

EXPERIMENTAL STUDY ON COMPOSITE SOLID PROPELLANT MATERIAL BURNING RATE USING MATLAB ALGORITHM

by

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In rocketry application, now-a-days instead of monopropellants slowly composite propellants are introduced. Burning rate of a solid state composite propellant depends on many factors like oxidizer-binder ratio, oxidizer particle size and distribution, particle size and its distribution, pressure, temperature, etc. Several researchers had taken the mass varied composite propellant. In that, the ammonium perchlorate mainly varied from 85 to 90%. This paper deals with the oxidizer rich propellant by allowing small variation of fuel cum binder ranging from 2%, 4%, 6%, and 8% by mass. Since the percent of the binder is very less compared to the oxidizer, the mixture remains in a powder form. The powder samples are used to make a pressed pellet. Experiments were conducted in closed window bomb set-up at pressures of 2, 3.5, and 7 MN/m². The burning rates are calculated from the combustion photography (images) taken by a high-speed camera. These images were processed frame by frame in MATLAB, detecting the edges in the images of the frames. The burning rate is obtained as the slope of the linear fit from MATLAB and observed that the burn rate increases with the mass variation of constituents present in solid state composite propellant. The result indicates a remarkable increase in burn rate of 26.66%, 20%, 16.66%, and 3.33% for Mix 1, 2, 3, 4 compared with Mix 5 at 7 MN/m². The percentage variations in burn rate between Mix 1 and Mix 5 at 2, 3.5, and 7 MN/m² are 25.833%, 32.322%, and 26.185%, respectively.

Key words: *composite, solid propellant material, burning rate, image processing, particle size, binder, ammonium perchlorate, hydroxyl terminated poly-butadiene, toluene di-isocyanate, MATLAB*

Introduction

Many of the researchers performed several types of research on composite solid propellants, and much progress has been made but still to enhance the performance of solid propellants by considering the effects of various additives and the impact of fuel and oxidizer particle sizes on burning behavior is under research. This paper, propellant made is oxidizer rich, which is ammonium perchlorate (AP) content, is more compared to fuel. This article contributes an oxidizer rich propellant giving a more burning rate. The solid propellant is in pellet form. The AP particle is fine sized particles, which was obtained by grinding the coarse size and sieving to the required size as mentioned in the paper. The AP hydroxyl terminated poly-butodiene (HTPB),

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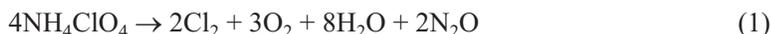
and toluene di-isocyanate (TDI) are taken in different masses which were not taken by any of the researchers so far.

Heterogeneous solid propellants are mainly mixing of oxidizer and fuel. The oxidizer is AP and binder is mostly made up of HTPB (pre-polymer), and TDI is the curing agent. Solid propellant combustion of five different proportions was taken for analysis. A solid propellant consists of several chemical ingredients such as an oxidizer, fuel, binder, plasticizer, curing agent, stabilizer, and a cross-linking agent. The specific chemical composition depends on the desired combustion characteristics for a particular mission. Different chemical ingredients and their proportions result in different physical and chemical properties, combustion characteristics, and performance.

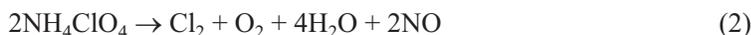
Introduction to solid propellant

Description of oxidizer ingredient

The AP, with a chemical formula of $(\text{NH}_4\text{ClO}_4)$, is the most widely used oxidizer. It is a white crystalline material [1] that is usually orthorhombic but transforms into cubic form at 513 K. It starts to decompose at approximately 470 K according to the next global chemical reaction:



Beyond 620 K, it decomposes according to this global chemical reaction:



When AP is burned with polymeric hydrocarbon fuels [2], it produces mainly CO_2 , H_2O , N_2 , and HCl . The AP is widely used for propellants [3] and explosives due to its high oxygen balance and relative stability to mechanical shocks.

Preparation of AP particles

The AP particles are available from 400 to 500 μm . The AP particle size was brought down by crushing with the planetary ball mill. The range of different particle sizes obtained with the help of sieves and sieve shaker. The AP particle is an affinity to moisture, and it will clump together. The prepared AP particles are stored in desiccators and to remove moisture, AP kept in hot air oven at temperature 333 K. The size of AP particles considered here was 63 to 75 μm . Burning rate depends on oxidizer particles size also [4, 5].

Description of fuel binder

The fuel binder provides the structural glue or matrix in which solid granular ingredients (such as oxidizer particles and metal fuels) are held together in heterogeneous (composite) propellants. In the present study, the fuel cum binder is HTPB. The HTPB is the most commonly used prepolymer binder material [6]. It allows a high solid fraction (88% to 90% of AP and Al by mass) and relatively good physical properties at the temperature range from 223 K to 333 K.

Description of curing agent

A curing agent or cross-linker causes the prepolymers to form longer chains of larger molecular mass and interlocks between chains. Even though these materials are present in small amounts, a minor change in the percentage can have a major effect on the propellant physical properties, manufacturability, and aging. These ingredients cause the binder to solidify and be-

come hard. Curing agents are used to maintaining the structural integrity of a propellant grain. In this paper, the curing agent used is TDI. The choice and selection of curing agents depend on type of pre-polymer used. The curing agent influences the mixing process, burning rate, and combustion.

Composition of solid propellant

Solid propellant [7] was prepared by mixing measured mass of oxidizer particles and binder in a proper proportion as shown in tabs. 1 and 2.

Table 1. Ingredients details

Ingredient type	Type	Source
AP (NH_4ClO_4)	Oxidizer	Tamil Chlorates, Madurai
HTPB [$\text{C}_4\text{H}_6(\text{OH})_2$] _n	Binder and fuel	Satish Dhawan space center, Sriharikota

Table 2. Composition of samples

Mix no.	% Binder	% Total solids	% HTPB	% TDI
1	0	100	–	–
2	2	98	1.88	0.12
3	4	96	3.76	0.24
4	6	94	5.64	0.36
5	8	92	7.52	0.48

Preparation of an AP pellet

A stainless steel die of a circular cross-section of one-inch diameter as shown in fig. 1 is used to make AP pellets. The AP, as obtained, is ground into a fine powder of size between 63 and 75 microns. Moisture from AP is removed by keeping the powdered AP in a hot air oven at 333 K for 24 hours. Around 1.8 g of this dried powder is used to make the pellets using a stainless steel die. A pressure of 200 bar is applied on this die for two hours with a hydraulic press as shown in fig. 2. The pellets thus obtained are maintained at 303 K in an oven for a day to achieve a uniform temperature throughout the sample. The density of pellet is 1900 kg/m^3 which is close to the single crystal density of 1950 kg/m^3 . To conduct experiments on the pellets, samples of size $5 \text{ mm} \times 1.68 \text{ mm} \times 10 \text{ mm}$ are obtained. Five different compositions of propellant were made, and the experiment was conducted.



Figure 1. Circular cross-section stainless steel dies

Preparation of solid propellant

The AP, HTPB, and TDI are hand mixed for more than 30 min in a beaker of 100 ml capacity, and the mixture is kept in a hot air oven at 333 K for seven days for curing. From each of Mix number, 2 to 5 [8], 1.8 gram is taken to make a pellet in a hydraulic press of about 150 kg/cm^2 for two hours. The pellets made with the mix no 2 to 5 are kept in a hot air oven at 333 K to remove the moisture from them before conducting the experiment. Figure 3 shows the distribution of AP particles where the binder is present at the interface between the particles.



Figure 2. Hydraulic press

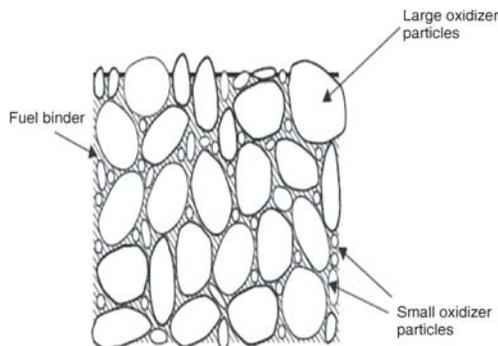


Figure 3. Distribution of AP particles with binder

Experimental set-up

The present study employs experiment [9] to determine burning rate measurements based on combustion photography. Five types of samples are explored in this current work. First sample pure AP and the remaining all samples contain HTPB binder. The binder is made of HTPB pre-polymer and TDI as the curing agent. Samples primarily vary in the AP and binder composition. The mass ratio of AP and binder are shown in the tab. 1.



Figure 4. Window bomb set-up



Figure 5. Sample images

All samples are tested for burning rate measurements by combustion photography in the pressure range of 2, 3.5, and 7 MN/m². The combustion chamber is pressurized with the nitrogen and constant pressure in the combustion chamber during sample burning is maintained by venting the small amount of flow. The experiments are performed in an optical set-up called window bomb set-up shown in fig. 4. A CCD camera captured the combustion photography and recorded. The window bomb is a cylindrical pressure vessel. The cylindrical chamber has two windows made of toughened glass along its curved surface. The purpose of the smaller window is to facilitate the imaging of the burning process, and the other window is used to provide external illumination. The recorded images can be viewed frame-by-frame for determining the burning rate of the samples by using the shuttle ring control in the video cassette recorder. Before ignition, the chamber is flushed with nitrogen gas followed by maintaining the pressure required for combustion.

The sample is then ignited, and the images were recorded. The consecutive images of the burning samples are shown in fig. 5. The images were viewed frame-by-frame, and the location

of the flame front in each frame is noted. The actual position of the flame front is plotted against the time of each frame, after adjusting for the magnification of the images. The burning rate is obtained as the slope of the linear fit from MATLAB plot.

Combustion photography

The chemical reaction in the solid propellant is much faster. Hence, the burning rate is fast and thus consumes the propellant faster. To capture the faster regression rate of the propellant, a high-speed camera (max 5000 frame rate per second) is used to capture the frames. The movement of the edges of the flame is detected frame by frame, with image processing technique, an algorithm is written to identify the flame edges to determine burning rate.

Burning rate can be calculated by knowing the height of the propellant and the time taken by the propellant (strand burner technique). If there is any void or air gap in the propellant, the correct burning rate can not be achieved. With high-speed camera at higher frame rates, even if there is any void in a fraction of millimeter distance can be avoided by considering other frames due to other continuous flame regression. The following fig. 5 shows few sample images of combustion photography taken in high-speed camera.

Image analysis in MATLAB

Image analysis technique in MATLAB [10] gives information about the structure of an image. Some of the image analysis techniques are; (a) edge detection, (b) boundary tracing (c) quadtree decomposition edge detection.

The edge function to detect edges, which corresponds to object boundaries in an image. This function determines the intensity changes rapidly in the image and finds the edges. Edges are recognized in places based on two criteria; (a) where the threshold value is less than the magnitude of the first derivative of the intensity and (b) where the second derivative of the intensity has a zero crossing edge provides a number of derivative estimators, each of which implements one of the previous definitions. The Canny method is the most powerful edge-detection method which differs from the other edge-detection methods. This approach uses two different thresholds to detect strong and weak edges. It includes the weak edges in the output only if they are connected to strong edges. This method is, therefore, less likely than the others to be fooled by noise, and more likely to detect true weak edges. The following algorithm was developed in MATLAB to process the images, to plot a graph between pixels and frame number.

Burning rate calculation

The burning rate of a solid propellant is also given by the empirical Saint-Robert's law (or Vieille's law) as, $r_b = ap^n$, where a usually is a function of the initial temperature, p is the pressure, and n is the pressure index. The burning rate increases as the pressure increases [11-13]. The burning rate of pressed AP (Mix 1) measured by Atwood [14] is similar to the current values obtained. By considering the number of pixels and pixel distance in mm, and the number of frames and frame rates, the slope of each case is determined MATLAB, which gives the burning rate measurement for five samples in mm per second.

Results and graph

The following graph were obtained by running the algorithm in MATLAB and plotted pressure [Mpa] vs. burning rate [mms^{-1}] (fig. 6, tab. 3).

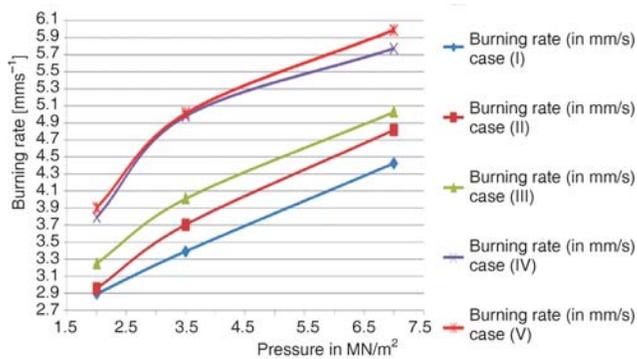


Figure 6. Burning rate results for all the samples

Table 3. Percentage increase in burn rate at 7 MN/m² and binder content

Mix no's	% increase in burn rate at 7 MN/m ²	% increase in binder content	% decrease in oxidizer content
1 to 5	26.66	100	8
2 to 5	20.00	75	6.12
3 to 5	16.66	50	4.16
4 to 5	3.33	25	2.12

at 2 MN/m², 1.62 mm/sec at 3.5 MN/m², and 1.569 mm/sec at 7 MN/m². The percentage variation in burn rate between mix 1 and mix 5 at 2 MN/m² is 25.833, at 3.5 MN/m² is 32.322, and at 7 MN/m² is 26.185. The accuracy of the burning rate value entirely depends on the clarity of the images; blurred images may not give good results.

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Nomenclature

AP – ammonium perchlorate

TDI – toluene di-isocyanate

HTPB – hydroxyl terminated poly-butadiene

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Conclusions

The burning rate for five mixes at three different pressures was calculated from the combustion photography (frame by frame) captured by a high-speed camera. The acquired images were run by writing an algorithm in MATLAB. It is concluded that the oxidizer rich fine particles exhibit more burn rate with increase in pressure. Mix 1 to 5, the burn rate increases due to increase in small increase in binder content and more increase in pressure. As burn rate depends on pressure also, tab. 3 shows the variation of burn rate at 7 MN/m² among the various mixes. Therefore, the oxidizer rich propellant with the small variation of binder content shows a remarkable increase in burn rate.

The results between mix 1 and mix 5 shows that there is an increase in burn rate of 1.008 mm/sec

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