KNSU SOLID PROPELLANT GRANULOMETRY EFFECTS ANALYSIS INVOLVING A BALL MILL

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Abstract. *This paper shows the effects of the propellants components granulometry in its combustion, essential to thruster's efficiency. The manufacturing of solid propellant for rocket motors requires the milling process of each component separately. It is fundamental that its grains are in the proper dimension in order to reduce the reaction´s activation energy. This happens once the contact area is enlarged. The research is based on KNSU, a mixture of sucrose and potassium nitrate. The research goal lies on the grinding time, aiming a homogeneous and efficient combustion. A ball mill was designed and built to assist the research. The mill products go through tests that verify the grains size and, then, are mixed together. Samples are tested in calorimeters and the residues are collected for analysis in a mass spectrometer. The propellant tests also involve burning time, mass consumption/grinding time rate and residues mass proportion.With the acquired data, it will be possible to better describe and manipulate solid propellants combustion and, consequently, improve computational models that are used on rocket engine nozzle and combustion chamber design.*

Keywords*: KNSU solid propellants, granulometry, milling*

1. INTRODUCTION

Solid propellants motors are the simplest rocket design. Its advantages, such as high density, low volume, low costs, instant ignition without fuelling operations, low technical risk, long-time storage life and high reliability, make them ideal for military and aerospace applications. The main disadvantages are the heavy and large combustion chamber required for a long period of combustion and the fact that once the combustion starts there is no shutoff option or thrust adjustment. However, the greatest danger is that oxidant and fuel are stored together, which increases the chances of an accidental combustion. They can be found in military rockets, in launch vehicles first stage or in payloads that need to be boosted to higher orbits.

In solid propellant rockets, all the propellant to be burned is contained within the combustion chamber. This propulsion type unit is particularly adaptable to short duration firing. The propellant charge contains all the chemical elements for complete burning. Once ignited, it burns at a nearly constant rate on the exposed surface of the charge. Since there are no feed systems, valves or pumps, such as the required by liquid units, solid propellant rockets are relatively simple construction (Sutton, 1949a).

The oldest known source in which rockets are mentioned is Chinese, and the invention and the first use dates the earliest 1232. It is believed that possibly the Chinese heard, from an occasional traveler, of a Greek invention called "Wildfire." It was a variable flammable mixture of varied composition. Ingredients often used were tow, pitch, turpentine, sulphur, charcoal, and occasionally naphtha, petroleum, and incense. This or a similar powder may have propelled the Chinese rocket. It is believed the rocket idea reached Europe between 1249 and 1280, probably brought by Arabs. By 1400 experiments of military engineers of various countries had produced more than one type of rocket (Sutton, 1949b). The nineteenth century is marked by the war rocket improvements. Finally, composite motors were developed with a high performance in the 1950's. In the 1990's, U.S. Air Force used a expendable rocket named Titan IV that could launch military or civilian intelligence agency payloads which had two solid-rocket motors.

As listed above, solid propellants were the most used throughout history. This is because it is the easiest to handle, to use and to storage. Currently, due to those benefits, it is the propellant most studied. Among solid propellants there are the sugar propellants such as KN-Sucrose Propellant (KNSU), KN-Dextrose Propellant (KNDX) and the KN-Sorbitol Propellant (KNSB). The most used is the KNSU propellant that consists of potassium nitrate and common table sugar. Thus, it is easy to obtain, to produce and to manipulate. Moreover, its combustion produces a large amount of smoke that is mainly water vapor and Potassium Carbonate dust, and therefore results on non-toxic exhaustion gases. For all these reasons, it is a solid propellant widely disseminated among researchers and amateurs.

In this context, this paper seeks for improvements in the use of KNSU solid propellant. Focusing on that, firstly potassium nitrate and sugar, both commercially available, were treated in a ball mill, aiming combustion efficiency.

A series of tests were devised to analyze the grinding effects on the combustion efficiency, such as microscopic grain size analysis using SEM (Scanning Electron Microscopy), the study of burning duration of propellants made of mixtures of different milling times, propellant cooking time influence study, propellants calorimetric analysis and combustion residues analysis.

2. METHODOLOGY

In the first procedures, each component went through different grinding periods in a ball mill developed by Monteiro *et al.* (2010) and the consequences of this process in the components were analyzed. The grinding effect on the propellant behavior was analyzed both in the fuel and oxidizer separately, as it was analyzed on the propellant when ready.

The grain size analysis was performed for each component separately. The grinding time ranges initially imagined were a half-hour, one hour, two hours and four hours, after which the components and their respective propellants would be subjected to the tests proposed by the research. However, the components, when still isolated, began to show an increasing hygroscopic effect and, after two hours of milling, the masses that were removed from the mill were aggregated by absorbing air humidity and this made the analysis impractical once the grains were agglomerated.

Another factor that changed the research course was the grain size behavior in the early grinding periods. The reached substances grains showed a threshold beyond which there would be no significant variation in the grains size if the grinding time were increased. Then it was decided that the research would proceed at shorter ranges in which the limit would be one hour (the last viable point shown what was discovered and previously mentioned). The grinding periods would then be 15 minutes, 30 minutes, 45 minutes and 60 minutes. In them the components samples are in their raw state.

Another grain size decrease problem occurred during the propellant cooking process, due to the fact that a propellant with smaller grains needs less activation energy to be ignited. Thus, it would probably ignite during the cooking process because of pan heat, endangering those who worked with it.

The first test was to determine the grain size using the SEM technique, analyzing each grinding period sample of each component, totalizing ten samples. These samples, after conditioning and subjected to the microscope, resulted in a pictures sequence that showed each sample under different magnifications (from 42x to 5000x), as shown on Fig. 1.

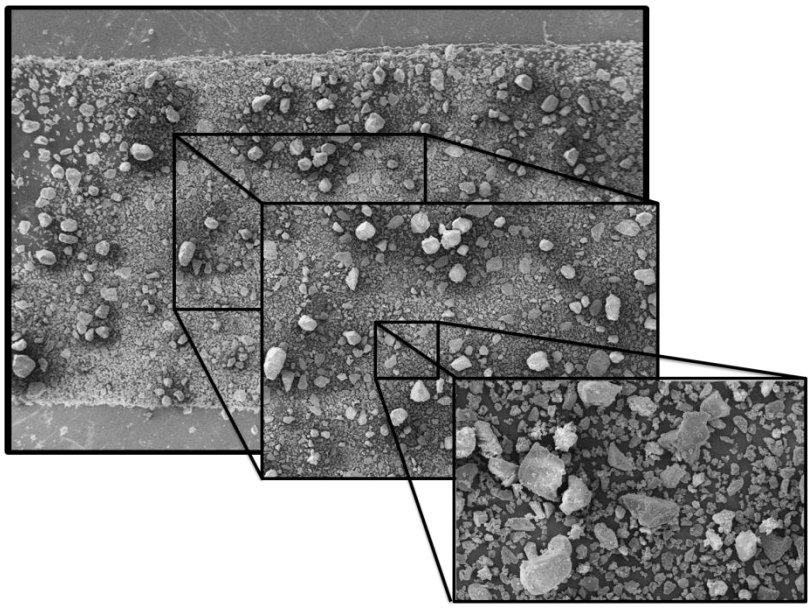

Figure 1. Potassium Nitrate SEM analyses steps

The UTHSCSA ImageTool Version 3.0 was used to measure the grains in each picture. With settings properly adjusted, it was possible to obtain the actual size of each grain in micrometers and find the average grain size resulting from each grinding period.

The following test examined the propellant calorific power through five samples, each representing a grinding period, which was taken into a bomb calorimeter.

All samples had less than 500mg, an equipment requirement [calorimeter IKA], and were burned in an oxygen-rich atmosphere (100% oxygenated) and with a pressure higher than 1 atm.

In order to determine the granulometry influence on propellant's burning velocity, new samples of each propellant were prepared. Each grinding period was represented by three samples of propellant which filled phenolite tubes with a 50mm length, a 25mm internal diameter and a 29mm external diameter.

The fifteen samples, of which five are shown in the Fig.2, were burned in a chapel, capable of absorbing all the released gas. The burning was recorded and each grinding period associated average was calculated.

Figure 2. Burned phenolite examples. Each had a different grinding period propellant burned inside it.

Continuing the analysis of what influences the propellant burning velocity, the cooking time (which the propellant undergoes) was studied. Potassium nitrate does not react under the temperatures reached during cooking. However, sugar does. So, it was studied once the propellant's power is affected.

Samples were cooked until the stage of curing (moment when the propellant is ready) and then cooked for more time, the over cooking time. All samples came from Potassium Nitrate and Sucrose grinded for 30 minutes.

When the propellant reaches the curing point, it is removed from the pan and loaded into the combustion chamber. However, for the test proposed in this section, this point has been exceeded, and a total of eighteen samples were generated from cure point, with a gap of 1 minute between them. These samples are shown in the Fig. 3.

Figure 3. Cooking overtime samples. The top left: 1 minute of overcooking; the bottom right: 18 minutes of overcooking. The ones between have 1 minute more of overcooking.

The last test performed in this sequence analyzed if there was a relation between combustion's residues composition and grinding time. Thus, propellants samples of different grinding periods were again prepared and taken to a laboratory to be burned. Each sample, from a total of 15, was inserted into a crucible and burned in a chapel. Firstly, the ratio between the combustion's remaining mass and the initial mass was calculated. After, these residues were collected and sent for Elementry Analysis, which provides the percentages of nitrogen, carbon, hydrogen and sulfur in the samples. At the Elementry Analysis, the residues are heated to 1200º Celsius until they vaporize. After, the gas passes through a series of metals and fibers to separate the gases that the machine cannot describe. Then, the percentage of nitrogen, oxygen, sulfur and carbon is measured.

3. RESULTS

This section condenses the results obtained from the research and their respective considerations.

The SEM photographs and the ImageTool measurements lead to Fig. 4 and Fig. 5 graphs, which show the potassium nitrate and sugar grain size change when they are subjected to grinding. Table 1 shows the grain size averages obtained for both components in each grinding period.

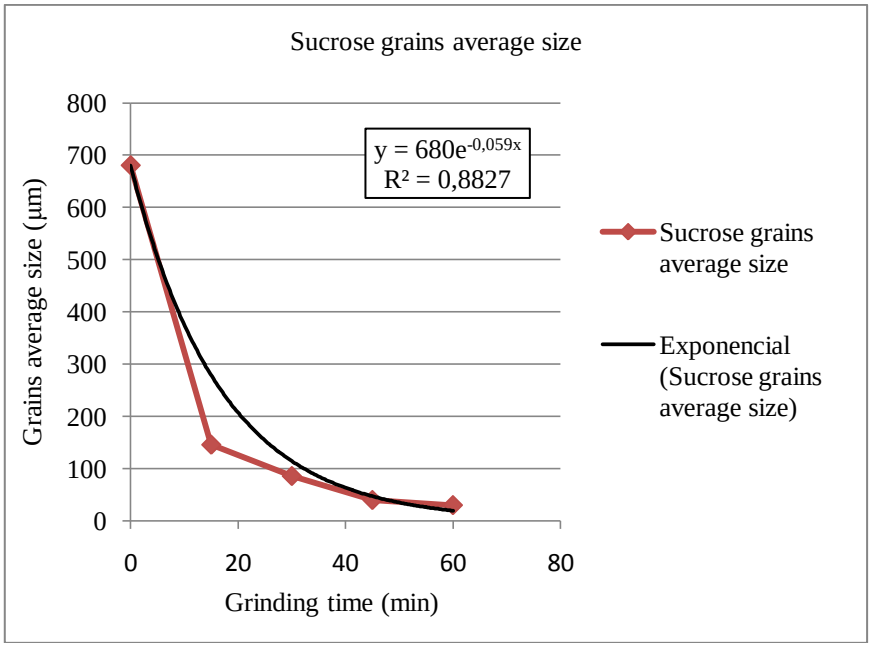

Figure 4. Sucrose grain analysis

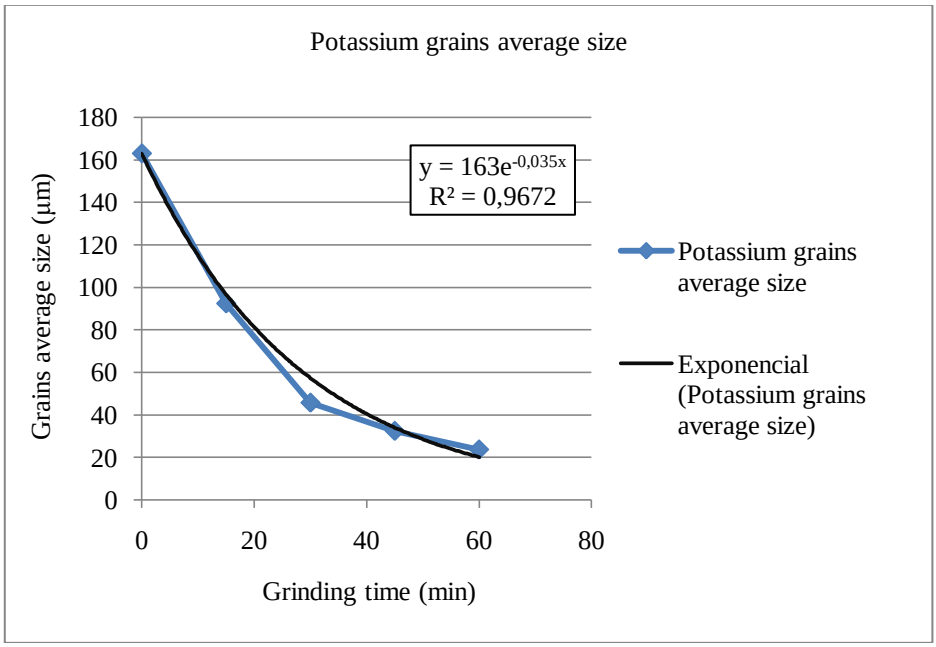

Figure 5. Potassium nitrate grain analysis

Table 1. Grinding time and grain average size analysis

Grinding time (min)	grain average size (µm)	
	KN	SU
0	163,1	680,6
15	92,4	145,3
30	45,7	85,3
45	32,4	39,4
60	23,6	29,6

The curves formed by the obtained data show the predicted asymptotic behavior, proving the initial considerations regarding the grinding time right. From the trend lines shown in Fig. 4 and 5, which were obtained with the 0 to 60 minutes data, it was possible to estipulate how the grain size would decrease with time. Tables 2 and 3 below show, along with this behavior, the asymptotic edge were the grains stop diminishing considerably. At this edge, there is no longer worthy to continue the grinding.

Table 2. Potassium nitrate grain size stipulation

KN												
x	grinding time (min)	0	5	10	15	30	50	55	60	65	90	95
y	theoretical grain size reached (μm)	163,0	136,8	114,9	96,4	57,0	28,3	23,8	20,0	16,8	7,0	5,9
	grain size decrease/initial grain size (%)	16,1	13,5	11,3	9,5	5,6	2,8	2,3	2,0	1,7	0,7	0,6

Table 3. Sucrose grain size stipulation

SU												
x	grinding time (min)	0	5	10	15	30	50	55	60	65	90	95
y	theoretical grain size reached (μm)	680,0	506,3	376,9	280,6	115,8	35,6	26,5	19,7	14,7	3,4	2,5
	grain size decrease/initial grain size (%)	25,5	19,0	14,2	10,5	4,4	1,8	1,3	1,0	0,7	0,2	0,1

As for the high heating tests, Fig. 6 graph shows the heating averages for each propellant type.

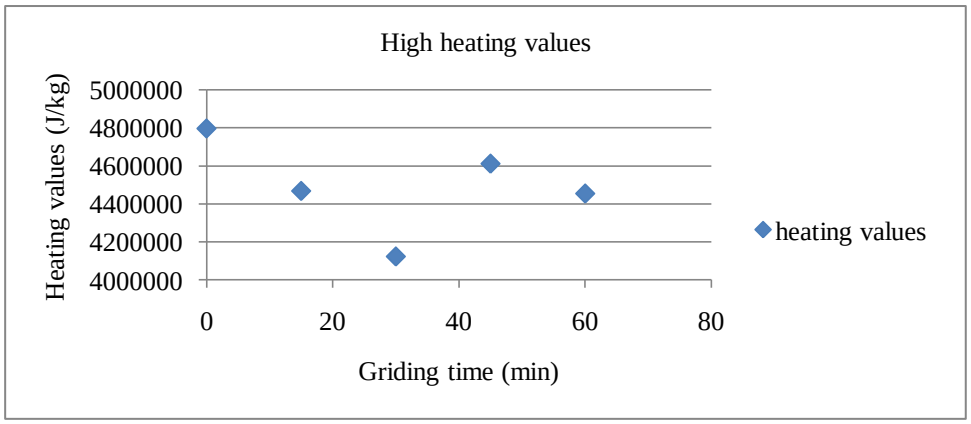

Figure 6. C2000 IKA Calorimeter results

A pattern among these data was not possible to find. A confidence intervals statistic analyses was then used and its result is shown in Fig. 7. It is possible infer that the samples are similar, because their confidence intervals intersect themselves.

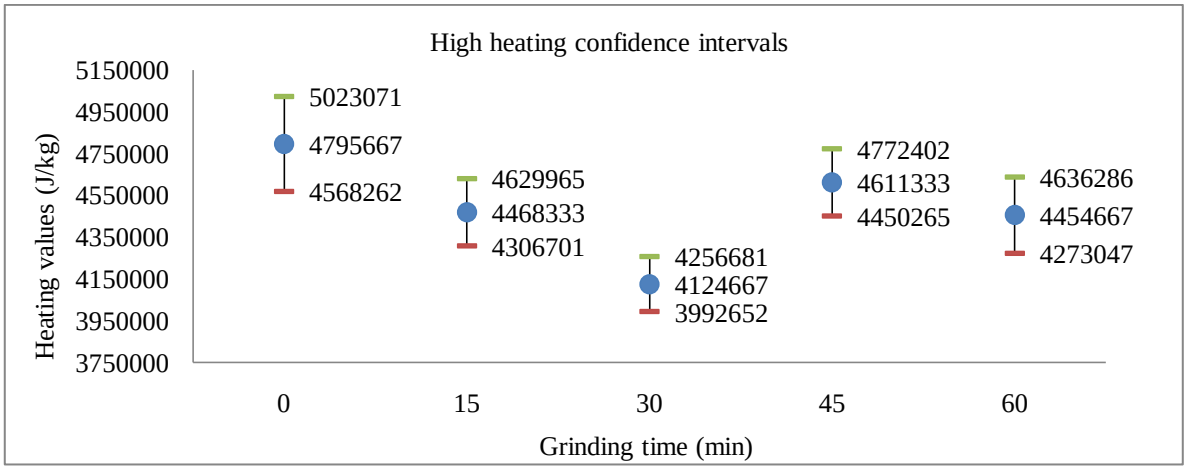

Figure 7. Confidence intervals statistic analysis

This test is conducted in a pure high pressure oxygen atmosphere, which makes combustion easier to occur. Once all samples start from the same point, KNSU, and burn completely because of the atmosphere, the results were similar. The high heating values, then, could not be connected to grinding.

As for the combustion velocity, Table 4 shows the results for the 15 samples. Figure 8 graph, which has the values outlined in the table, demonstrates that the burning time follows the grain size chart profile. The smaller grain size, the faster the burning process occurs.

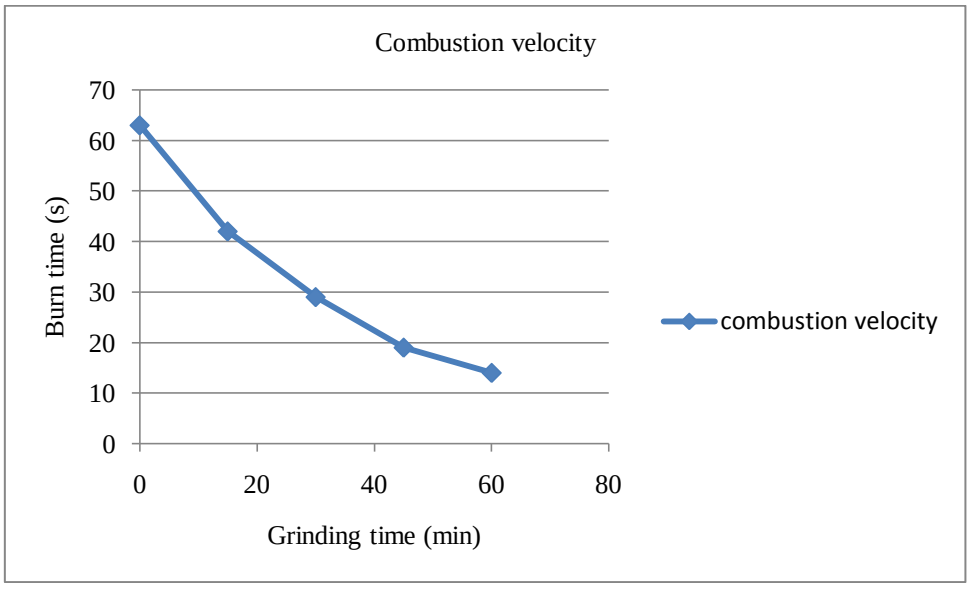

Figure 8. Combustion velocity

Table 4. Combustion time

grinding time (min)	combustion time (s)
0	63
15	42
30	29
45	19
60	14

Along with this analysis, the phenolite tubes tests showed structural implications from combustion velocity. It was noticeable that the tube suffered degradation under the temperature produced by the propellant burn. The longer the burn, more damaged the inner walls would be, compromising the structural integrity. A faster burn would be more interesting once it would not seriously compromise a combustion chamber designed to heat resistance.

The after-curing-cooking-time burn speed influence was evident when the combustion time and propellant mass were compared. As sugar is oxidized during this after-time, part of its calorific power is lost and, therefore, the reaction becomes inefficient, with incomplete combustions, and the chance of not occurring. Such propellant degradation is evident through the ready mixture, in which a darker color takes place. This color indicates calorific properties loss. In addition, samples with longer cooking time took a longer time to burn, as the Fig. 9 graph shows.

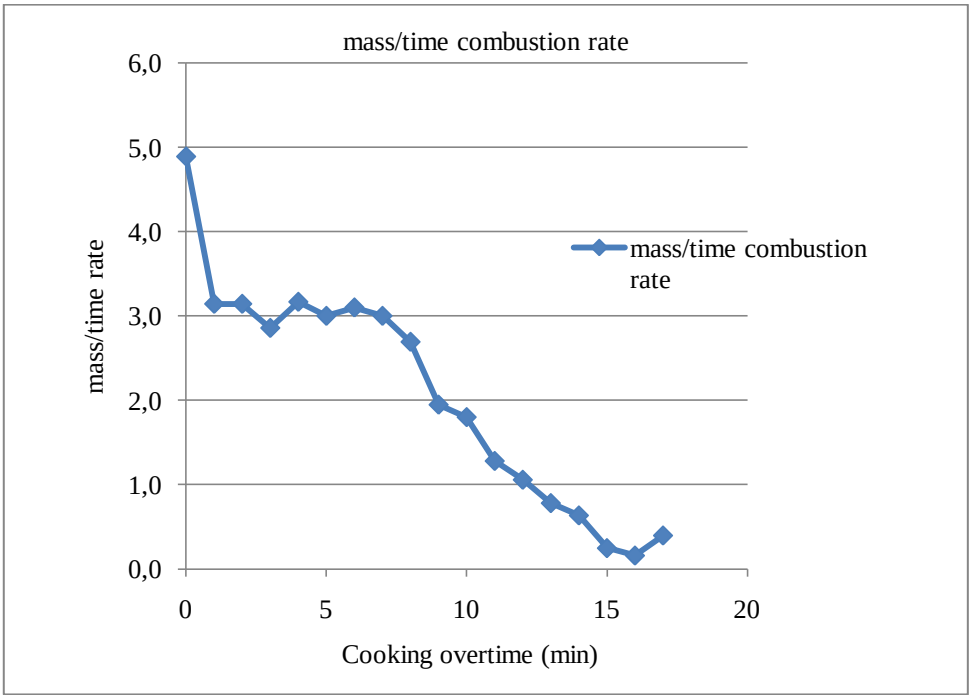

Figure 9. Mass/time combustion rate

During the residues studies, once again it was noticeable a similarity between Fig. 10 curve and the grain size curve. This indicates that the longer the milling time, the lower the generated residues mass is.

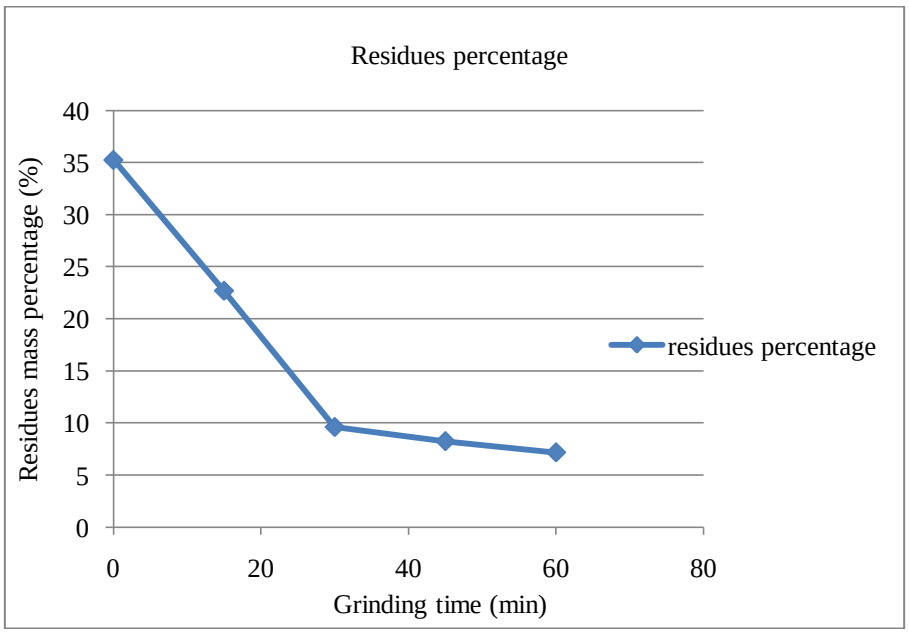

Figure 10. Residues percentage

Besides the percentual residues mass reduction, it was investigated if the particle size would affect its composition. After the Elementry analysis, it was obtained the mean weight percentage of nitrogen, carbon, hydrogen and sulfur contents of each propellant type, shown in Tab. 5.

Table 5. Nitrogen, carbon, hydrogen and sulfur percentage

grinding time (min)	Elements percentage			
	N (%)	C (%)	H (%)	S (%)
0	1,1	7,4	3,2	2,0
15	0,5	5,4	3,1	2,4
30	1,6	6,9	3,4	0,5
45	1,2	7,6	3,4	2,0
60	2,9	10,5	2,2	0,0

It was not found a reasonable patterned among these values and, therefore, it was not possible to associate grinding time with them.

4. CONCLUSION

After analyzing the samples, it is possible to conclude that grinding affects the speed of burning and, consequently, the burning time. The increasing of the milling time, which results in a decrease in the average grain size, leads to the increasing of the burning rate due to the expanded contact area between them (Kuo, 1986).

The cooking time affects the amount of power remaining in the propellant. The longer the duration of preparation, the smaller energy and combustion efficiency remaining, resulting in the inability of the grain to be ignited.

Associated with this, it is possible to see that this study enhances the use of KNSU in rocket motors, as a reaction more efficient can decrease the release of residues and achieve more efficient propulsions.

The main application of this study is to provide design parameters for the combustion nozzles in such a way that allows some control over the burning time, because changing the variables, you can modify the combustion efficiency. This effect is particularly important for engine design with application in assisted takeoff or to limit the acceleration of the rocket in order to protect any internal components.

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6. REFERENCES

Hunter, M. W., 1966, “Thrust Into Space”, Ed. Holt, Rinehart and Winston, New York, USA.

Kuo, K. K., 1986, “Principles of Combustion”, Ed. Wiley, New York, USA.

Monteiro, F.N., Bakkenist, H. and Garcia, V.H.V., 2010, “Moinho de Bolas para Combustível de Foguetes”, 18th International Symposium of Scientific Initiation of University of São Paulo, Brazil.

Sutton, G. P., 1949, “Rocket Propulsion Elements”, Ed. Wiley, New York, USA.

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