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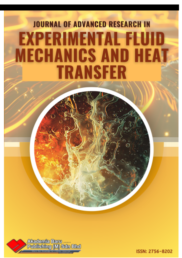

Heat of Combustion and Thermal Decomposition of Ammonium Perchlorate/Sorbitol Solid Propellant with Metal Additives

Muhammad Zakwan Azizi[1], Ahmad Hussein Abdul Hamid[1,*], Zuraidah Salleh[1], Zulkifli Abdul Ghaffar[1], Alif Abni Adnan[1], Izzat Najmi Yaacob[2], Noraafiza Salleh[3], Wilfredo Jr. Kintanar Pardorla[4]

[1] School of Mechanical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam Selangor, Malaysia
[2] Department of Aerospace Engineering, Universiti Putra Malaysia, 43400 Serdang, Malaysia
[3] Science & Technology Research Institute for Defence (STRIDE), Malaysia
[4] Aerospace Engineering Faculty, Ateneo de Davao University, Philippines

ABSTRACT

Ammonium perchlorate (AP) stands out as the predominant oxidizer in solid rocket propulsion systems, while sorbitol, frequently employed as a fuel in sounding rockets, is expected to generate non-hazardous gases during combustion. This study reports on the effect of sorbitol as potential organic green fuel in addition to additive metals on heat of combustion, thermal properties and specific impulse for ammonium perchlorate based solid propellant. The specific impulse was measured using PROPEP 3.0 (Propellant evaluation program). Solid propellant characterization included measuring energy using a bomb calorimeter and conducting thermal analysis using DSC and TGA. Various compositions were prepared by varying the AP and sorbitol content in the propellant, and additive metals like magnesium and ferrite were introduced. The findings reveal that the formulation of 77% ammonium perchlorate into the 23 % sorbitol releases the optimum combustion energy at 2144.47 KJ/mol and lowers the thermal decomposition temperature of AP to 210.38 °C compared to common AP solid propellant formulation. Meanwhile, the addition of 1 % magnesium metal to the mixture significantly improves the combustion energy, propellant performance, and decreases thermal decomposition temperature of the AP/sorbitol. The addition of 0.05% ferrous oxide shows a catalytic capability to decrease the ignition temperature of the sample to 199.15 °C and merge two exothermic peaks into single peak. The main outcome of this work is, AP can interact synergistically with sorbitol mixed with both additive metals by increase the heat combustion, specific impulse, improve AP sensitivity toward heat and lower the thermal decomposition temperature.

Keywords:

Ammonium perchlorate; sorbitol; specific impulse; thermal decomposition; ignition temperature; catalyst

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1. Introduction

Composite propellants are solid propellants that are made up of oxidizing agents, binders, fuels, and various additives. Ammonium perchlorate (AP) is a common oxidizing agent, hydroxy-terminated polybutadiene (HTPB) is a binder that can also be used as fuel, and aluminum (Al) powder is a metal fuel. AP is a popular oxidizing agent that is typically employed as the primary component of solid rocket propellants. The solid rocket propellant, which comprises ammonium perchlorate, aluminium

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[*] *Corresponding author.*
E-mail address: hussein@uitm.edu.my

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powder, and hydroxyl-terminated polybutadiene, stands as the primary choice for most contemporary missiles and rockets [1]. Ammonium perchlorate is a chemical compound which is stable that decomposes gradually at low temperatures. Consequently, it is crucial to enhance the decomposition performance of AP to achieve high energy generation at lower temperatures. As a result, researchers are becoming increasingly interested in the thermal behaviour and ignition of ammonium perchlorate. Because of its thermal properties, AP is extremely sensitive to the addition of trace amounts of additives [2].

Ammonium perchlorate is a remarkable oxidizing agent, it is compatible with various materials, including metal oxides, transition metals, alkali metals, and other polymeric binders. It serves as the predominant oxidizer in composite propellants, and many research investigations have been conducted to explain its thermal decomposition mechanism [3,4]. A low percentage (0.1 wt%-2 wt%), catalyst can offer a significant increase in propellant combustion performances [5]. Recent research has focused on exploring the compatibility of AP with additive materials to enhance the performance of solid propellants. Metal oxides (such as Fe_2O_3, CuO, Ni_2O_3, and others) and Transition metals (such as Fe, Ni, Mn, Co, Cu and etc) are extensively utilised as combustion catalysts. Superior catalytic activity was demonstrated by CuO, TiO_2, and $BaFe_{12}O_{19}$, which not only reduced the necessary energy for AP initial decomposition, but also combined the two following exothermic decomposition peaks into one, resulting in a significant increase in overall heat release [6-9]. The most efficient burning rate modifiers for composite propellants are observed to be transition metal oxides (TMOs) and their complexes [10]. This includes investigations with materials like strontium titanate, aluminium/iron and metal oxide nanoparticles such as CuZnO, CoZnO, and NiZnO [3,11,12]. In addition, Fe_2O_3 produced merely during propellant firing provided increased thermal conductivity, a large interfacial surface area, and has a high catalytic activity decrease the ignition temperature [13]. Moreover, the metal additives increase the solid propellant energy performance. Adding metal powder to composite propellants raises the heat of combustion value equivalent to AP based propellants [14].

Metal like magnesium (Mg) not only act as chlorine scavenger but also improve the solid propellant performance and work well with other materials. According to previous study, Al-Mg lowers the ignition temperature of the particles while increasing their reactivity [15]. The influence of magnesium-based compounds on the thermal decomposition of ammonium perchlorate was studied, and it was discovered that magnesium-based compounds reduce the thermal decomposition peak temperature while increasing the total released heat of decomposition. These compounds can boost the effect of the propellant's thermal breakdown [16]. The thermal decomposition properties of ammonium perchlorate, play a crucial role in measure performance of solid rocket propellant. In fact, the thermal decomposition performance of AP can directly influence the combustion behaviour of solid propellants [17,18]. The composition of the propellant influences its physical, mechanical, and ballistic qualities [19]. Furthermore, catalysts can clearly influence the decomposition performance of ammonium perchlorate.

The fundamental compromise from most experimental studies supports that the process of AP thermal decomposition involves proton transfer [20]. Previous thermogravimetric analysis (TGA)/ differential scanning calorimeter (DSC) findings indicated that the thermal decomposition of ammonium perchlorate is a two-stage process, comprising low-temperature thermal decomposition and high-temperature thermal decomposition. The low-temperature decomposition (LTD) stage takes place between 300 to 350 °C, while the high-temperature decomposition (HTD) stage occurs within the range of 350 to 450 °C. The mass loss ratio during the low-temperature stage is approximately 30%, and the solid residue after this stage remains AP compound. Additionally, an endothermic peak at 240 °C suggests a structural transition of AP from a cubic crystal type to an orthorhombic form [21].

Due to the intrinsic versatility of AP, researchers gain a significant advantage in their effort to enhance the quality and applicability of AP in the of solid propellants through the exploration of innovative materials. From the existing literatures, it is noted that researchers worldwide have not conducted a comprehensive study using sorbitol as a fuel mixed with ammonium perchlorate and magnesium as a solid propellant. Few studies have investigated the synergy of perchlorate metal with sorbitol in solid propellant formulations. This outcome is attributed to the convincing oxidizing properties of potassium perchlorate, boasting a high positive oxygen balance of 39% [22–25]. This adjustment in the oxygen balance of the mixture optimizes the availability of oxygen, ensuring a more thorough combustion process. The capacity to achieve a good balance between thermal stability, sensitivity, propellant density, oxygen balance (OB), and combustion/detonation parameters is the fundamental concern with energetic materials [26]. These investigations into propellant compositions are essential for understanding its effect on thermal decomposition as well as for evaluating the impact of their exothermic breakdown and ignition temperature on possible risks during handling, use, and storage. This information reveals how sensitive solid propellant compositions are to different accidental factors.

Studies on the characterisation of a combination of ammonium perchlorate with sorbitol and additional metal were carried out by Azizi *et al.,* [27], although there are still gaps in the data about thermal analysis. To the best of author knowledge there is no literature been published about thermal decomposition and combustion analysis of AP/sorbitol with magnesium metal. In this investigation, mixture of sorbitol and magnesium powders with AP oxidizer were formulated, and the impact of chemical composition on thermal decomposition, ignition temperature and heat of combustion release was examined through thermal analysis experiments and their heat of combustion subsequently was measured. These studies have produced recommendations for improving these propellant compositions stability and reducing their susceptibility to certain unintentional circumstances. Another objective of this work is to measure the effect of metals inclusion in solid propellant on Thermal behaviour, ignition temperature and heat of combustion.

2. Methodology

2.1 Materials

Five sample containing sorbitol as fuel and AP as oxidizing agent were developed. Two sample were prepared at difference amount of oxidizing agent. Additive metals such as magnesium and ferric oxide were mixed in 3 sample containing AP/sorbitol and compare to the AP /sorbitol alone. Table 1 provides an overview of the mass fractions of the components utilized in the preparation of the solid propellant based on Azizi *et al.,* [27] formulation. The percentage of the metal additive used in this formulation relative to the entire mixture of ammonium perchlorate and sorbitol.

Table 1
Mass fractions of the components used in minimum signature solid propellant

Sample Name	Formulation Wt. %	Metal Additive
SP4	AP 77% Sorbitol 23 %	-
SP5	AP 80% Sorbitol 20%	-
SP6	AP 77% Sorbitol 23 %	Mg 1% of total weight propellant
SP7	AP 77% Sorbitol 23 %	Ferric oxide 0.05% of total weight propellant
SP8	AP 77% Sorbitol 23 %	Mg 1%, Ferric oxide 0.05% of total weight propellant

2.2 Design of the Propellants

The AP/Sorbitol solid propellant impulse was determined using PROPEP 3.0 (Propellant evaluation program) software. PROPEP is a thermochemical software tool enabling users to assess the theoretical performance of solid rocket propellants. It proves especially valuable for assessing the feasibility of potential propellant formulations. Additionally, it enables the fast determination of optimal ingredient ratios to achieve desired performance based on theoretical considerations [28]. The amount of composition product and specific impulse were determined using PROPEP 3.0 at condition P_C/P_e =70/1 and T_o=298K in which P_c is chamber pressure and P_e is nozzle pressure [29]. Specific impulse determined from PROPEP was compared with the calculated energy according to thermochemistry Hess law and experimental results.

2.3 Preparation of the Ammonium Perchlorate/Sorbitol Propellants

This phase of the experimental work involves the creation of various formulations for a composite solid propellant with a total mass of 20 g. The formulations include ammonium perchlorate, Sorbitol, Magnesium, and Ferrous oxide. Magnesium metal is used as additive to increase combustion energy while ferrous oxide is used as catalyst and neutralize HCl gas.

Sorbitol needs to be melted first to prevent any incidents, given that its melting point is lower than that of Ammonium perchlorate. Sorbitol serves a dual role as both a fuel and a binder medium for dispersing oxidizers and additive metals. The ammonium perchlorate and metals additives are blended at a temperature range of 60°C to 80°C until a uniform mixture is achieved. The mixing process is a crucial step in the production of composite propellants, requiring sufficient duration to produce a homogeneous paste suitable for pouring. This process was conducted within a temperature range of 60°C to 80°C, involving the mixing of the prepared sorbitol with ammonium perchlorate, magnesium metal, and ferrous oxide. The addition takes place in small quantities in a Teflon beaker until a homogeneous mixture is achieved, requiring approximately 10 minutes for the process. The propellant cooling process was conducted gradually over the course of one day.

2.4 Evaluation of Energy Performance

Energy performance is assessed through the explosion heat (Qv), determined using an adiabatic calorimetric oxygen bomb under vacuum conditions. Each sample's Qv was measured three times, and the average results were reported. The IKA C 2000 oxygen bomb calorimeter was employed to measure the combustion heat value of the 0.5g sample using the resistance nichrome wire ignition method, and subsequently, the combustion heat ratio was calculated [30]. The experiment took place within an ambient temperature range of 20 to 30°C, and an oxygen pressure of 3.0 MPa was applied under nitrogen gas atmosphere [11,31].

2.5 Thermal Analysis

The influence of sorbitol, mg, and iron Oxide on AP thermal behaviour was analysed using differential scanning calorimetry (DSC) to assess alterations in endothermic and exothermic decomposition peaks, along with the total heat released during complete oxidizer decomposition. The DSC Q20 instrument by TA, USA was utilized for these experiments. The sample underwent heating from 30°C to 500°C at a rate of 5°C per minute under a continuous flow of nitrogen gas at 20 ml per minute. Thermogravimetric (TG) analysis was conducted using a Thermogravimetric Analyzer model Q500. Each sample, weighing approximately 10 mg, underwent heating under a nitrogen atmosphere, ranging from 30 to 500°C at a rate of 5°C per minute [5]. The DSC instrument was calibrated one month prior to the experiment using the procedure outlined in the manufacturer's technical guide. Various calibration phases were followed, including baseline, temperature, and enthalpy of reaction calibration.

3. Results

3.1 Heat of Combustion Analysis

The results of the experiments are illustrated in Table 2. A clear observation from Table 2 is that the heat of combustion is lower for the 80% AP-based propellants compared to the 77% AP-based propellants. The lower heat of combustion for SP5 AP-based propellants compared to SP4 AP-based propellant is attributed to the fact that excessive oxygen content can reduce the energy combustion same as reported in literature [32]. Furthermore, the specific impulse for SP5 is the lowest compared to others sample. From this analyse the oxygen balance value plays a critical role in determining the energy performance and specific impulse of solid rocket propellant.

The calorific value of the metalized propellant has shown a substantial higher compared to the baseline propellant. However, the heat of combustion for Mg alone, SP6 was higher than that for iron oxide in SP7. The iron oxide propellant exhibits a lower calorific value than the pure magnesium metal addition in samples, primarily due to the highly exothermic nature of the formation of two metal chloride and metal oxide (MgCl and MgO), contributing to high heat of combustion values. Additionally, iron oxide serves merely as a catalyst, it lowering the activation energy and providing an easier path for the reaction to take place. The interesting observation from Table 2 is the mixing of two magnesium metal and ferric oxide into the propellant increase heat of combustion significantly. From all the formulations propellant, it reveals that SP8 demonstrates the highest calorific value. This highlights the interaction between iron oxide, magnesium metal, and the AP/sorbitol propellant. It reveals that AP/sorbitol containing magnesium and ferric oxide showing a synergetic effect of high heat of combustion for the solid propellant.

The sample containing magnesium metal provide not only high heat combustion energy but improve the specific impulse of the solid propellant. Although the high energy combustion does not mean to produce high specific impulse but the effect of magnesium metal clearly has significant impact on performance increment. Based on the analysis presented in Table 2, it is evident that magnesium maintains its role as the primary contributor to both the energy performance and specific impulse of the propellant.

Table 2
Heat combustion and specific impulse of AP/Sorbitol with additive metal

Samples	Additive Metals	Heat of Combustion (KJ/mol)	Specific Impulse PROPEP (s)
SP4 (Base line)	-	-2134.47	181.26
SP5	-	-1979.55	177.45
SP6	Magnesium Metal	-4142.57	183.56
SP7	Ferric oxide	-2988.35	180.24
SP8	Magnesium + Ferric oxdide	-5344.88	182.71

Additionally, the presence of iron oxide as a catalyst slightly increases the energy released compared to the baseline SP4. This suggests that the catalyst does not have a substantial impact on combustion energy and specific impulse rocket performance.

3.2 Thermal Behavior of Propellant
3.2.1 Thermal properties of mixtures

The rate of thermal decomposition of any solid propellant substance is important because it affects both the material's ability to deliver thrust and its shelf life. The DSC and TGA analysis were utilised to investigate the thermal properties of the solid propellants. A decomposition temperature of >200 °C is typically required for solid propellant to be utilised in practical applications [33]. In this study, the thermal stability of the five samples AP/Sorbitol solid propellant with and without additives was evaluated using DSC and TGA. The result of the analysis at heating rate 5 °C/min for five samples illustrated in Figure 1 and Figure 2.

The DSC trace for thermal decomposition of pure AP reveals that the initial endothermic decomposition occurred at 242°C corresponding to the phase transition from orthorhombic to cubic and for the exothermic peak of pure ammonium perchlorate is happened at 431-479°C at high thermal decomposition (HTD) [29,34]. The inclusion of sorbitol with AP causes a shift in the thermal degradation pattern of the AP, as seen in Figure 2 the peak is shifted to high temperature compared to pure AP. Figure 2 display the DSC graph of AP/sorbitol with 3 different peaks expect for SP7. The endothermic peak is identified at 250 C°, which reflected the crystal structural change from orthorhombic to cubic. According to the thermal decomposition mechanism of sorbitol, its melting point is observed at 98.7°C, and its decomposition temperature ranges from 211 to 360 °C. In this study, the solid propellant consisting of AP and sorbitol exhibits an initial exothermic decomposition at 210°C for all samples. It increasing to 218°C with increasing weight of AP. This suggests that sorbitol interacts with AP in terms of the ionic lattice by enhancing movement, eventually leading to the mixture decomposing immediately after sorbitol decomposes [35]. Based on the data gathered from Figure 1, Figure 2 and Table 3, show that SP5 has a greater decomposition temperature than SP4.

Furthermore, result also demonstrates the effect of ferric oxide and Mg on thermal decomposition of AP/sorbitol. Samples containing ferric oxide lead high temperature decomposition to lower temperature decomposition for the propellants. The subsequent two exothermic decomposition peaks were merged into one single exothermic broad peak as illustrated in Figure 1 and Figure 2. It can be concluded that AP/sorbitol with ferric oxide have better catalytic properties to decrease the decomposition temperature of solid propellant [36]. Moreover, the energy performance of the AP/sorbitol propellant is not significantly affected by the amount of ferrite supplied. By referring Figure 2, result of sample containing Mg reveal that it amazingly catalysed the decomposition temperature process by lowering the thermal decomposition (LTD, HTD) and increasing the heat release valid the result mentioned in many previous literature [37-39]. However, the combination of magnesium and ferrite synergistically improves the energy release of the propellant and mass loss at lower temperature as illustrated in Figure 1, but there is not much change in terms of thermal decomposition temperature.

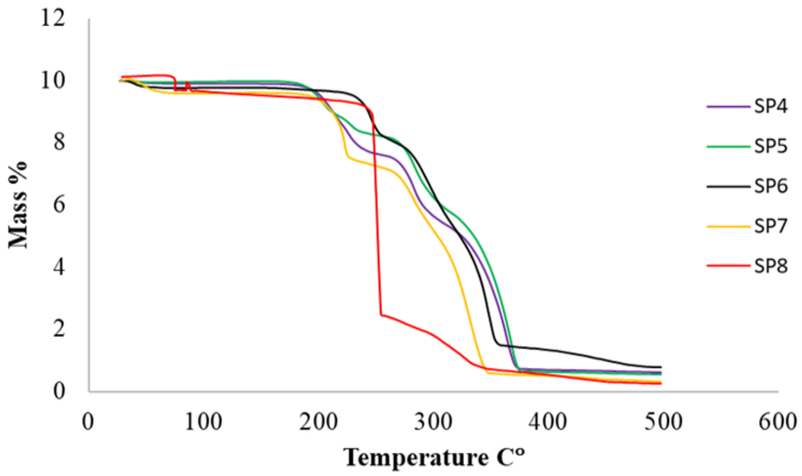


Fig. 1. TGA thermogram of Ammonium Perchlorate/Sorbitol with and without metal additives

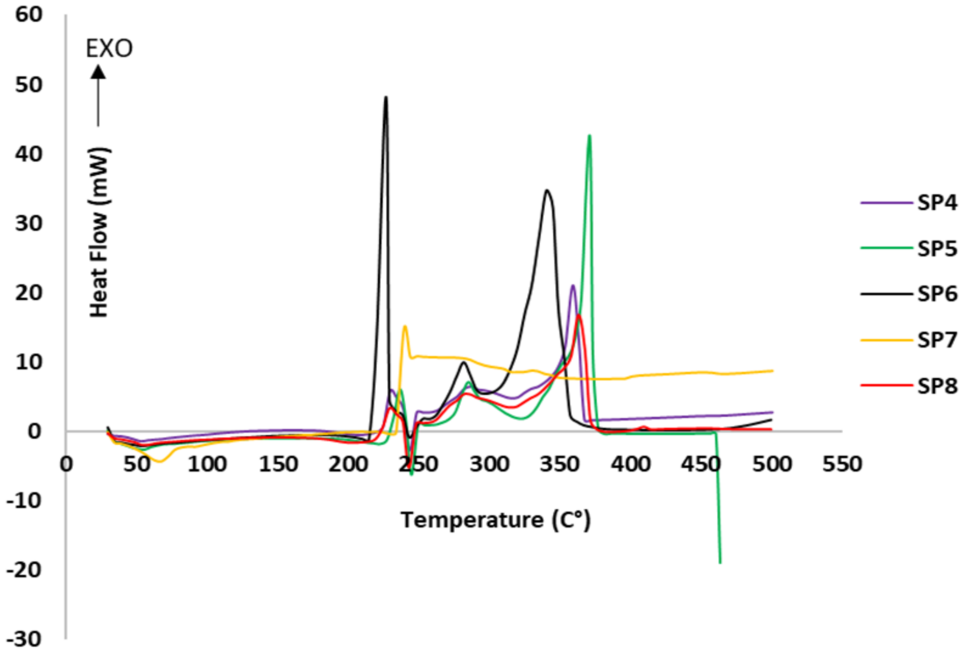


Fig. 2. DSC Thermogram of Ammonium Perchlorate/sorbitol with and without metal additives

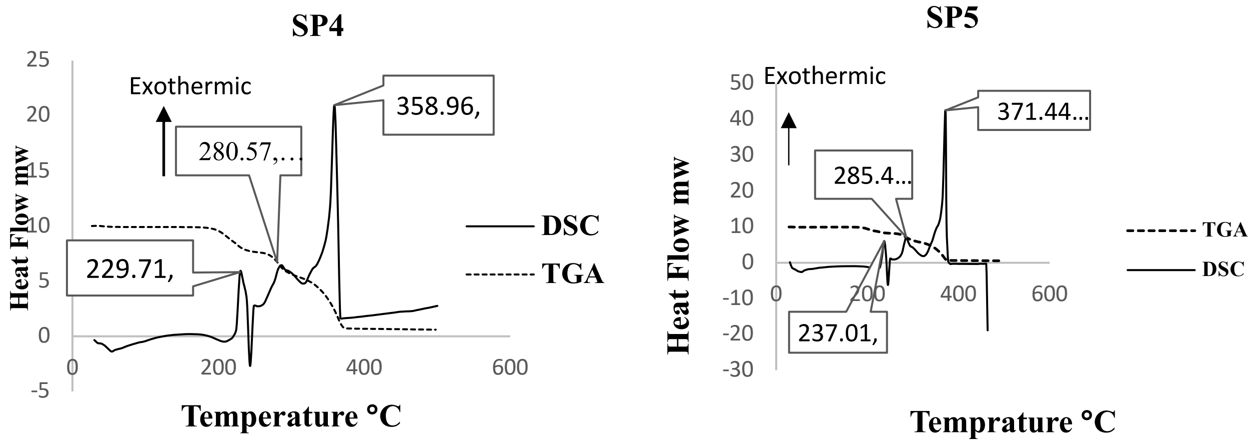
The weight loss in the TG thermograms presented in Figure 1 which has been combined with the DSC data revealed in Figure 3. In the comparative TGA analysis of mass loss, SP5 demonstrates a 15% mass breakdown, whereas SP4 has a 20% mass loss at the same temperature. This finding suggests that more sorbitol is decomposed, especially in SP4 because of its high sorbitol level. It implies that sorbitol has a synergistic impact on decreasing the thermal breakdown temperature of an ammonium perchlorate combination [40,41]. This difference can be attributed to the higher percentage of Ammonium perchlorate in SP5 which is effect of high oxygen balance. Figure 1 show all samples have 3 step weight loss except for the SP8. The first partial decomposition for all samples could be correlated to the initial exothermic decomposition peak in DSC at temperatures ranging from 210 to 230 °C. While adding ferric oxide and Mg in AP it demonstrated weight loss in two steps only. Figure 1 demonstrated that SP8 has higher mass loss 80% at initial stage decomposition temperature 243.34 °C compared to other samples. This decomposition stage could be correlated to the initial exothermic decomposition peak in DSC. The second main decomposition stage at 363.44 °C with wt % loss of 20 % this could be ascribed to complete dissociation of AP/sorbitol. The catalytic capability synergetic of ferric oxide and Mg on the quick oxidation of the AP gives the initial step decomposition at a lower temperature.

3.2.2 Comparison ignition temperature of mixtures

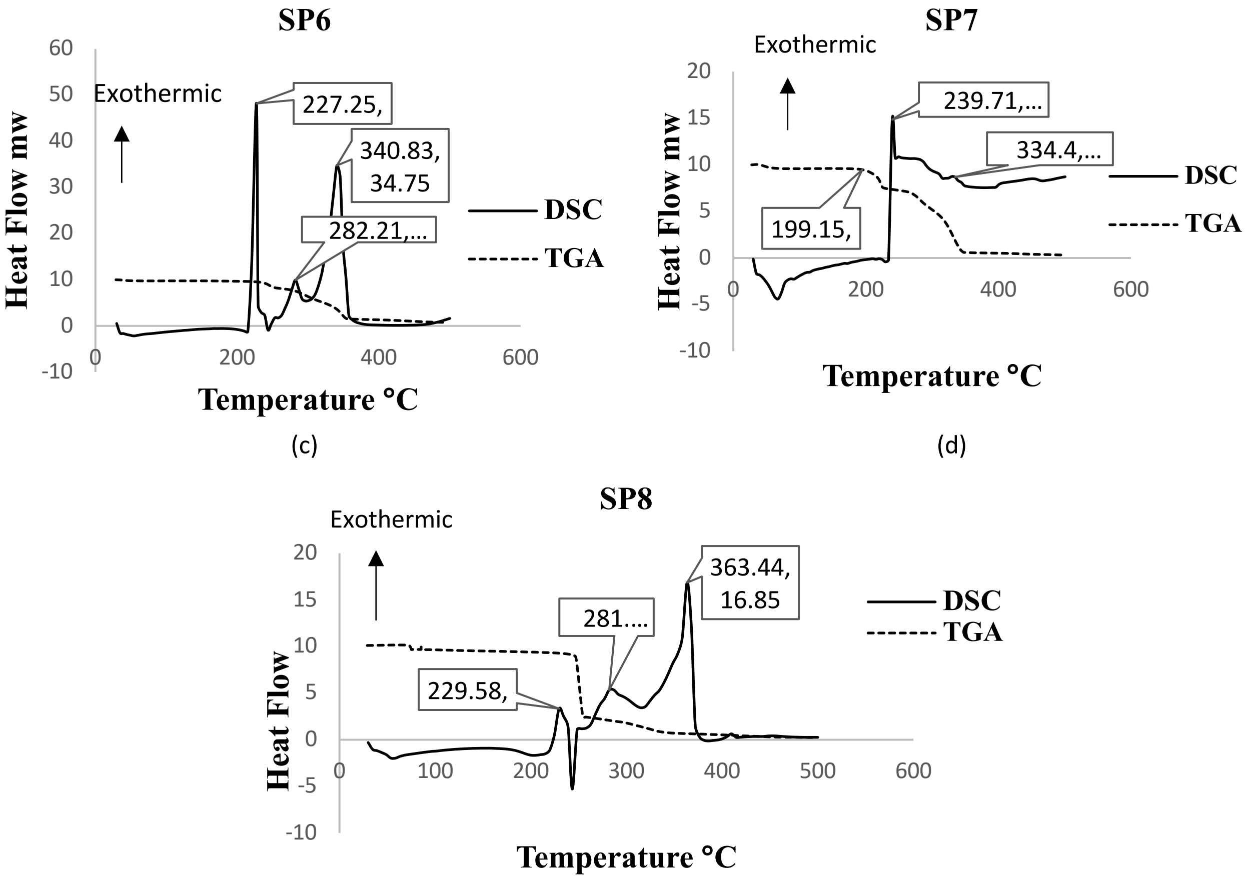
Since a specific feature of thermal decomposition of AP is extremely high sensitivity to the action of various additives, the additives may affect the deflagration delay time. It was assumed that there existed a correlation between the effect of the additives on the rate of thermal decomposition and their influence on the combustion rates of mix compositions incorporating ammonium perchlorate as the major component [20,42].

As observed in Figure 3, SP6 exhibits the lowest ignition temperature. A comparison between sample with additive to sample without additive reveals that the catalyst plays a crucial role in lowering the ignition temperature, thereby improving the burning rate and increase propellant sensitivity toward heat. For AP/sorbitol + Mg, melting occurs at 227.25 C° at same temperature with ignition temperature indicate that rapid decomposition happens immediately after the fuel melts [43]. Figure 3 illustrate the influence of Mg metal and ferric oxide addition on the thermal decomposition of the AP/Sorbitol mixture. As depicted in Figure 3, the introduction of magnesium metal to the mixture improves the ignition temperature to 229.58°C, above this temperature violence decomposition occur. Ignition temperature for SP7 is the lowest due to catalytic proficiency of ferric oxide. Therefore, catalytic ability effects the ignition temperature for AP based propellant tremendously.

Value of ignition temperature from Table 3 for each sample is lower than pure AP. Furthermore, high amount of AP slightly enhanced the ignition temperature. Thus, the decomposition reaction of Ammonium perchlorate is a key factor in the reaction mechanism [44]. This different thermal behaviour of AP/sorbitol and AP/sorbitol with additives is related to thermal stability of Ammonium perchlorate.



(a) (b)



(c)

(d)

(e)

Fig. 3. Combine plot of DSC & TGA thermogram Carried out at a Heating rate at 5 C°/min

Table 3
Summary DSC and TGA result of solid propellants

Samples	Transition temperature °C		
	Ignition Temperature °C	LTD °C	HTD °C
AP [45] (base line)	242	297.8	452.8
Sorbitol [40] (baseline)	211-360	-	-
SP4	229.71	280.57	358.96
SP5	237.01	285.47	371.44
SP6	227.25	282.2	340.83
SP7	199.15	239.71	334.4
SP8	229.58	281.93	363.44

^{a}LTD is lower temperature decomposition, ^{b}HTD is high temperature decomposition

4. Conclusions

The following conclusions can be derived from the investigations on the combustion characteristics of solid propellants based on AP as the oxidizer, sorbitol and Mg as the fuel binder, and ferric oxide as the catalyst

i. The investigation of the solid propellant of AP/Sorbitol reveals that an AP of 77 wt% weight percent represents the optimal condition in terms of energy release. It has been noticed that increasing the quantity of oxidising agent over this amount reduces the heat of combustion for solid propellants, reduce the solid propellant performance. On the other hand, high amount of AP increases the thermal stability and ignition temperature of solid propellant.

ii. The addition of Mg metal significantly improves both the energy release and performance of the AP/Sorbitol solid propellant, indicating that it does more than just operate as a chlorine scavenger. Furthermore, the thermal decomposition of the AP/sorbitol mix with Mg significantly improve its sensitivity toward heat.

iii. In contrast, ferric oxide, which acts as a catalyst, does not directly contribute to energy release or solid performance, but it does help to decrease the ignition and thermal decomposition temperatures, while it does not have effect on solid propellant performance. Finally, both Mg and ferrite additives work synergistically with AP/Sorbitol solid propellant to improve overall solid propellant performance and increase energy release.

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