wire electrode is connected towards the positive side of the DC supply.

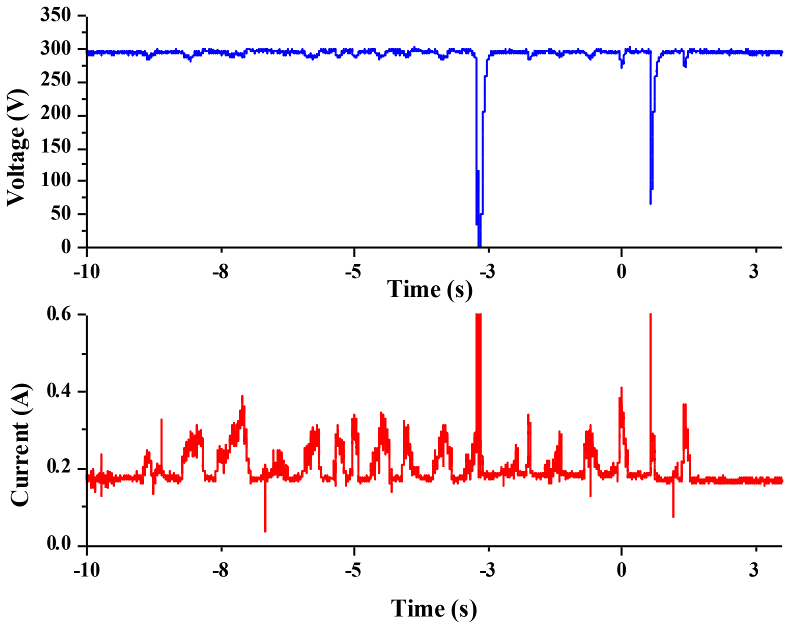


Figure 5: Typical voltage and current data for electrode with nichrome wire.

3.2 Flame visualization with mesh electrodes

Flame visualization experiments with mesh electrode provides insight into the usefulness of the exhasut gases during the burning. The thrust can be controlled by varying the applied voltage. To ignite the ECSP samples, mesh electrode made of Al have been fabricated, the voltage is supplied to the ends of the electrodes, and the combustion occurs at the electrode where the mesh is present. Sequential burning images of one of the ECSP sample are illustrated in Figure 6. The mesh size is an important factor in this design, as it determines the size of the openings through which the product gases can escape. A smaller mesh size will restrict the gas flow due to the formation of the condensed phase layers, while a larger mesh size will allow for more uniform ejection of the product gases. This mesh design can be used to control the direction of the thrust. Figure *6* shows the collage of images of the ECSP sample burning usingt he mesh electrode. Figure *7* shows the voltage and current signals for the mesh electrodes.

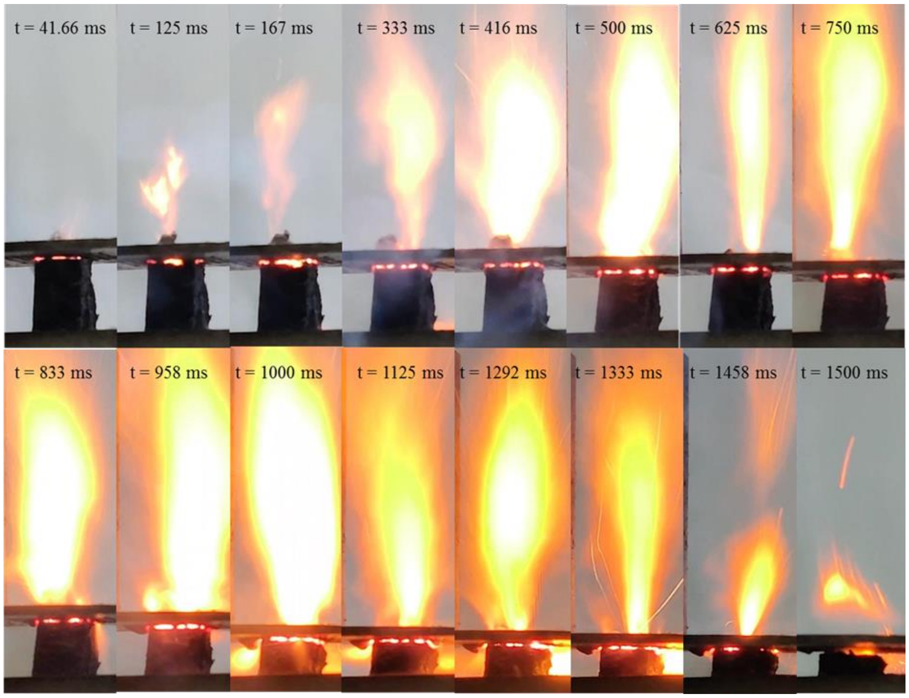


Figure 6: ECSP combustion using the mesh electrode.

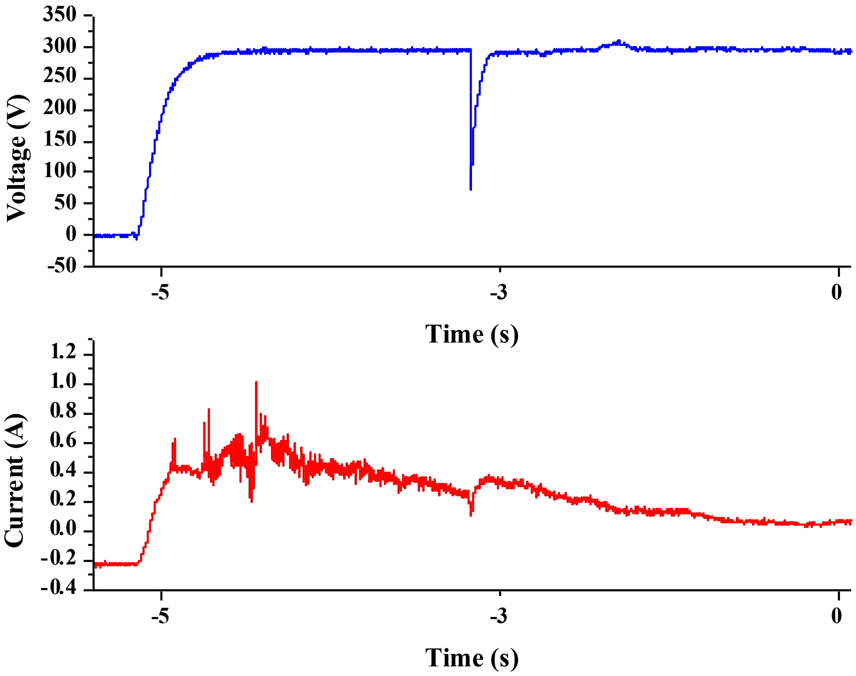
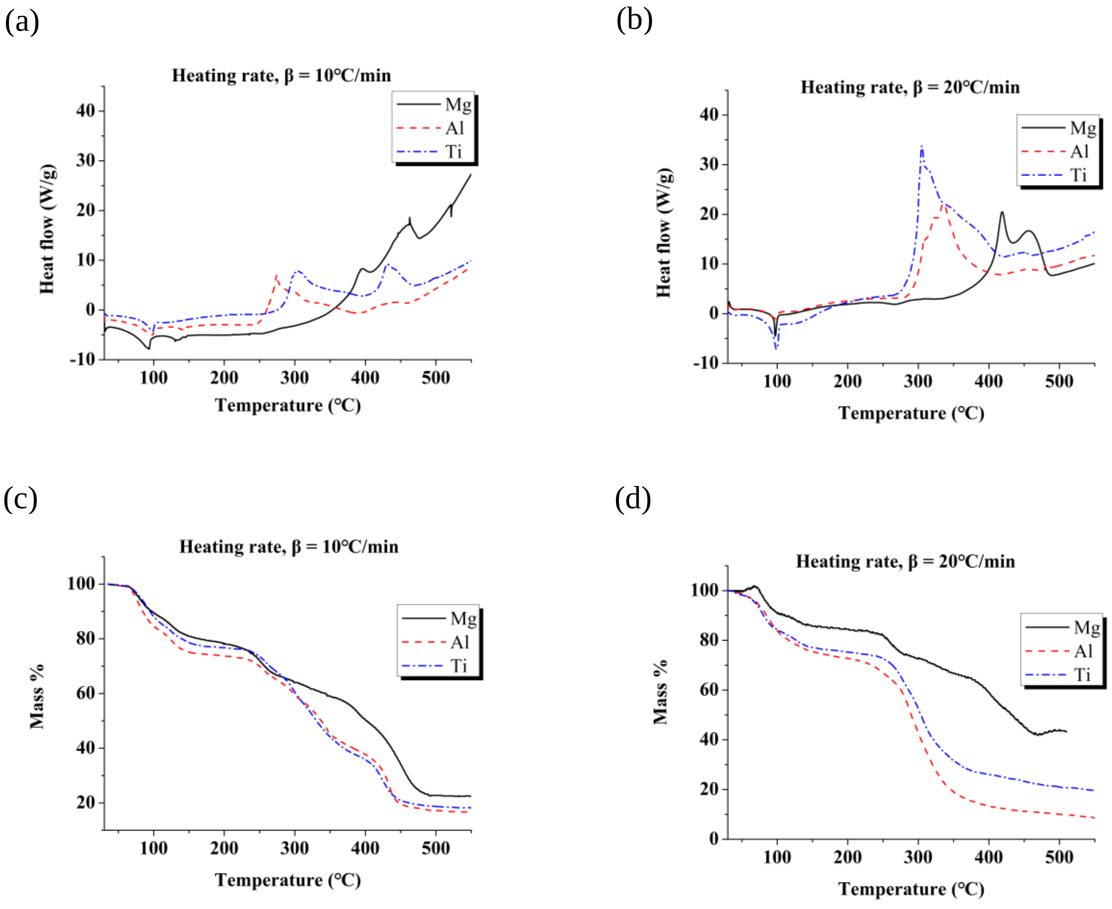


Figure 7: Typical voltage and current data for electrode with mesh.

3.3 Thermal analysis of the LP based ECSP samples

Thermal analysis of the different ECSP samples with different metal additives were conducted to obtain the heat of reaction and understand the thermal behaviour of the ECSP. The DSC, TGA and dTG curves for Mg, Al and Ti based ECSP samples at heating rates of 10 °C/min and 20 °C/min are shown in Figure 8.

It is observed that the thermal stability of the Mg based ECSP is much pronounced as compared to any other ECSP samples with other metal additives. The decomposition of the Mg based ECSP sample happens in the range of 400 °C - 500°C as compare to two other ECSP samples with Al and Ti that happens in the range of 300 °C – 400 °C. Mass loss of the Al-based compositions is higher as compare to the Ti and Mg based ECSP samples. It is noticed that the mass loss of the Mg based ECSP is lesser among the three compositions by well over 50%.



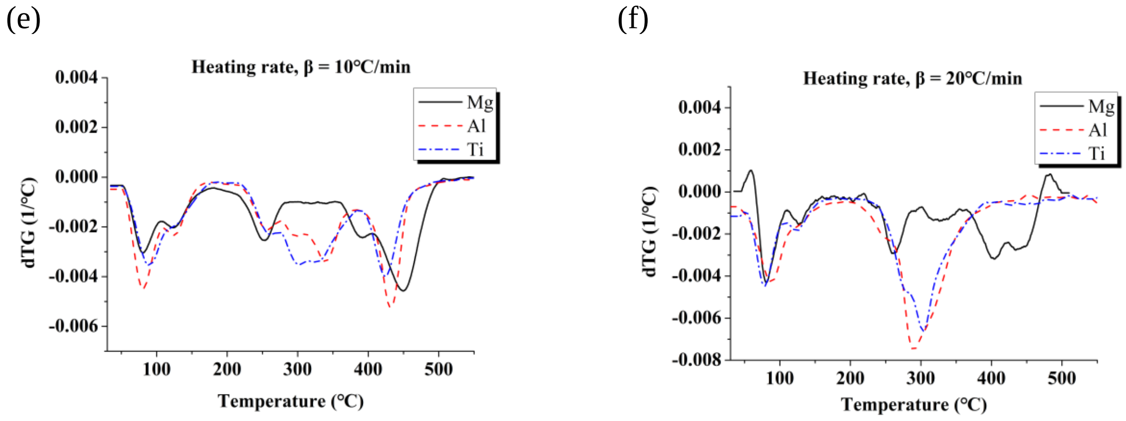


Figure 8: (a) DSC curve for Mg, Al and Ti based ECSP at heating rate of 10 ℃/min (b) DSC curve for Mg, Al and Ti based ECSP at heating rate of 20 ℃/min. (c) TGA curve for Mg, Al and Ti based ECSP at heating rate of 10 ℃/min (d) TGA curve for Mg, Al and Ti based ECSP at heating rate of 20 ℃/min (e) dTG curve for Mg, Al and Ti based ECSP at heating rate of 10 ℃/min (f) dTG curve for Mg, Al and Ti based ECSP at heating rate of 20 ℃/min

DSC and TGA of the three different metallized ECSP have been conducted to obtain the heat of reaction and understand the thermal stability. DSC experiments were conducted in the temperature range of 30 ℃ – 600 ℃ with two different heating rates of 10 ℃/min and 20 ℃/min under the nitrogen ambience. DSC curve of all the ECSP samples shows an endothermic peak at 98 ℃. The endothermic peaks at heating rates of 20 ℃/min are more pronounces as compared to the DCS curves of all the ECSP samples at the heating rate of 10 ℃/min. In both heating rates of 10 ℃/min and 20 ℃/min, it is seen that the decomposition temperature of the Al and Ti based ECSP samples fall in the temperature range of 280 ℃ to 380 ℃.

At heating rate of 10 ℃/min, it is noticed that the second exothermic peak for the Ti based ECSP is observed above 400 ℃. Similar observation is not noticed for the Ti based ECSP at the heating rate of 20 ℃/min. The relative mass loss for all the ECSP compositions is up to 10 % at different heating rates during the first endothermic peak at the DSC curve, and this mass loss is due to the evaporation of the water in all the ECSP samples considered in this study.

The decomposition of the Al and Ti based ECSP are initiated at around 280 ℃, where the exothermic peaks can be seen between the temperature range of 280 ℃ to 380 ℃. A significant amount of mass loss happens to the Al based ECSP as compare to the Ti based ECSP. For Al based ECSP, the mass loss after the decomposition is around 80 %, and for Ti based ECSP, the mass loss after the decomposition is around 50 %. This means that the thermal stability of the Al based ECSP is less relative to the Ti based ECSP. The decomposition of Mg based ECSP starts at the temperature around 380 ℃ at the heating rate of 10 ℃/min, and it starts at around 400 ℃ for the heating rate of 20 ℃/min. It is noticed that the thermal stability of the Mg based ECSP is higher than any other composition considered in this study. Table 2 lists the heat of reaction and the mass loss during the thermal analysis of the various ECSP samples.

Table 2: Heat of reaction and mass loss for different ECSP samples.

Samples	Average heat of reaction (J/g)	Average mass loss (%)	Heating rate (℃/min)	Decomposition temperature (℃)
Mg (5%)_ECSP	1862.9	55 & 65	10	389 & 462
Al (1%)_ ECSP	2552.17	22	10	275
Ti (1%)_ ECSP, First peak	1860.89	22	10	303
Ti (1%)_ ECSP, second peak	892.78	61	10	431
Al (1%)_ ECSP	2413.42	80	20	336.29
Mg (5%)_ECSP	2082	57 & 65	20	418.76 & 459.58
Ti (1%)_ ECSP	4209	30	20	305

The decomposition temperature for the Mg based ECSP is always above 400 ℃. When compared to other samples with different metal additions, it has been shown that the thermal stability of the Mg-based ECSP is significantly more prominent. The decomposition of the Mg-based ECSP sample takes place between 400 ℃ and 500 ℃, whereas the decomposition of the Ti and Al-based ECSP samples takes place between 300 and 400 ℃. Average heat of reaction for Ti based ECSP is highest among the considered metal additives for the ECSP formulation. Though Mg has the lowest heat of reaction relative to other ECSP compositions containing Al and Ti metal additive, Mg based ECSP does not burn owing to not formation of liquid layer over the ECSP sample surface during the DC power supply, whereas all other metal-ECSP compositions forms the liquid layer when DC power supply is applied.

3.3. Surface morphology and elemental mapping

Surface morphology of three ECSP samples are considered here. The SEM imaging of the samples shows that the homogeneity in the compositions after the mixing and the curing process. Figure 9 shows the SEM imaging of the Al based ECSP sample, and its elemental mapping is indicating the presence of Al in a homogenous manner in the ECSP composition.

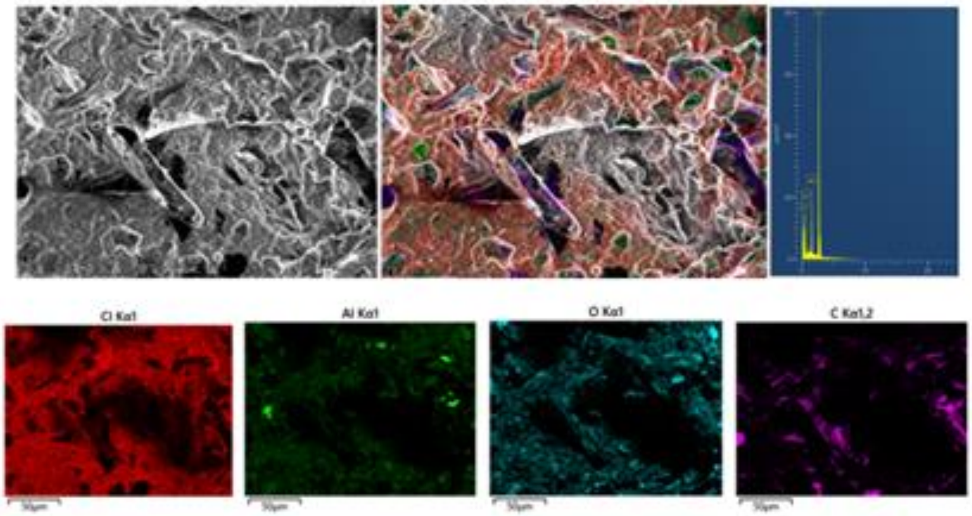


Figure 9: SEM imaging and elemental mapping.

3.4 Effect of Mg in LP based composition

ECSP composition with Mg has shown a peculiar behavior which has not been seen in any other composition tested. After adding Mg powder in the mixture consisting of water, LP, and PVA, a froth formation was observed, and the gases were evolved out of the mixture, probably the reaction of the Mg with the water. The evaporation of the water started rapidly. The potential reaction mechanism occurring during the mixing of Mg with water is suggested as follows:

Reactions of Mg with water: When exposed to steam, Mg changes from Mg to Mg-oxide and hydrogen.

$$Mg(s)+H_2O(g)\rightarrow MgO(s)+H_2(g) \tag{1}$$

When exposed to water, the reaction is a bit different. The reaction does not cease as Mg hydroxide becomes insoluble in the water.

$$Mg(s)+2H_2O(g)\rightarrow Mg(OH)_2(s)+H_2(g) \quad (2)$$

Reactions of Mg with oxygen: When exposed to oxygen, Mg turns into Mg-oxide.

$$2Mg(s)+O_2(g)\rightarrow 2MgO(s) \quad (3)$$

It was found that burning ECSP samples at different voltages resulted in the formation of a liquid layer at lower voltages, which enhanced combustion by distributing the decomposed products. This ensured adequate combustion through contact with the electrode. The liquid layer was seen only at low voltage conditions and not at high voltage conditions. It was observed that when voltage was supplied to ECSP samples, a liquid layer was formed. This behavior was not seen in samples containing Mg, as the reaction of Mg with water released gases that made the structure porous and reduced the electrical conductivity. With these set of experiments, it can be hypothesized that the formation of the liquid layer is an important step in the advancement of the ECSP combustion. In most of ECSP works of the past, researchers have used HAN a basis composition. HAN is hygroscopic in nature likely to absorb the moisture from the surrounding. This in turn increases the conductivity when applied with the voltage, and thereby an adequate combustion can follow. Thermal analysis revealed that the peak of the major exothermic reaction has shifted towards a higher temperature at high heating rates for all the composition of ECSP considered in this work.

4 Conclusion

The study aims to analyze the impact of adding different metal (Al, Ti, and Mg) to an electrically controlled solid propellant (ECSP) composition on the combustion behavior. The formation of a liquid layer at lower voltages enhanced the combustion process by distributing the decomposed products of the LP and PVA in the surrounding area of the ECSP where the contact of the electrode occurs, effectively ensuring adequate combustion. However, the formation of the liquid was seen only at low voltage conditions. Moreover, in the Mg-based ECSP samples, such behaviour is not observed. This is because when the Mg is mixed with water, it reacts and releases the gases that make the structure become porous and reduce the electrical conductivity of the Mg-based ECSP. The thermal analysis at heating rates of 10 ℃/min and 20 ℃/min tells that thermal stability of the Mg based ECSPs is higher. Also, the decomposition of Mg-based samples occurred at higher temperatures compared to both Al and Ti based ECSPs. Though the decomposition of the Mg based samples happens at high temperature, Mg metal additive is not suitable for the LP based composition as it doesn't burn while applying the DC power source. Mg-based ECSP composition ceases to burn owing to the fact that liquid formation on the ECSP surface does not happen in Mg-based ECSP as opposed to the other compositions considered in this study. This non-formation of liquid layers diminishes the conductivity completely for Mg-based ECSP, resulting in no combustion.

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