

## CHARACTERIZATION OF POTASSIUM NITRATE/DEXTROSE SOLID ROCKET PROPELLANT USING CALORIMETRY

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### ABSTRACT

The use of the Potassium Nitrate as an oxidizer in solid rocket propellants has been common in recent years for experimental research in solid rocket propulsion systems. This chemical is used as a compound of black powder and explosives, but in the last 30 years, it has been studied for applications in semiprofessional rocket propellant, implemented by Universities in research and academic projects. The Potassium Nitrate/Dextrose propellant is also known as Candy propellant and presents some advantages compared with other solid propellants. They are low production costs and ease of acquisition for the chemical components. These advantages are attractive for low-cost projects, compared to the low performance; this does not discourage its use.

The Candy propellant presented a lack of experimental data to characterize, for example, its thermal properties. For this reason, were used the Differential Scanning Calorimetry and the Semi-Micro Calorimetry tests, to obtain the heat of combustion, power, and temperatures of melting and ignition. The grains of Candy propellant were manufactured at the University of Concepción by the Propulsion Interest Team. This is the first time which is reported the calorimetric data of the Candy propellant (Potassium Nitrate/Dextrose). The results in this work presented a heat combustion in the range 523 - 567 Cal·g<sup>-1</sup>, decomposition temperature in the range 356 - 361 °C and a heat released in the range 1080 - 1809 J·g<sup>-1</sup> of the mixture KNO<sub>3</sub>/Dextrose, which contribute to the professional characterization of the propellant.

**Keywords:** Propellant combustion, Potassium Nitrate, calorimetry, solid rocket motor, solid propellant.

### 1. INTRODUCTION

The Potassium Nitrate (KNO<sub>3</sub>) is a common oxidizer used in the fabrication of solid propellants for propulsion systems, igniters or burners, and explosives (1,2). Since 1960, the KNO<sub>3</sub> with sugar as based fuel, was implemented for the first time in the fabrication of a low cost / low-performance propellant, showing low specific impulse compared to propellants with Ammonium Perchlorate (1). The implementation of this propellant and its derivatives (with sugar – SU, dextrose - DX, or sorbitol - SB) has been popular for the development of small and low cost Solid Rocket Motors Systems (SRMS) around the world, specifically in academic research, and also known as candy propellant, few successful projects were implemented in engineering departments of universities like TU Delft, see (3–5). The production of solid propellants allows the design and implementation of sounding rockets, essential for the development of aerospace technology. Focus in the case of countries without previous researches in aerospace propulsion systems, infrastructure for the development of large motors, regulations for the use and commercialization of high energetic chemicals and/or limitation of budget, the use of the KNO<sub>3</sub>-SU propellant is the only available option to introduce students and young professional to the technology of the propulsion. Some experiences around the world present the advantages and successful cases of the implementation of low cost propellant for the aerospace research, and are reported by Cho et al. (2008); (6–8). The most important academic and research projects using the Candy propellant, in the last decade (2010-2020), was development by the Delft Aerospace Rocket Engineering (DARE). The validation of this propellant during a flight test, resulting in the altitude record of a sounding rocket impulsed with candy propellant, reaching an apogee of 12.3 km. The DARE presents a detailed reports and publications of their experiments with candy, presenting the solution to problems like: the manufacturing process, in which it is needed the implementation of a (pre) heating of the grain molds, including the addition of mechanical pressure during the curing of the grain, to improve the final grain, and to reduce the defects of the fabrication. Also, the motors developed by DARE, were tested in ballistic evaluation to characterize the propellant, obtaining values of specific impulses larger than 128 s (5). Other characterization methods of the KNO<sub>3</sub>/SU were documented by Olaoye and Abdulhafeez (9), showing similar behaviour and properties.

In Brazil, the Rocket Design team from the Technology Aeronautical Institute (ITA), developed rocket motors with the KNO<sub>3</sub>/SO propellant, presenting practical methods for the manufacturing including the use of a casting machine (3,10) and the development of high-performance propellant (11). Other academic projects include the use of KNO<sub>3</sub>/SB in the second stage of a sounding rocket to

recover a glider, work that was presented by Baran et al. (12). Not only sugar is used, also, sucrose composed foods can be used for the design and fabrication of propellants. Results from experimentation with chocolates and other sucrose products were reported from the ballistic test and presented by Wada et al. (13), showing a low specific impulse, low thrust, and acceptable performance.

The manufacturing of the propellant begins with the selection and quality of the chemical components, a demanding task. For the use of the KNO<sub>3</sub> in the candy propellant, it is important the quality and the purity of the grain, due to the direct influence in the mixture homogeneity, resulting in the efficiency of combustion and the performance of the average thrust and specific impulse. Different methods are presented to obtain high purity KNO<sub>3</sub> from the fertilizer for use in propellants. Baldissera and Poletto (14) applied a method to reduce the sulfur component of the fertilizer after the process.

The specific impulse and the burning rate coefficients of the propellants KNO<sub>3</sub>/SU, KNO<sub>3</sub>/SB, and KNO<sub>3</sub>/DX with a mixture ratio of 65% of oxidizer with 35% of fuel (O/F) are presented in different reports (5,6). The mean value of the thermochemical analysis for this mixture is specific impulse I<sub>sp</sub>=164 s for KNO<sub>3</sub>/SB, with a mean characteristic velocity of 938 m/s, and combustion temperature around 1600 K. In the case of the KNO<sub>3</sub>/SU the ideal specific impulse is 166 s, with a theoretical temperature of combustion of 1720 K. The KNO<sub>3</sub>/DX presents the same I<sub>sp</sub> that the SB, with 912 m/s of characteristic velocity at 1710 K of combustion. These values were verified and validated during the propellant performance and applications in flight, presented by the DARE, resulting in successful implementation in sounding rockets.

The thermal, mechanical, chemical, and thermochemical data of the propellant and the products of the reaction are essential parameters to the nozzle and motor design also determines the rocket flight performance. In the case of the mechanical properties, it is important to analyse the propellant bars using dynamical mechanical analysis and stress relaxation tests, to reduce the risk of malfunctioning (15). One of the first tests with solid propellants is the measuring of the propellant burning rate with strain burners. The performance analysis of chemical propellants begins with the analysis of the combustion to determine the adiabatic flame temperature and thermochemical properties, traditionally using computational methods to simulate and solve the equilibrium equations, assuming an ideal rocket motor, also can be estimated from analytic and semi-analytic approximations. A detailed survey of the solid propellant thermochemical properties, including the influence of the temperature in the burning process and burning rate, is discussed by Kubota (16), and the modelling of characteristics of solid propellants by Oyedeko and Egwenu (17).

When the motor is developed, data from the propellant performance can be obtained from static thrust testing, like the Ballistic Evaluation Motor (BEM) (16,18–20). At this point, none of the analysis determines the quantity of energy, which is necessary to ignite the propellant, and the quantity of heat generated by the combustion process, which means the stability of the propellant.

The development process of a propellant requires multiple levels of Technology Readiness (TRL), in this case, called the Propellant Readiness Level (PRL). In the PRL 1 are defined the basic properties of the candidate propellant, including the density, melting, and boiling points, the heat of formation, latent heat of vaporization, viscosity, specific heat, and others. The highest level is the PRL 9, used to describe the successful application of the propellant during a flight testing, ending the characterization (21).

For the characterization of the propellant and to obtain the heating rates, the application of the calorimetric tests during the PRL-1 is necessary, and not only for the solid rocket, for aerospace propulsion systems, like was presented by Pal et al. (22) for hybrid rocket, comparing the theoretical results of the mixture with the experimental data.

The calorimetric analysis of the propellant is a complex work, due to the explosive grade of the chemical components. This analysis is necessary to determine the influence of the propellant mass and humidity in the generated heat and the compatibility of the compounds (23,24), also, when a new compound is added or changing the mixture, for example when applied to a nitrocellulose base (25), and to determine the impact of storage and time in aged propellants (26). Results of calorimetric analysis of composite propellants to obtain the total heat of the propellant as a function of the temperature and time, were presented by (27–29) and, in the case of propellants with Ammonium Perchlorate, the results were presented by (30). The most studied solid propellants are the composite, due to the stability, good mechanical properties, high performance, and application in space transportation systems like launcher rockets, also in commercial applications for security forces like ammunition and missiles. There are multiple results of research in composite solid propellants reported in the last years due to the commercial applications, but some materials for the manufacturing are restricted for civil use, or restricted for exports. However, results from the calorimetric analysis of Candy propellants have not been published until this date.

This contribution describes the analysis as part of the PRL-1. Therefore, in this article we present for the first time are presented the results of the  $\text{KNO}_3/\text{DX}$  propellant characterization using calorimetric techniques in facilities affiliated with the University of Concepcion.

## 2. EXPERIMENTAL SECTION

### 2.1 General Information

All the chemicals and solvents used were of analytical grade and purchased commercially. The Potassium Nitrate ( $\text{KNO}_3$ ) was obtained from retail as fertilizer from Sociedad Química y Minera de Chile (31). Dextrose was obtained from Furet.

#### 2.1.1 Propellant manufacturing process

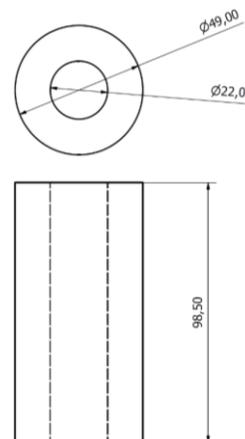
The Potassium Nitrate (Oxidizer) is obtained from retail as fertilizer, hence a purification process based on dissolution and recrystallization is used to remove the insoluble impurities and provide a consistent raw  $\text{KNO}_3$  for the propellant manufacturing. After the crystals are dried, their pH is measured to be in the range between 6 and 7. pH for  $\text{KNO}_3$  can be between 5.0 and 7.5.

According to the manufacturing process, and the access to testing facilities, only four propellant grains samples were produced, but due to defects, only three tested. Their properties are presented in Table 1.

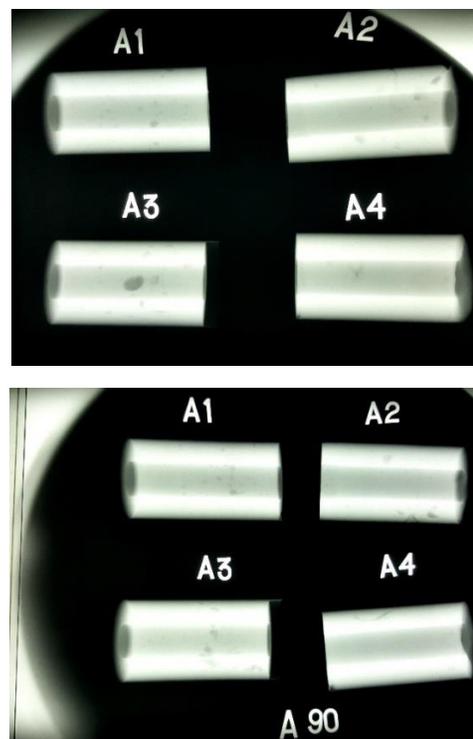
**Table 1.** Summary propellant sample grains.

ID	Composition in dry mass	Manufacture date
A1	65,0% $\text{KNO}_3$ + 35,0% DX	January 16 <sup>th</sup> , 2019
A3	65,0% $\text{KNO}_3$ + 35,0% DX	January 11 <sup>th</sup> , 2019
A4	62,8% $\text{KNO}_3$ + 37,2 DX	December 19 <sup>th</sup> , 2018

The probes were melted and molded into a cylindrical form, resulting in the grain of **Figure 1**. To guarantee the isolation of the grain and prevent humidity, the samples were stored under vacuum. The grains are inspected to detect fissures, air bubbles, or other imperfections during the fabrication. The X-rays were used to inspect the sample grains. Note that the tests performed to take a minor sample of the grain so the manufacturing defects identified do not pose a problem for the calorimetric characterization of the propellant described in the next section.



**Figure 1.** Grain dimensions in mm.



**Figure 2.** X-rays of grain samples. Each (left and right) are taken with 90 degrees rotation.

### 2.2 Test description

Two techniques were used to measure calorimetry in the samples, one is the Semi-Micro Calorimetry (SMC) to obtain the heat of combustion, and the second one is the Differential Scanning Calorimetry (DSC) to obtain the heat released and the decomposition temperature. The techniques were applied in the analysis of composite and hybrid propellants (22,23,26).

Samples from 1 to 2 mm of diameter were obtained from each base of the grains. After this, the samples are further reduced to that amount, approximately 0.2 g, to produce a 0.2 to 0.3 °C temperature change in the SMC.

2.2.1 Semi-Micro Calorimetry (SMC) - combustion heat

The measures were obtained using a model 1425 PARR calorimeter, where its procedure states the sample analysis should be reduced to grains of approximately 1 to 2 mm in diameter due to the dimensions of the chamber (32). Then, the sample quantity must be selected to produce a variation in temperature of 0.2 °C to 0.3 °C, in this case corresponding to 0.2 g (mass of the sample). Due to the unknown value for  $H_c$  of the propellant (since no values are available in literature and it has not previously been characterized), a small sample was used in order to comply with maximum calories limit for the calorimeter.

The relation that describes the heat of Combustion ( $H_c$ ), according to (32), is:

$$H_c = \frac{(W \times dT) - e}{m} \quad (1)$$

where  $H_c$  is measured in Cal/g,  $W$  is the calorimetry water equivalence in Cal/°C,  $dT$  is the temperature variation in °C,  $m$  the mass of the sample in gr, and  $e$  is the correction due to the ignition wire in Cal.

2.2.2. Differential Scanning Calorimetry (DSC)

The METTLER TOLEDO DSC822 Differential Scanning Calorimeter was used for the testing. The procedure followed was, taking a sample between 4 a 15 mg, placing it into a 40 microliter Aluminium cresol, and heating from room temperature at a rate of 10 °C/min in a Nitrogen (0.01% Oxygen) environment in the range of 25-500 °C. The main goal of this test was to assess the ignition temperature of the propellant.

3. RESULTS AND DISCUSSIONS

The information on the thermal characteristics is important for the operation and safety manipulation of the propellant. Also, observe and describe the regions with energy peaks.

In this case, is selected the mixture of  $KNO_3$ /DX composed of 65/35 O/F due to group heritage experience as well as other reference teams using the same ratio (5,6). With the selected propellant, were molded three hollowed test tubes or grains probes, with a mass around 270.0 g, and dimensions height 98.5 mm, external diameter 49.0 mm, and internal diameter 22.0 mm (as can be seen in **Figure 1**). Before the production of the grain, the  $KNO_3$  is purified by a recrystallization process. After this process, the solid power is grounded to obtain 50  $\mu$ m of granulometric, at this point, it is possible mixing with Dextrose, to guarantee a homogeneous distribution of the molecules. The propellant manufacturing process is further described in the following section.

The grain production process is presented schematically in Figure 3. Other approaches for the grain production process are discussed by Olde (5). Note that the process is not done completely for every sample, as an inventory of materials in different stages of the process exists, hence the process normally will start at the Entry Level Quality Control step to check for water contamination in the Dextrose due to its hygroscopic nature (5).

As described in the previous section, the purpose of the tests is to characterize the calorimetric properties of the propellant and not of the grains. Hence, this was only visually and X-ray inspected in search of manufacturing defects, as can be seen in **Figure 2**. Furthermore, the reason for manufacturing grains and not only small samples was to assess the homogeneity of the mixture process.

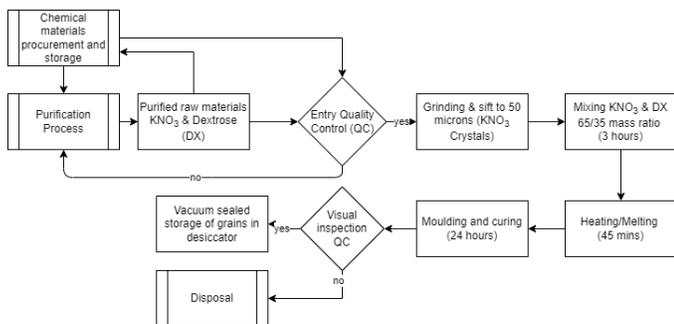


Figure 3. High-level propellant production process.

The SMC is determined by the combustion of a known quantity of sample, pressurized with nitrogen. The sample is submerged in a known quantity of water, inside an adiabatic chamber. The determination of the Heat of Combustion of the sample is obtained by measuring the temperature variation in the water. With an exact measurement of the temperature variation inside the chamber and knowing the calorimeter water equivalent (Equivalent Energy Factor), the number of heat units released can be calculated according equation 1. The Calorimeter water equivalent corresponds to the amount of heat (Cal) needed to raise the temperature of the components inside the adiabatic chamber by 1-degree Celsius (°C).

The heat of combustion was determined for all samples and is summarized in **Table 2**. Note that a variation in the measures for the same grain varied between 3.0% and 4.9% (when taking the difference between both measures and dividing by the mean value). Furthermore, in the visual inspection conducted after the combustion a significant amount of residue was observed.

Table 2. Heat of combustion measures for the different samples (Test conducted April 26<sup>th</sup>, 2019).

Sample ID	Heat of Combustion (Cal/g)	Sample Mass (mg)
A1-1	545.25	7.3000
A1-2	561.63	14.0000
A3-1	567.05	13.1000
A3-2	565.88	8.9000
A4-1	550.13	6.3000
A4-2	523.89	8.2000

As was presented in **Table 1**, the composition of sample A4 is different from that of A1 and A3, to assess the impact of the hygroscopic nature of Dextrose a different mass ratio was used in the mixing process.

The DSC is used as an instrument that allows the measurement of the differences in the amount of heat between the sample and a reference, obtaining characteristics of the propellants presented in the thermograms. Differences in the amount of heat between the sample and the reference are presented as a peak on a Heat as a function of the Temperature (Thermogram) and indicating that an endothermic or exothermic process has occurred. The comparison of the thermogram of the sample with the thermogram of a standard sample allows quantifying the variations of heat in the analyzed sample. When a variation is detected the nature of this change can be explained according to the type and magnitude of the peak.

Regarding the DSC curves, the results are summarized in **Figure 4**. Since the properties of the propellant were unknown at the time of the test, the lab technician and solid rocket propellant experts recommended a 10°C/min rate to determine the temperature points at which the mixture will ignite.

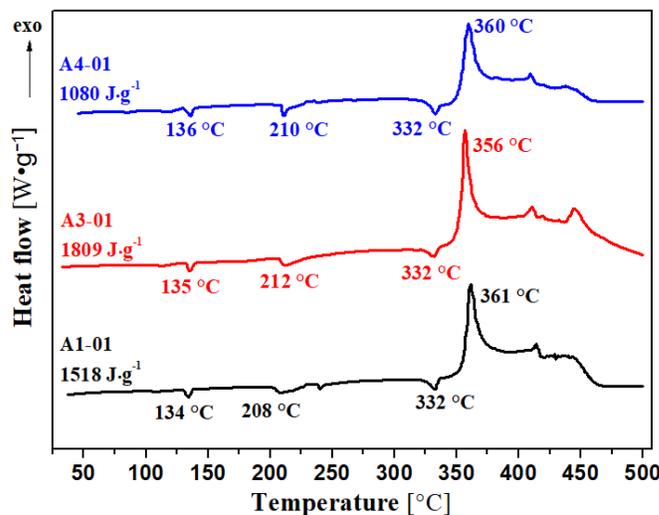


Figure 4. Results of the DSC tests conducted on March 12<sup>th</sup> and April 8<sup>th</sup>, 2019.

From left to right, i.e. lower to higher temperatures in the DSC plot of **Figure 4**, we can observe:

A sharp endothermic peak at 135 °C corresponds to solid-solid transition change of KNO<sub>3</sub> from one crystal structure orthorhombic to rhombohedral (14).

The second endothermic process at 210 °C corresponds to a fusion/melting temperature of dextrose (33,34).

The third endothermic peak at 332 °C corresponds to the melting of purified Potassium Nitrate (14).

After these three endothermic reactions, a clear exothermic peak is achieved in the range 356 – 361 °C, which corresponds to the ignition temperature for the propellant.

The second exothermic reaction is attributed to residual combustion of elements as well as residual heat, as the abundant residue is observed through visual inspection in all the samples after combustion.

It was noticed that the exothermic decomposition processes exist with peaks located in the range of 356 – 450 °C and the exothermic heats were in the range of 1080 – 1809 J·g<sup>-1</sup>.

### CONCLUSIONS

The propellant KNO<sub>3</sub>/DX was selected for calorimetric analysis using DSC and SMC techniques. Three grains with two samples each were tested obtaining the behaviour of the energy as a function of temperature. The results presented a similar behaviour, presenting a maximum of around 357 °C, and a heat released in the range 1080 - 1809 J·g<sup>-1</sup> of the mixture KNO<sub>3</sub>/Dextrose., however, those are preliminary analysis and it is required more testes to obtain a more accuracy behaviour.

The calorimetric analyses are important to the propellant characterization, and the results describing the principal thermodynamic characteristics of the KNO<sub>3</sub> and DX. These results complement the scientific reports of the Candy propellant to future applications in aerospace research. The methods used in this paper allow to validate the propellant with the reported data obtained in ballistic tests and thermochemical analyses.

Further statistical analysis requires a bigger number of samples and is left for future research as the purpose of this was to characterize for the first time the propellant. Furthermore, DSC analysis of the component elements will support the characterization of the propellant once mixed.

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