DEVELOPMENT OF AMMONIUM PERCHLORATE BASED SOLID PROPELLANT

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This thesis is submitted as partial fulfillment of the requirements for the award of the Bachelor of Mechanical Engineering with Automotive Engineering

> Faculty of Mechanical Engineering UNIVERSITI MALAYSIA PAHANG

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I certify that the project entitled "Development Of Ammonium Perchlorate Based Solid Propellant" is written by Ahmad Hakimi Bin Abdul Nasir. I have examined the final copy of this report and in my opinion, it is fully adequate in terms of the language standard, and report formatting requirement for the award of the degree of Bachelor of Engineering. I herewith recommend that it be accepted in partial fulfillment of the requirements for the degree of Bachelor in Mechanical Engineering with Automotive Engineering.

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Especially to my beloved parent, Mr Hj. Abdul Nasir Bin Muhammad and Mdm Hjh. Sharifah Binti Hj Ismail, my beloved siblings Ahmad Nasharuddin, Ahmad Hafizi, Ahmad Shafiq and Anis Afina, and to all my friends....

Thank you for the support and motivation you guys until I can reach up to this level. Only Allah is the One able to give back to you guys.

Amin....

Life doesn't give yon what yon want.. It gives yon what yon deserve...

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ABSTRACT

There are a lot of development and study of ammonium perchlorate (AP) based solid propellant are already existing in the world. However, there are no detail data and information on the low burning rate AP based solid propellant. This thesis discusses the study of development AP based solid propellant. The objective of the study is to develop low burning rate AP based solid propellant including the selection of a propellant formulation, preparation and development of the propellant, and propellant burning rate experiments with the strand burner at atmospheric pressure. Together with the literature study and theoretical performance, five sets of different mixture were chosen based on their efficiency of propellant mixture. The propellant was a mixture of AP as an oxidizer, aluminum (Al) as fuel and hydroxy-terminated polybutadiene (HTPB) as a binder. For each mixture, HTPB was set at 15% and cured with isophorone diisocyanate (IPDI) (9.33% per weight of HTPB). By changing the AP and Al, the effect of oxidizer- fuel (O/F) ratio on the whole propellant can be determined. The lowest value of the testing obtained are propellant f80, that is 1.47mm/sec with O/F ratio 16.0. The study found, increased ratio O/F will affect to the combustion rate reduction factors of propellant.

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LIST OF SYMBOL

AP	Ammounium Perchlorate
НТРВ	Hydroxyl terminated polybutadiene
Al	Aluminum powder
IPDI	Isophorone Diisocyanate
r	Burning rate
a	Empirical constant
Р	Pressure
n	Pressure exponent
=	Equal
kg	Kilogram
m ³	Meter cubic
⁰ C	Degree Celsius
%	Percent
ТМО	Transition metal oxide
Fe2O3	Iron oxide
CuO	Copper oxide
TGA	Thermogravimetric analysis
DSC	Differential scanning calorimetry
DTA	Differential thermal analysis

CHAPTER 1

INTRODUCTION

1.1 Introduction

Ammonium Perchlorate (AP) is a mixture of materials that have long been used in the production of ballistic missiles, military attack missiles, space applications, and others application. It is known as the powerful oxidizer for the solid propellant. There are two main advantages of Ammonium Perchlorate (AP), stability rocket or bullet resulting in safe and the ability to control the rate of combustion stability in the propellant. [1]

As we know, the study of solid propellant and on Ammonium Perchlorate has done much in this world and has substantial data collected and analyzed. Despite this, there is still no complete and detailed data on Ammonium Perchlorate base on Solid Propellant. This report is a study of Ammonium Perchlorate base on Solid Propellant that covers and include a mixture of methods propellant fuels, burning rate test and the method of preparing and producing solid propellant.

Knowing the limited data and technique used is not enough since other parameters such as the size and type of device used to generate the baseline data is not fully taken into account, then the data cannot be correctly interpreted and errors due to the scale up may result. The this project, this solid propellant use Ammonium Perchlorate (AP) acting as the oxidant, Aluminum (Al) as fuel and Hydroxy-terminated Polybutadiene (HTPB) as binder/fuel. For the Hydroxy-terminated Polybutadiene (HTPB), there is another mixture contained in the Hydroxy-terminated Polybutadiene (HTPB), namely Isophorone Diisocyanate (IPDI). Isophorone Diisocyanate (IPDI) was calculated based on the percentage of weight of Hydroxy-terminated Polybutadiene (HTPB).

1.2 Objective Study

• To develop low burning rate Ammonium Perchlorate (AP) based solid propellant.

1.3 Research Methodology

The study began with a request for information from literature, journals and books that involve the development of Ammonium Perchlorate based solid propellant. In examining the ways mentioned, the most effective and a lot of help is by referring supervisor and lecturer who has been involved and has had experience in the development of this Solid Propellant based on Ammonium Perchlorate.

After the strand burner test, another measure of the burning rate will be obtained, and the data obtained will facilitate and expedite the process of combustion in the test of propellant later. Lastly, the basic ingredients that commonly used in solid propellants were reviewed.

1.4 Project Methodology

After referring journals and books related, for the first step, the estimated mixture of fuel, oxidizer and binder tested and burned using the easiest and simple method. With a straw, the three types of mixture of materials included in the straw and burnt at open air or normal condition. Each percentage in the test mixture is burnt several times and the average of data will be measured and calculated. For each burning test, the results will be measured burning rate with related tools.

All data obtained from the burning rate test will be collected for the averaging to know the where of mixture percentage is the most efficient.

After getting the data and best results, mix will be tested by applying pressure on the mixture. Given pressure is through by the strand burner (crowford bomb). The purpose of this mixture pressurized is to test the burning rate and to see what reaction this mixture in a high pressure. This is because the mixture should be able to survive and be able to produce a high burning rate in high pressure in the rocket motor.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter describes the details of the study of solid rocket propellant. The information is intended is concerned with the history of solid rocket propellant, ingredients and properties of the propellant, what the function of binder, metal fuel, oxidizer, solid propellant burning rate and burning rate test Equations. With the availability of this literature will surely facilitate the process of conducting experiments burning rate test because all the information is disclosed.

2.2 History Of Solid Propellant Rocket

The history of solid rockets in Japan started from which the Imperial Japanese Navy developed the Funryu (raging dragon) series missiles at its Dai-Ichi Kaigun Koku Gijutsusho (1st Naval Air Technical Arsenal) at Yokosuka, its principal research and development center. Since then, several programs of missile development were carried out through 1945, the end of World War II, in which solid rocket motors were used as propulsion systems. After the blank period of two years, namely in 1947, solid propellant rocket motors were beginning to study again. Research and development of solid rocket motors were directed in two ways, space use and defense use. In space development, many types of solid rocket motors were studied and developed from the successful launch of Pencil rocket in 1955 to the solid rocket motors of H- II rocket and M-V rocket under development.

On the other hand, in defense development, many types of solid rocket motors were also studied and developed from the initiation of 70 mm diameter air to air rocket development to solid rocket motors of various types of rockets and missiles under new development.

Until now, the researches on the history of solid propellant development in Japan were carried out as follows; the paper on the solid rockets in Japan, the papers on the history and status of solid rockets for space use in Japan, the paper on the history of solid propellants for space use in Japan, the papers on the history of solid propellant in Japan, and the paper on the status of solid propellants in Japan. [1]

2.3 Solid Rocket Propellant

Much work has been devoted in various countries to investigating the combustion mechanisms of solid propellants. It is timely to bring together the information obtained by the authors and compared to that of the literature on the combustion of the individual components as well as of their combination into propellants.

The viewpoint adopted here is that of the understanding of the combustion behavior of propellants. Therefore as much information as possible is presented about the fundamentals of the processes (thermal properties, kinetics in the condensed phase and in the gas phase), whereas no attempt is made to establish a complete catalog of practical results on various propellants with different particle sizes, catalysts, variations on the percentage of ingredients. The aim is to give a clear, as conclusive as possible picture. This will then be non compatible with a complete discussion of the various, sometimes contradictory, mechanisms proposed in the literature. Also precluding such a discussion is the will to compare the different components and the corresponding propellants. [2] There were two types of solid propellants which are homogeneous and heterogeneous propellant. In homogeneous propellant, three main ingredients were combined together with an ionic combination. A common example for homogeneous propellant is a combination of nitroglycerin (NG) and nitrocellulose (NC). Both NC and NG are single base propellant which itself has fuel and oxidizer and could self burning and produce hot gaseous if thermally decomposed. In heterogeneous propellant or sometimes called composite propellant, the basic ingredients are a crystalline oxidizer, metallic fuel and polymeric binder. The term 'composite' states that, the propellant is made at least two different kinds of substances in a heterogeneous mixture, without any chemical bond. [3]

The composite propellants also contain additives to enhance the mechanical strength and alter the burning rate. [4]

Some space is taken up by physical-chemical modeling. The aim is not so much to give the elements of mathematical descriptions which could be used in a priori computation of the burning characteristics of propellants (to the extent that such computations are possible). The point is more to put to test the hypotheses made on the mechanisms of combustion by incorporating them in reasonable models and confronting the results thus obtained with experimental data.

These descriptions can also be viewed, alongside with the data given for each component or propellant, as useful for mastering the regimes of combustion which go beyond stationary combustion, that is erosive burning and unsteady (under pressure excursions or pressure oscillations) combustion responses.

Double-base propellants (made by the extrusion or powder casting techniques) are used in anti-tank rockets or missiles and in some tactical missiles. Their main advantage is that they produce a minimum amount of smoke (only for a small amount of additives).

Composite propellants, based on ammonium perchlorate (AP) without aluminum, generate reduced smoke, HCl and H2O vapor will precipitate into droplets in

the plume under given temperature and humidity conditions. They are used for various tactical missiles. With aluminum, they are widely used in missiles and space launchers. They produce alumina smoke, which, in the case of space launchers, could be considered in the future to be undesirable (along with HCl).

Composite propellants based on nitramines and an "active" binder (cross linked polymer with nitroglycerin or other liquid nitrate esters) are used more and more. Without aluminum, they are in the minimum smoke category and they replace DB propellants. With aluminum, they reach the highest specific impulse and density and are used so far for upper stages of strategic missiles. [2]

2.4 **Propellant Ingredients And Properties**

In a real situation, there are very large numbers of parameters involved in the manufacture of solid propellants including the ingredients and the step of manufacturing. Table 2.1 shows that there would be at least ten different ingredients used in producing solid propellant. While, Table 2.2 shows the common ingredient used in composite solid propellants. Table 2.4.1 shows example ingredients properties of solid propellant.

Ingredient	Function	Percentages
Ammonium Perchlorate	Oxidizer	73.00
HTPB	Binder	14.95
Oxamine	Burn rate modifier	5.00
Iron Oxide	Burn rate modifier	2.00
Dioctyl Azelate	Plasticizer	2.00
IPDI	Curing agent	1.40
Zirconium carbide	Ballistic modifier	1.00
Tepanol	Binding agent	0.30
Agerite white	Anti oxidant	0.15
Triphenyl bismuth	Cure modifier	0.10
Magnesium oxide	Cure modifier	0.10
Total		100

Table 2.1Propellant ingredients used by Martin S.M. and Hughes E.H.[5]

Curing Agent				
AP	Al	HTPD	IPDI	References
74.5	-	18.9	1.93	[6]
70.0	18	8.935	0.615	[7]
80.0	-	20.0	1.60	[8]
72.5	15	9.47	0.74	[9]
80.0	-	14.67	1.15	[10]
87.5	-	9.47	0.74	[11]
68.0	18	13.2	0.80	[12]
86.0	-	8.8	-	[13]

21.6

10.085

11.0

10.56

10.6

14.67

-

-

-

-

_

1.15

[14]

[15]

[16]

[17]

[18]

[19]

Table2.2Example of the major ingredients in AP based solid propellants

2.5 Binder

73.0

68.0

66.0

67.3

67.5

80.0

_

18

16

20

20

-

The most important ingredient in solid propellant is a binder. Binder provides the structural glue or matrix in which solid loading are held. The common binder used in solid propellant is elastomeric binders. These binders included polyesters, polysulfide, polybutadiene acrylonitrile (PBAN), carboxyl terminated polybutadiene (CTPB) and Hydroxyl terminated polybutadiene (HTPB).

Composite solid propellants mainly contain a polymeric fuel binder, a metallic fuel such as aluminium (*Al*) powder and an oxidizer usually ammonium perchlorate (AP). The polymeric binder, which constitutes 15-25 wt per cent of the propellant generally consists of a telechelic liquid prepolymer, curing agent, plastisizer, ballistic modifier, bonding agent, and an antioxidant.[20]

Binders provide the structural glue or matrix in which solid granular ingredients are held together in a composite propellant. The raw materials are liquid prepolymers or monomers. The binder impacts the mechanical and chemical properties, propellant processing, and aging of the propellant. Binder materials typically act as a fuel, which gets oxidized in the combustion processes. Commonly used binders are HTPB, CTPB, and NC. Sometimes GAP is also used as energetic binder, which increases the energy density and performance of the propellant. HTPB has been abundantly used in the recent years, as it allows higher solid fractions (total 88–90% of AP and Al) and relatively good physical properties.[23] Composite propellants based on hydroxyl terminated polybutadiene (HTPB) resin are the most widely used solid propellants for launch vehicle and missile applications. [20]

Hydroxyl terminated polybutadiene (HTPB) liquid prepolymers find extensive application as binders in composite solid propellants for launch vehicle technology. The composite solid propellant comprises about 12.20% of polymeric fuel binder, a metal additive such as aluminium powder and an oxidizer, usually ammonium perchlorate. The binder in the solid propellant imparts dimensional stability and structural integrity to the grain and also acts as a fuel during combustion. The mechanical properties of the propellant are largely determined by the extent of polyurethane formation. Hence a knowledge of the kinetics of polyurethane formation will be very useful in the design of propellants, liners, etc., possessing mechanical properties needed for specific purposes.

Chemical name	Hydroxyl Terminated Polybutadiene (HTPB)	
Chemical Formula	C _{7.075} H _{10.65} O _{0.223} N _{0.063} (approximate)	
Appearance	Yellowish liquid	
CAS No.	69102-90-5	
Molecular Weight	NA	
Density	989 kg/m ³ [conducted manually]	
Boiling Point	300 ⁰ C	
Melting Point	NA	
Solubility	Negligible	
Stability	Stable under ordinary conditions of use and storage	
Conditions to Avoid	Oxidizing condition and extreme temperature	

Table 2.3HTPB as a polymeric binder [21,22]

2.6 Metal Fuel

The aluminium powder, from millimetric flakes to nanoparticles, is often used in various applications. A direct link exists between its initial oxidation state and its combustion properties, such as the Minimum Ignition Energy (MIE) and the flame velocity. The nanoscale is often related to propulsion applications and pyrotechnic compounds because of the high burning rate of such small particles due to their high specific contact surface. The nanometric powder also presents the advantage to reduce the waste after combustion [24].

Several facets of this application have been reported, such as production, thermal decomposition in combination with ammonium perchlorate (AP), surface coating of aluminium particles, ignition and oxidation or combustion of aluminium particles including bimodal blends of micrometer and sub-micrometer sized particles, combustion of pressed pellets of aluminium and AP with additives, aluminized composite propellants, and collection of aluminium agglomerates formed during the combustion of aluminized propellants [25].

On the other site, aluminum powder is a high concentration in the earth's crust and relatively high calorific value another perspective energy storage. Unlike hydrogen, it is transportable and easy-to-storage. Aluminum can be used for both energy and hydrogen production. Its oxidation products (aluminum oxide or hydroxide) can be returned to the cycle of aluminum reproduction or used as a vendible product for ceramics, adsorbent, catalyst and other.

One of the main selection criteria for aluminum based energy technology is aluminum oxidation kinetics. Oxidation rate and conversion degree first of all depend on the surface area of initial reagents, the reaction temperature and the type of oxidant [26].

It has been mentioned that depending on the alumina thickness of Al particles, it could be possible to control the explosibility, and so the risk of this powder. The present bench (with the described optimized method for oxidizing the Al powder) will be used in order to provide the required samples with oxide content that are missing. If a given level of safety is wanted for an industrial configuration, this figure can be used to estimate the required oxide content of the Al powder. [24]

2.7 Oxidizer

Properties of ammonium perchlorate in general and particularly its thermal decomposition have been a subject of extensive literature including reviews.

It was revealed during investigation of thermal decomposition of ammonium perchlorate that the process is characterized by a number of features which were purely scientific interest irrespective of applied problems at which these investigations had been aimed at first [27].

Ammonium perchlorate (AP) is one of the main oxidizing agents that have been used in various propellants. The burning behavior of the propellants is highly relevant to the thermal decomposition of AP. Cupric and ferric oxides are proving to be quite effective on thermal decomposition of AP. Several methods have been employed to prepare MOs, such as, solid state reaction, homogeneous precipitation, soil–gel method, citric acid complexion approach and auto-combustion [28].

It is well established that many transition metal oxide (TMO) catalysts including iron oxide (Fe2O3) and copper oxide (CuO) accelerate the decomposition of pure AP. Jacobs and Whitehead summarize the work done to characterize the decomposition of pure AP in the presence of TMO catalyst using various techniques such as thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), differential thermal analysis (DTA), and mass spectroscopy. In general, it has been shown that both Fe2O3 and CuO lower the ignition temperature of AP, lower the low-pressure deflagration limit, and accelerate decomposition through all temperature regimes. Studies have also investigated the catalytic effect of polymeric binders used in propellants [29].

2.8 Strand Burner

For about 60 years, the industry standard apparatus for routine measurements of linear burning rates has been the so-called strand burner or Crawford bomb proposed by 8 Crawford in 1947. This method, very quick, simple, and economic, is particularly suitable for exploring new propellant compositions, characterizing a propellant's burning rate over a defined pressure and temperature range, or performing quality control of established compositions.

The propellant sample being tested, referred to as a *strand*, is burned within the confines of a pressure tank pressurized with an inert gas. The strand is in the form of a pencil-like stick, and is ignited at one end. The time duration for the strand to burn along its length in a cigarette fashion is measured.

The two basic approaches to economics, experimental characterization of a solid propellant's burning rate are closed and isobaric strand burners. The closed burner technique characterizes the isothermal burning rate function in a continuous manner over a small pressure range with a single burn while the isobaric burner method provides a discrete measurement requiring several burns. Over the years, three major advanced techniques to improve the accuracy of the measurement of the regression rate of strands have been implemented and characterized.

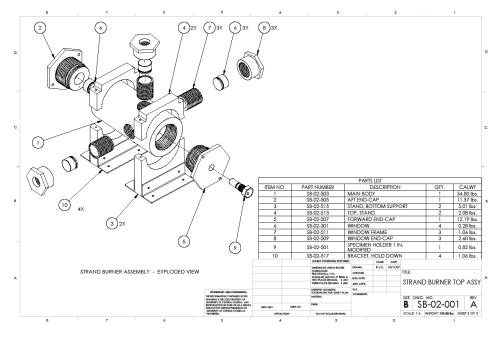


Figure 2.1 Example of Strand Burner [30]

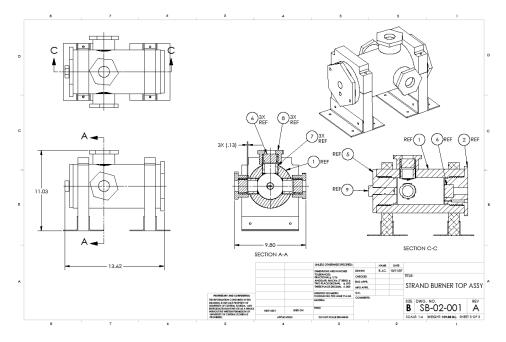


Figure 2.2 Example of Strand Burner [30]

Hermance presented in 1969 a method that consists of using the strand as the dielectric material of a capacitor which forms a part of a resonant inductor-capacitor circuit oscillating at a predetermined center frequency. Bozic et al. Presented the principle of the measurement and data reduction for their method using microwave reflection interferometry in 1995.

Lately, high accuracy internal ballistic measurement has been performed using ultrasonic instrumentation. Actually, the experiment is designed in a fashion that places efficiency and safety at the highest priority [30].

2.9 Solid Propellant Burning Rate Test And Burning Rate Equation

At any instant the burning rate governs the burning time and the mass flow rate of hot gas generated and flowing from the motor combustion chamber to the nozzle and therefore the thrust, and the specific impulse, of the rocket.

The empirical relation relating the burning rate, r, and the combustion chamber pressure, P, is

$$r = a P^n$$

Where a is a dimensionless empirical constant influenced by ambient grain temperature (the *temperature coefficient*) and n is the *burning rate exponent* also called the *combustion index*. The later is independent of the initial grain temperature and describes the influence of chamber pressure on the burning rate. For stable operation, n has values greater than 0 and less than 1.0. High values of n give a rapid change of burning rate with pressure and can be determined for the motor.

Measuring rocket propellant burning rates cover various phases (research and technology, screening, development, performance verification, and production control) and each requires suitable tools. Correspondingly, a variety of experimental rigs and procedures is in use worldwide, ranging from the simple strand burners to an array of closed or vented vessels, from different small-scale (or subscale) test motors (ballistic evaluation motors) up to full-scale motors tested first on the ground and eventually in flight conditions [30].

CHAPTER 3

METHODOLOGY

3.1 Introduction

Chapter 3 will discuss the five main factors that influence the selection, preparation and methods of the project. The first step is to select propellant formulation. When getting an accurate formulation, the next step is fabricated propellant strand. With the availability of propellant strand, this will make it easier for preliminary testing at atmospheric condition and burning rate test at pressurize condition. Finally, with the collected data, we can analysis the data easier.

3.2 Selected Propellant Formulation

The selection of propellant type is at the core of any solid rocket motor design. The desirable characteristics of a solid propellant are high specific impulse, predictable and reproducible burning rate and ignition characteristics, high density, ease of manufacturing, low cost, and good ageing characteristics. Propellants should produce low smoke exhaust and not be prone to combustion instability. In addition, they should have adequate thermophysical and mechanical properties over the intended range of operating and storage conditions. At about this same time, interest also increased in using solid propellants in applications other than missile propulsion. One of the first major uses was jet assisted take off (JATO) as an aid in launching heavily loaded jet bombers [31]. This is Typical Propellant Formulation.

CASTABLE PROPELLANT		
Ingredient	Weight Per Cent (%)	
Liquid Polymer	10.8	
Plasticizer	3.0	
Curative	1.0	
Metal Powder	16.0	
Oxidizer	68.0	
Catalyst	1.0	
Antioxidant	0.2	
	100.0	

Table 3.1Typical Propellant Formulation [31]

Table 3.2Typical Propellant Formulation. [31]

EXTRUDABLE PROPELLANT		
Ingredient	Weight Per Cent (%)	
Rubber Polymer	12.0	
Filler	2.5	
Plasticizer	2.5	
Curative	0.5	
Antioxidant	0.4	
Oxidizer	80.0	
Catalyst	2.1	
	100.0	

But for this 'Development Ammonium Perchlorate Base on Solid Propellant' project, the burning rate test will be undertaken over the formulation below. We just focus on this percentage.

AP	Al	HTPD	IPDI
74.5	-	18.9	1.93
70.0	18	8.935	0.615
80.0	-	20.0	1.60
72.5	15	9.47	0.74
80.0	-	14.67	1.15
87.5	-	9.47	0.74
68.0	18	13.2	0.80
86.0	-	8.8	-
73.0	-	21.6	-
68.0	18	10.085	-
66.0	16	11.0	-
67.3	20	10.56	-
67.5	20	10.6	-
80.0	-	14.67	1.15

Table 3.3Propellant Ingredient (%) [6-19]

3.2.1 Ammonium Perchlorate (AP)

Ammonium perchlorate (AP) is one of the important oxidants, actually was being used in many types of propellants. Considering its limitation in loading in the rockets, it is important to improve the decomposition efficiency of AP to get the good requirements of high energy generation at low burning temperature [32]. Thermal decomposition of ammonium perchlorate content in general and in particular has been the subject of extensive literature including reviews [33]. In a commercial process, AP is produced from sodium perchlorate, gaseous ammonia and hydrochloric acid. Sodium chloride is also formed in this metathesis reaction. Ammonium perchlorate crystals are generally produced from the reaction of residual crystallinity fractions on a large scale. However, no detailed information in the literature regarding the crystallization kinetics of ammonium perchlorate. Kinetic data on the growth and dissolution rates are very important in the design and control of production in the AP crystals in a certain size [34].

Opinion from Makoto Kohga and Hirotatsu Tsuzuki [35], ammonium perchlorate based solid propellants prepared with good and higher ammonium perchlorate contents are required to obtain a high burning rate.

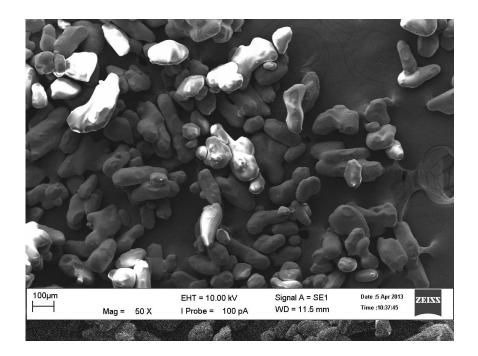


Figure 3.1 Sample Of AP in Scanning Electron Microscope (SEM) 50X

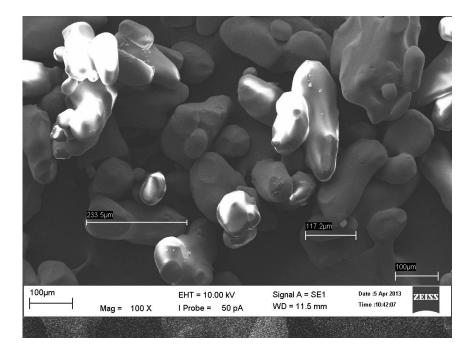


Figure 3.2 Sample Of AP in Scanning Electron Microscope (SEM) 100X



Figure 3.3 Sample Of AP

3.2.2 Aluminum Powder (Al)

The aluminium powder, from millimetric flakes to nanoparticles, actually was being used in various applications [35], widely used as high-energy fuel in areas such as propellants [36]. In modern solid rocket propellants for aerospace applications, it was commonly contained aluminum powder as a fuel, because of it have high energy release in the oxidation process to alumina [37]. But agglomeration of Al particles in the propellant surface layer also leads to incomplete burning of Al particles and promotes slag formation. All these can effects for result in the limited realization of the energy potential of the fuel [35].

However, including Al in fuel compositions is accompanied by a number of undesired features. One of the critical problem is the ignition of Al particles. Due to the protective oxide (alumina) layer on the particle surface, Al typically ignites in oxidizing atmosphere only after heating up to temperatures close to the melting point of alumina at 2300 K [36].

In fact, aluminum powder is a very reactive metal and its oxidation can occur either in thermite or in dust explosions. Because of their severity, aluminum dust explosions are often mentioned in accident reports as "fatal" or "devastating" [38].

Myers [38] proposed a method for reducing aluminum dust explosion hazards in aluminum buffing operations by addition of flame retardant. Testing showed that even the finest fraction of the residue in its dried state has ignition sensitivity and explosion severity parameters significantly decreased.

From Gascoin et al. experiment, investigated the influence of the oxidation rate of seven treatment parameters and provided a controlled method to furnish reproducible homogeneous set of aluminum powders for safety utilization [38].

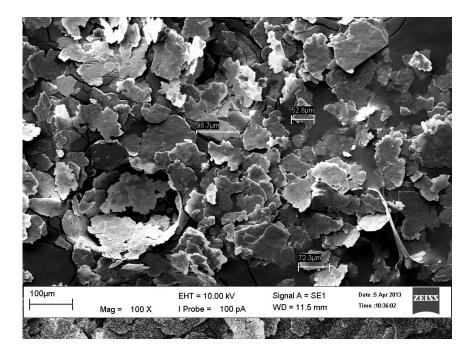


Figure 3.4 Sample Of Al in Scanning Electron Microscope (SEM) 100X

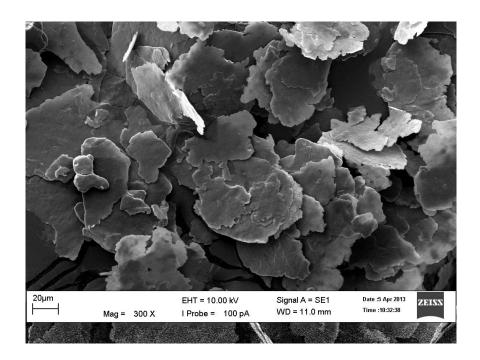


Figure 3.5 Sample Of Al in Scanning Electron Microscope (SEM) 300X



Figure 3.6 Sample Of Al

3.2.3 Hydroxyl Terminated Polybutadiene (HTPB)

Hydroxyl terminated polybutadiene (HTPB) is liquid prepolymers find extensive application as binders in composite solid propellants for launch vehicle technology, for rockets and for missile applications [41]. This liquid prepolymer has very good physical properties such as low glass transition temperature, high tensile and tear strength, and good chemical resistance [39]. It is physically and chemically compatible with the conventional oxidizers and other ingredients at normal storage conditions. As it contains mostly carbon and hydrogen, during combustion, it is decomposed to give large volume of stable molecules like carbon dioxide, carbon monoxide, and water vapors increasing the specific impulse of the rocket motor [39].

Nowadays, it becomes the best binder because of its processability and higher energy content compared to other binders [42]. The flow behavior of HTPB-based propellants assumed have great importance because the propellant has many grain defects in the large scale motors. Though several projects have studied the rheological behavior of composite propellants but it's still not very clearly understood. Several parameters such as the raw materials, formulation, vacuum level, and casting rate influences significant role in the flow behavior of the propellants. To make a logical decision regarding propellant mixing and casting, the effect of temperature and time on viscosity and pseudoplasticity are considered to be essential in addition to the parameters such as volume of propellant slurry, bowl and casting pipe dimensions [40].



Figure 3.7 Hydroxyl terminated polybutadiene (HTPB)

3.2.4 Isophorone Diisocyanate (IPDI)

Isophorone diisocyanate (IPDI) is an organic compound in the class known as isocyanates and for specifically, it is an aliphatic diisocyanate. It is produced in relatively small quantities, accounting for (with hexamethylene diisocyanate) only 3.4% of the global diisocyanate market in the year 2000. Aliphatic diisocyanates are used, not in the production of polyurethane foam, but in special applications, such as enamel coatings which are resistant to abrasion and degradation from ultraviolet light. These properties are particularly desirable in, for instance, the exterior paint applied to aircraft.

From Chemical site, IPDI exists in two stereoisormers, cis and trans. Their reactivities are similar. Each stereoisomer is an unsymmetrical molecule, and thus has isocyanate groups with different reactivities. The secondary isocyanate group is more reactive than the primary isocyanate group [43].



Figure 3.8 Isophorone diisocyanate

3.2.5 Selected Formulation Mixtures

For the first step in fabricating the strands is the preparations of the ingredients. There have four main ingredients used in this study, this is AP,Al,HTPB and IPDI. When the ingredients are ready, the first process starts with the preparation of the binder, this is done by consider and after that mixing the HTPB binder with IPDI according to the specific proportion. The ingredients were mixed together by using a stainless steel rod and it must mixed until the mixture becomes well and homogeneous.

The next step is adding the ammonium perchlorate (AP) and Al powder to the binder and stirred together until all the Al and AP was coated with the binder and curing agent. It is essential to make sure that all Al and AP are well coated to reduce the risk of combustion when the oxidizer is added later and make sure the accurate and good burning rate.

Number	Formulation	Ammonium	Aluminum	HTPB	IPDI
	Code	Perchlorate (AP)	Powder (Al)		
1	f60	60	25	15	9.33
2	f65	65	20	15	9.33
3	f70	70	15	15	9.33
4	f75	75	10	15	9.33
5	f80	80	5	15	9.33

Table 3.4Formulation Percentage % (Based on 100g)

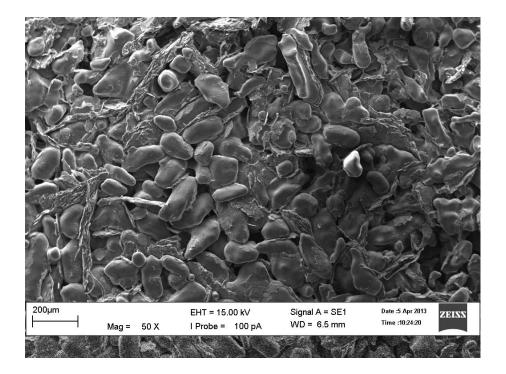


Figure 3.9Sample Of Propellant f70 in SEM 50X

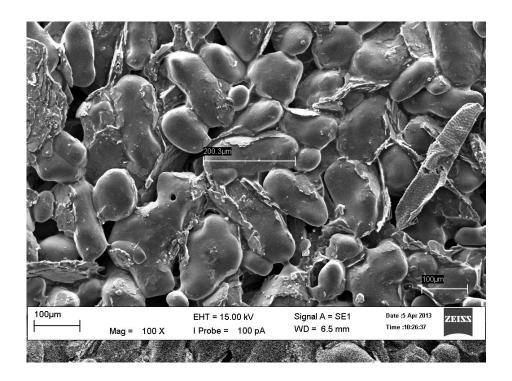


Figure 3.10Sample Of Propellant f70 in SEM 100X

3.3 Design Of Strand Burner

At the core of the burner facility are the two high-pressure bombs. The original strand burner, was designed and built by Space Launch Corporation (SLC) to handle test pressures in excess of 360 atm (5300 psi). The low-carbon steel alloy body offers one side window along the strand and one end window opposite to the strand.

A detailed procedure was established for preparing and burning the propellant samples. This procedure was refined during the course of the setup and early investigations to improve the quality of the data collection and to establish a quicker turn around between samples while increasing the safety of the operations. The length of the strand was chosen to be around 2.54 cm (1 in) for several reasons.

The first reason was to keep the pressure and temperature increase to a minimum since the vessel pressure and grain temperature directly affect the burning rate. Depending on the mixture, the combustion of a 1-in strand increases the pressure inside the bomb from 5 to 20%. This pressure variation was demonstrated in subsequent experiments to have a minor influence on the burning rate of the tested sample. Howbeit the internal volume of the new strand burner was increased by 13% to further reduce the pressure variation during combustion without altering its distinct onsets and ends. Moreover, each burning time is related to the pressure average between the ignition pressure and the pressure at extinction.

Secondly, the 1-in strand minimizes the re-circulating flow field generated by the inhibited, end-burning strand in a closed vessel as described and modeled by Glick and Haun. Depending on the composition of the strand, burning a longer strand may generate enough smoke to hinder the proper acquisition of the light emitted by the burning surface.

Finally, the strand size selected reduces the material cost, handling and storage of hazardous material, and data storage of each experiment while maintaining adequate resolution and signal-to-noise ratio of the data acquisition [31].

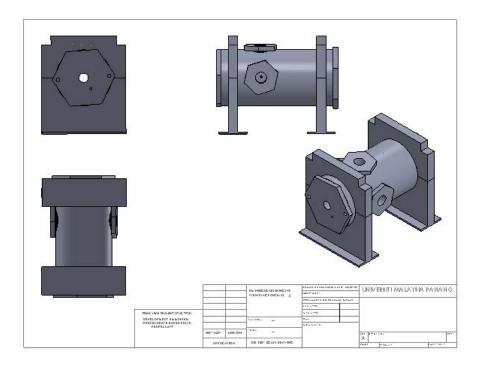


Figure 3.11Assembly Of Strand Burner main body

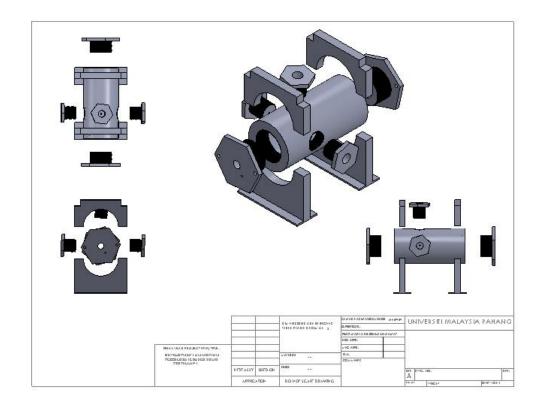
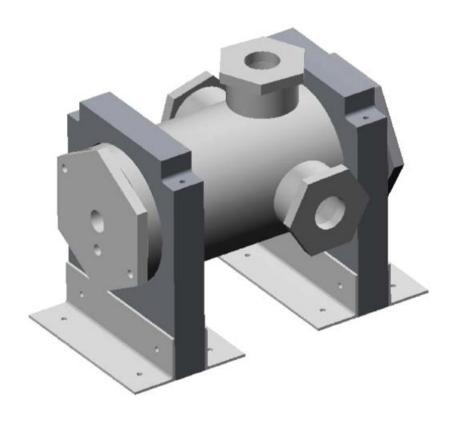
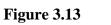


Figure 3.12Assembly Of Strand Burner body part





Strand Burner [30]

3.4 Burning Rate Timer

To get the time right burning rate test, a device used to measure time is important. The burning rate for this test, a timer that has been connected to a stopwatch and simple electronic circuit was used. This timer has been made and used by previous studies.



Figure 3.14 Burning rate timer

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

This chapter starts with the basic assumptions that must be made in order to simplify the analysis of a rocket motor. Then, the example of a given propellant formulation was analyzed from the combustion equation until the properties of the propellant reaction products such the combustion temperature, average molecular weight specific heat ratio and burning rate, but for this project, what we focus is just burning rate.

4.2 Effect of combination of each ingredient

As we know, the propellant is too easy and sensitive to heat or any spark and to any igniter. In producing that propellant, combination and formulations of ingredients must be obtain the accurate percentage and value. An ingredient like HTPB is inert at low temperature and could not burn or self ignited. In an observation at conducting some testing on the burning capabilities of various ingredient combinations has resulted in burning characteristic shown in Table 4.1 and Table 4.2 [44]. This testing was run at the atmospheric pressure and 'industrial igniter lighter' was used to initiate the burning process for all combinations. To minimize the risk, each of samples was tested with a small amount of materialism and all burning testing we do in prospect glass box. Figure 4.1 shows the flame of f60 and Figure 4.2 show the prospect glass box.

Compounds	Combinations										
АР	✓				~	\checkmark	~				√
AI		~			~			\checkmark	\checkmark		~
НТРВ			~			~	~	~	\checkmark	~	\checkmark
IPDI				~			~		~	~	~
Note	Ν	N	N	N	N	BD	BR	N	N	BD	BL

Table 4.1Effect of combination of each ingredient [44]

Table 4.2Description [44]

Note	Observations
N	Cannot burnt
BR	Burnt with red flame and dark smoke
BD	Burn with dark smoke
BL	Burnt with luminous flame and white smoke (Error! Reference source not found.)





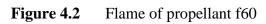




Figure 4.3Burning test prospect glass box

4.3 Burning Rate Test

Time for the burning rate test taken by using burning rate timer. Techniques to take readings of the propellant burning is to poke into the propellant 'finex solder lead' in two places at the top and bottom. When the propellant started to burn at the top until it cut the finex solder leads, stopwatch in timer will start counting time. When propellant burning is in a measure that has been marked, another finex solder lead at the bottom will be cut off and this will cause the time in stopwatch will stop counting. The time recorded on the timer will be taken as the reading time for that propellant burning. The same technique was repeated for the next propellant.

30mm 100mm 25mm

4.3.1 Burning time measure

Figure 4.4 Propellant strand

Propellant combustion testing time is calculated based on the measurements that have been marked. The length of most of the propellant is around 150-170mm based on the length of the straws. But the true measure of the combustion rate of this test is only 100mm. This is because the propellant burning rate at the beginning and end of combustion is unstable. So we decided just to measure only burning in the middle of the most effective burning of a propellant.

Formulation Code	O/F
f80	80/20
f75	75/25
f70	70/30
f65	65/35
f60	60/40

Table 4.3O/F Table

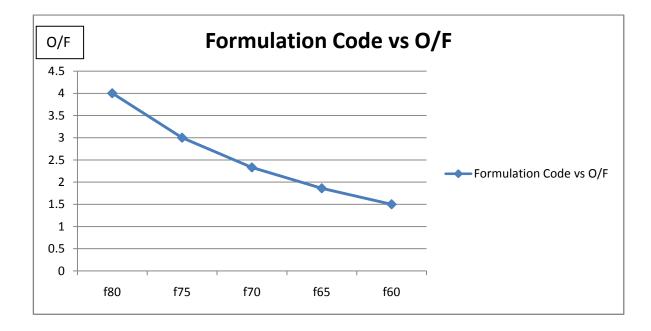


Figure 4.5 Formulation Code vs O/F

From the graph above, the data showed formulation verses ratio O/F. When a higher percentage of AP in propellant formulations, that mean the ratio of O/F is more high. This is because when the percentage of AP increase in 100g formulation, it will reduce the percentage of Al as fuel.

4.3.2 Result and discussion

Formulation Code	O/F	Time (Sec)
f80	80/20	68.04
f75	75/25	63.75
f70	70/30	62.20
f65	65/35	60.19
f60	60/40	58.26

Table 4.4Time of burning test at 1atm

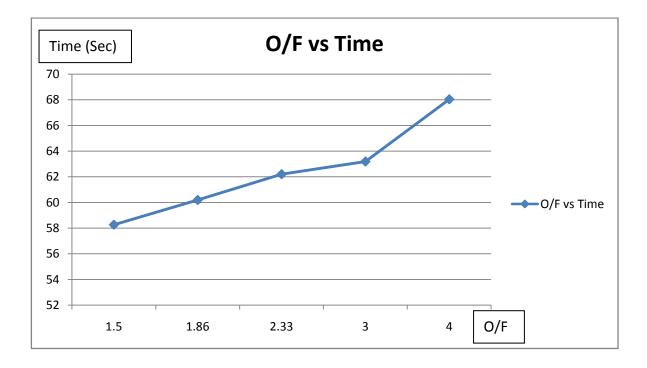


Figure 4.6 O/F vs Time

4.3.3 Burning Rate Equation and Calculation

$$r = \frac{Lp}{Tb}$$

 L_p = Length of propellant measured (mm) t_b = Burning time of propellant measured (sec)

Calculation

f60
$$r = \frac{100 \text{ mm}}{58.26 \text{ sec}} = 1.716 \text{ mm/sec} = 1.72 \text{mm/sec}$$

f65
$$r = \frac{100 \text{ mm}}{60.19 \text{ sec}} = 1.661 \text{ mm/sec} = 1.67 \text{mm/sec}$$

f70
$$r = \frac{100 \text{ mm}}{62.20 \text{ sec}} = 1.607 \text{mm/sec} = 1.61 \text{mm/sec}$$

f75
$$r = \frac{100 \text{ mm}}{63.75 \text{ sec}} = 1.568 \text{mm/sec} = 1.57 \text{mm/sec}$$

f80
$$r = \frac{100 \text{ mm}}{68.04 \text{ sec}} = 1.469 \text{mm/sec} = 1.47 \text{mm/sec}$$

Formulation Code	AP(%)	Al(%)	O/F	r (mm/sec)
f80	80	5	80/20	1.47
f75	75	10	75/25	1.57
f70	70	15	70/30	1.61
f65	65	20	65/35	1.66
f60	60	25	60/40	1.72

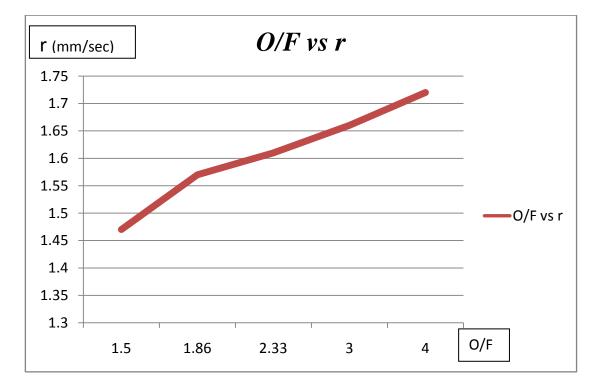


Figure 4.7 Burning rate vs O/F

The graphs above show higher rates of O/F will cause the lower propellant burning rate. This is because the higher the content of AP in the propellant would disturb the burning rate caused by the high oxidizer element, causing the fuel elements difficult to burn the propellant mixture.

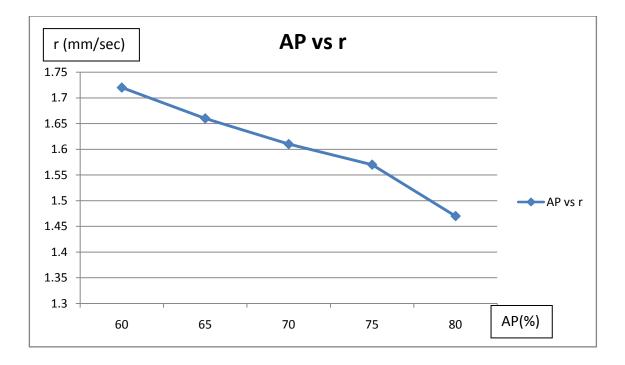
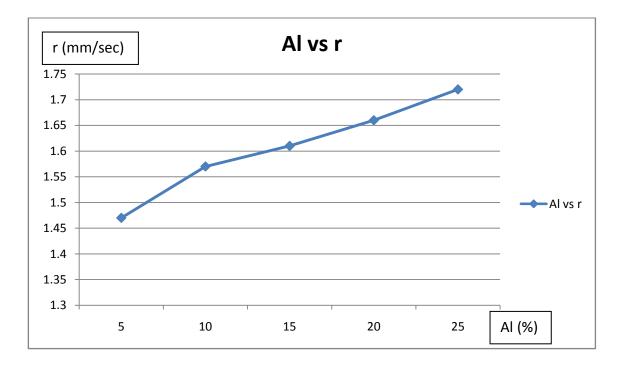


Figure 4.8 Ammonium Perchlorate (AP) vs Γ



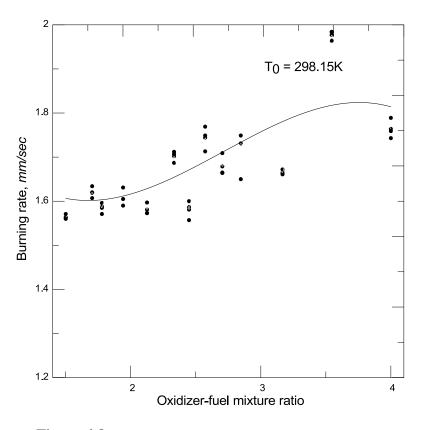


Figure 4.8 Burning rate at 1atm for all formulations [44]

At the atmospheric pressure, the O/F ratio will increase the burning rate of the propellant and this has been mentioned by Steinz J.A. This is evident when considering doubling O/F ratio from 1.941 to 4.0 which only increases the burning rate by 9.6% [44].

According to tests conducted by the authors, the results found that the O / F's really affecting the propellant burning rate, but the rate of the increasing of the burning rate with the increase of O/F ratio is very small at low combustion pressure as mentioned by Steinz J.A [44], that is different compared with result from this testing, when the rate of O / F is higher, the propellant burning rate is more lower. This may be caused by environmental conditions, compression methods and method to uniformly the formulation.

4.3.4 Effect of Ammonium Perchlorate (AP)

The widespread use of AP has been driven, in part, by the fact that the burning rate can be tailored by varying the particle size or by the addition of catalysts such as Fe2O3, CuO, Cu2O, MnO2, ZnO and MgO. It is well established that many transition metal oxide (TMO) catalysts including iron (III) oxide (Fe2O3) and copper (II) oxide (CuO) accelerate the decomposition of pure AP.

In general, it has been shown that both Fe2O3 and CuO lower the ignition temperature of AP, lower the low-pressure deflagration limit, and accelerate decomposition through all temperature regimes [45].

From the observations and in the opinion of the writer, heavily influenced AP burning rate of a propellant because of the high percentage of AP will cause a slow burning rate, that because too much oxidizer will cause the burning cannot efficiently, due to the low percentage of fuel.

4.3.5 Effect Of Aluminum Powder (Al)

During the last century a growing interest has been shown for the use of ultrafine metal powders (UFPs) as an additive to composite solid propellants.

This is associated with the fact that conventional composite solid propellants (CSPs) containing industrial micron-sized aluminum powders with an average particle size of $3-20 \ \mu\text{m}$ have completely exhausted their potential, while the progress in the technology of producing metal ultrafine metal powders (UFPs) with an average particle size of ~0.1 μ m (particularly, by wires electric explosion method) allows preparing rather large batches of metal powders with controlled properties and high stability. Conventional aluminum powders with the UFPs can increase the composite solid propellants burning rate by more than 2.5 times, reduce the factor V in exponential law of burning rate, and shorten the ignition time [46].

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

Reflecting on the objectives of this study, five sets of ammonium perchlorate based solid propellant namely f60, f65, f70, f75 and f80 have been successfully developed and tested for their burning rate. The results from the burning rate test are as follows.

Formulation Code	r (mm/sec)
f80	1.47
f75	1.57
f70	1.61
f65	1.66
f60	1.72

Table 5.1	Result of burning rate	test
-----------	------------------------	------

According to the table above, which refers to the burning rate test results, it was found that the percentage of AP and Al influence the burning rate of a propellant. Propellant f80 was selected for further evaluation in determining the performance of the low burning rate propellant for the next study.

Experimental results show:

- Solid propellant ammonium perchlorate can be produced through a technique compressed molding method.
- 2. The higher percentage of ammonium perchlorate as oxidizer in the propellant can make the lower burning rate propellant.
- 3. The higher percentage of aluminum powder as metal fuel in the propellant can make the lower burning rate of propellant.
- 4. The burning rate of propellant is dependent on the ratio mix of oxidizer-fuel and compression pressure applied during the process of producing the propellant.
- 5. The greater the value of the pressure can make the lower burning rate of propellant.
- 6. Maximum burning rate achieved in this experiment was 1.72 mm/sec at 60% of ammonium perchlorate (AP) and 25% of aluminum powder (Al).

5.2 Recommendation

Of the studies that have been made, there are some suggestions that may be considered for future students to improve and develop further research on AP based solid propellant. The proposal is as follows

- 1. Review the design and make a new strand burner that can withstand high pressure for the next test in burning rate gain in high-pressure conditions. Actual combustion pressure for a rocket is very high.
- 2. Change the variable material used for propellant formulations. If the percentage change in this study only the AP and Al, for future research, the proposed formulation for HTPB and IPDI also changed and modified to evaluate the effectiveness and an improvement in AP based solid propellant.

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