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The Influence of Nano-Scale Aluminum on the Energetic Potential of Energetic Materials - Theoretical and Experimental Observations

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Metal powders have been utilized for a long time in energetic materials to improve some specific performance. In recent years, metal particles in energetic materials have been reduced in size since smaller particles, particularly nano-sized, have highly beneficial qualities. These positive aspects are evident in the reduction of ignition and combustion times, resulting in enhanced combustion in volume-constrained systems, improved heat transmission, and increased total thermal energy release. Aluminum is one of the first metals to be used in form of nanoparticles in energetic formulations, with its concentration ranging from a few percent to up to a third of the total composition. In this paper, the effect of applying nano-scale aluminum on the energetic characteristics of explosives, pyrotechnic mixtures, and rocket propellants is observed. The differences in the energetic potential due to varying sizes of aluminum particles (micrometer and nanometer sizes) are shown. For selected energetic materials, the energetic potential during combustion in an inert atmosphere was determined using the method of isoperibolic calorimetry. The theoretical values of the energetic potential for the selected compositions were determined using the thermo-chemical computer code EXPLO5. Numerical modeling using the software package may anticipate the behavior of energetic materials quickly, cost-effectively, and in an environment-friendly manner. Furthermore, the combustion efficiency of the chosen compositions can be assessed by comparing the theoretical and experimental values.

Key words: **nano-sized aluminum, explosives, pyrotechnic mixtures, rocket propellants, energetic potential, calorimetry.**

Introduction

IN recent years, the military industry has intensively applied knowledge from the field of nanotechnology, in order to improve the performance of existing materials, electronics and information systems and to develop new ones that improve the combat capabilities of units on the field. Most of them are designed in combination with conventional and already developed technological processes [1, 2]. The attention of scientists has been especially occupied by the application of nano-sized metals in energetic materials (EM).

Reactive metals are added to EM to enhance the energy and achieve certain desired characteristics. The influence of metal particles is particularly pronounced in explosives, which, by their incorporation, acquire a thermobaric effect. Reactive metals are added to explosives to increase the total energy produced during the explosion. This is achieved by their reaction with the gaseous products of the explosion or oxygen from the surroundings, which amplifies the shock wave. The advantage of their utilization can be simply observed by comparing the burning energy of metals in air (20-30 kJ/g of metal) with the detonation energy of classic explosives (6 kJ/g of explosives) [3]. All this led to the fact that metal particles found their application in energetic materials at the beginning of the 20th century. Many metals, such as aluminum (Al), boron (B), silicon (Si) or magnesium (Mg) show a positive effect on the energetic characteristics of rocket propellants, pyrotechnic mixtures and explosives. Among them, aluminum stood out as the most frequently applied metal in EM due to its energetic potential in the reaction with oxygen (31 kJ/g), availability and favorable price [4].

Recently, there has been a shift towards replacing micrometer metal fuel components in energetic materials with nanoscale counterparts, aiming performance enhancement. Nanoparticles have a higher number of surface atoms compared to micro and macro structures, leading to an increase in the contribution of atoms to the formulation's total energy. However, there is always a thin coating of metal oxide on the surface of these particles, so it is important to know the purity of metal component used in formulations. In addition, energy increase of nano-scaled powders is attributed to large specific surface area, high surface energy and strong surface activity [5-6]. Replacing micro-sized aluminum with nano-sized aluminum led to a decrease in initiation time, a rise in impulse and an enhancement in heat transfer. These changes create new possibilities for modeling the performance of energetic materials by adjusting various factors that influence the functioning of the final product - ammunition. Despite some crucial benefits of nano scale Al, there are reports about some issues which should still be investigated such as high sensitivity to electrostatic discharge (ESD)[6-8], impact [6,9-11], friction [6-8,11], sintering and aggregation due to solid-state diffusion [12-15]. Considering the vast array

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of potential applications of nanomaterials within the security and defense sector, this paper is focused on a single aspect of nano chemistry - the impact of nano-sized aluminum on energetic potential of EMs, which is a crucial factor affecting the functionality and effectiveness of ammunition.

Energetic potential (heat of combustion) is the difference between the energy of material formation and the energy of formation of combustion/explosion products [16]. The amount of heat released during the combustion of energetic mixtures was determined experimentally in a calorimeter bomb, by isoperibolic calorimetry. It is a parameter that is defined as the amount of heat released by the decomposition reaction (combustion) of a certain mass of energetic material in a calorimeter bomb at an operating temperature of 25 ºC [17]. Energetic potential is a quantitative parameter that indicates the heat capacity of energetic materials in an efficient and safe way. This method provides information that directly impacts the usability and efficiency of the compositions being analyzed. This method is crucial for studying new compositions that possess specific effects and for managing energetic materials that have been produced and kept for an extended period. Powdered metals are applied in varying amounts to EMs, which makes them a good choice to compare the impact of changing particle size from micro to nano structure on thermal potential.

Aluminum powder is frequently incorporated in pyrotechnic mixtures, as well as in PBX (Plastic bonded explosives) formulations [18-20], to increase the pyrotechnics energetic output and to obtain thermobaric effects of explosives, as specific increased secondary combustion, and its impact on this effect varies based on its size and shape.

High mass proportion of nano aluminum is not cost-effective in PBX charges, and it may also cause an increase in mixture viscosity. To achieve optimal performance and cost-effectiveness in the final product, it is essential to use a bimodal mixture of aluminum, that is a combination of nano and micro metal fractions. This approach combines nano-sized aluminum for enhanced energy performance and micro-sized aluminum for a reasonable price. Latest research indicates multiple advantages of utilizing nano aluminum in explosives - aluminum particles enhance the heat, temperature, and energy release duration of the explosive product when they react with it. Comparing the explosive properties of RDX with micro and nano-sized aluminum revealed that the heat of explosion is lower for explosives with nano Al compared to those with micro-sized Al, with an addition of 20-40 wt.%. Introducing 30wt.% micro and 5% nano Al raises the heat of explosion by 5.83%. Adding a little amount of nano Al improves the rate of oxidation of micro-Al [21].

Composite solid rocket propellants are heterogeneous mixtures of inorganic oxidizer, such as ammonium perchlorate, metallic fuel components and polymer matrix. For application and stable operation during combustion, it is necessary for the rocket propellants to release large quantities of low-molecular gases of high temperature, whose direction through the nozzle opening provides the kinetic energy required for propulsion. Due to the ability to improve performance, various metals, as Al, are used in the compositions of composite solid rocket propellants. Aluminum does not primarily serve as a fuel component in these compositions, as non-aluminized fuels already have an abundance of fuel components. Even a modest amount of aluminum reduces unstable combustion [22]. Adding aluminum to these compositions greatly alters the internal ballistics properties. In these compositions, the burning rate and heat potential increase, and the law governing the burning rate is altered due to a fall in the pressure exponent [23].

To determine the combustion efficiency of the selected compositions, the theoretical values were calculated using the thermo-chemical computer code EXPLO5. EXPLO5 provides fast prediction of the behavior of energetic materials in a more cost-effective way. Using software programs such as EXPLO 5 saves time and resources and has no adverse environmental impact, making it a significant factor to consider.

Materials and methods

Materials

Three compositions of different types of EMs were evaluated in this study:

-pyrotechnic mixtures, labeled as PIR KAM M and PIR KAM N,

- explosives PBX-M and PBX-N,

- rocket propellants, RP-M and RP-N.

All samples of EM contained of micro- and/or nano-scaled aluminum as a fuel component in their composition. Aluminum with a mean diameter of 5 µm (*Alcan Toyo*) and 70 nm (*SpeedUp International*) were used.

Preparation of the samples

Considering the various EMs being discussed, the preparation methods for each category varied. All samples were manufactured at the laboratory level.

Pyrotechnic mixtures were prepared with aluminum, $KClO_4$ as the oxidant, and Viton A as a binder, in contents given in Table 1. Viton A plays a crucial role in enhancing homogeneity, improving pressing and mechanical properties. Mentioned ingredients weighing 2.0 g in total are prepared in a polyethylene bags for evaluating thermal potential. The bag contains an ignition head with electrodes connected to the bomb cover's poles, as shown in Figure 1.

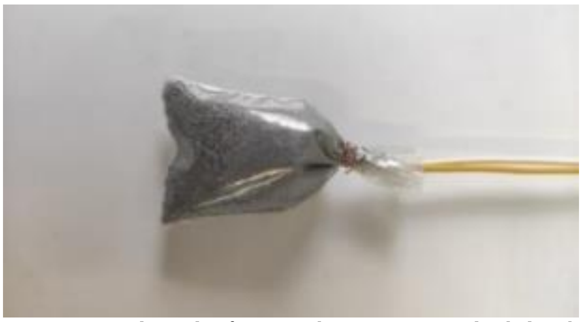

Figure 1. Prepared sample of a pyrotechnic mixture in polyethylene bag

Table 1. Composition of the pyrotechnic mixtures

Sample label	Component mass ratio (%)			
	Al (µm)	Al (nm)	$KClO_4$	Viton A
PIR KAM M	32.38	/	62.86	4.76
PIR KAM N	/	32.38	62.86	4.76

Considering that the samples were produced at the laboratory level to evaluate energetic potential, the full mass of micro- aluminum (sample label PIR KAM M) was replaced with nano aluminum (sample label PIR KAM N). Aside from the varying size of the metal particles, the composition of the pyrotechnic mixtures remains consistent. Figure 2 shows the appearance of the prepared pyrotechnic mixture with micro aluminum.

Figure 2. PIR KAM M pyrotechnic mixture

Two cast PBX formulations were produced using the kneading process. Each of them had identical composition: 14% of binder, 50% of brisance explosive, 10% of ammonium perchlorate (AP) as an oxidant and 26% metal powder, where the first sample PBX-M has the total quantity of aluminum in micron dimensions, while the other one (PBX-N, Figure 3) has bimodal mixture made up of 18% micro Al and 8% of nano Al, Table 2 [24].

Table 2. Compositions of the prepared explosives

Sample label	Component mass ratio (%)				
	Al (μm)	Al (nm)	PETN	AP	PPG
PBX-M	26	/	50	10	14
PBX-N	18	8	50	10	14

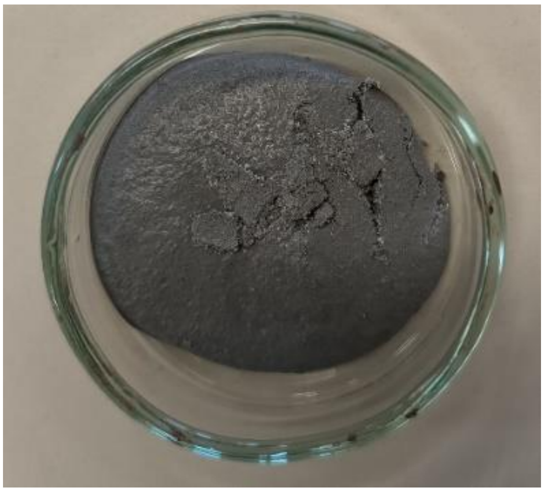

Figure 3. Manufactured sample of PBX-N explosives

The influence of nano aluminum on the energetic potential of composite solid rocket propellants was analyzed on the example of a laboratory prepared composition of small dimensions with realistic geometry, Figure 4. Both samples had the same content – 75% bimodal AP fraction, 24% of polymer binder with curing agent, and only 1% of Al. The first one sample (RP-M) had micro Al, while the other one RP-N had nano Al in their compositions, as it can be seen from the Table 3.

Table 3. Compositions of the prepared composite solid rocket propellants

Sample label	Component mass ratio (%)			
	Al (μm)	Al (nm)	Bimodal AP	polymer matrix
RP-M	1	/	75	24
RP-N	/	1	75	24

Both compositions had identical technological preparation, including component dosage order, mixing mode, and component homogenization duration. The composition was homogenized at the same temperature, at 60°C.

Figure 4. Manufactured sample of composite solid rocket propellant

Characterization methods

Scanning electron microscopy

Nano aluminum, utilized in EMs, is acquired as a raw material based on specific requirements that were monitored prior to utilization. Specifically, it involves establishing the specifications for the shape and size of aluminum, as these attributes directly impact the reactivity of nanoparticles. For this reason, scanning electron microscopy (SEM) was applied for observation of particles quality at various magnifications, using JEOL JSM-6610 LV device.

Isoperibolic calorimetry test

Calorimetry is a technique used to measure the heat content or heat capacity of a substance. It quantifies the heat energy absorbed or released in a physical or chemical process [25]. Calorimetric findings are used to measure the heat capacity of substances, which represents the energy content of fuel, food and other materials.

An isoperibolic calorimeter, IKA C2000 and a calorimetric bomb C5010 were utilized for experimental assessment of the heat release properties of energetic substances. A bomb calorimeter is utilized to measure the heat capacity of energetic materials. It includes a sealed steel chamber holding the sample, submerged in water at a specific temperature. The test sample, with a known mass, is incinerated in a bomb containing an inert gas like argon or nitrogen, at a pressure of 20 bar. The heat produced is then transported to a water jacket and the temperature variation is observed.

Heat capacity is a measure of the amount of heat required to raise or lower the temperature of a body by 1 K [26]. The definition involves the ratio of heat (Q) exchanged between the body and the environment and the change in body temperature (ΔT).

$$Q = \Delta T \cdot C_v \tag{1}$$

Q - calorific value, J;
ΔT - temperature change (difference between final and initial temperature), K;
C_v - calorimeter heat capacity (water equivalent), $J \cdot K^{-1}$.

The energetic potential or heat value of combustion per gram of sample mass (m) is then:

$$Q = \frac{\Delta T \cdot C_V}{m} \quad (2)$$

When testing energetic materials, the heat potential represents the amount of heat that a unit amount of explosive material releases upon complete combustion. For the calorimetry test, the samples need to be prepared as follows: explosives and cast composite rocket propellants need to be crushed before use, unlike pyrotechnic combinations. A mass of 2.0 - 2.4 g of the sample is chopped into stick shapes for testing, as in Figure 5. A single stick of shredded samples is wound around the incendiary wire, with the wire's loose ends secured to the bomb cover's poles, while the remaining portion is positioned around it.

Figure 5. Stick shaped samples of composite solid rocket propellants

Once the samples are prepared, the procedure (Figure 6) for operating the isoperibolic calorimeter is as follows [17]: the bomb is sealed, air is evacuated using a vacuum pump, followed by the introduction of an inert gas.

Figure 6. Analyzing the sample a) Positioning the sample in a quartz container, b) Introducing inert gas, c) Conducting the experiment in the calorimeter, d) Analyzing the acquired results

The bomb has been positioned inside the calorimeter. The measurement is conducted automatically to track the relationship between temperature change and time.

Thermo-chemical computer code EXPLO5

The modeling of combustion parameters was performed in EXPLO5, in isochoric combustion mode for all the observed energetic compositions. Software programs capable of accurately calculating critical characteristics for characterizing and assessing energetic materials have significantly expanded in recent years. One example of such software is EXPLO5, which solely requires input data on the actual density and mass fraction of the individual components in the studied EM to calculate the equilibrium composition and thermodynamic parameters of the product state under specified conditions of volume, pressure, and temperature. Thermochemical data, in conjunction with Chapman-Jouguet theory, enable the calculation of significant performance parameters for energetic materials, including detonation velocity, pressure, and energy. The program uses the equilibrium compositions and thermodynamic characteristics of the state during isentropic expansion to calculate the coefficients of the Jones-Wilkins-Lee (JWL) equation of state. This is accomplished with the integrated JWL subroutine. The program uses thermodynamic parameters and conservation equations to predict the theoretical performance of energetic mixtures that generate gas during constant pressure or constant volume combustion. Using software such as EXPLO5 reduces the amount of experimental work needed in laboratory and test range, leading to improved working safety and less environmental impact.

Results and discussion

Figure 7 shows the SEM images of Al nanopowder. The particles exhibit good quality as they are predominantly spherical and not agglomerated.

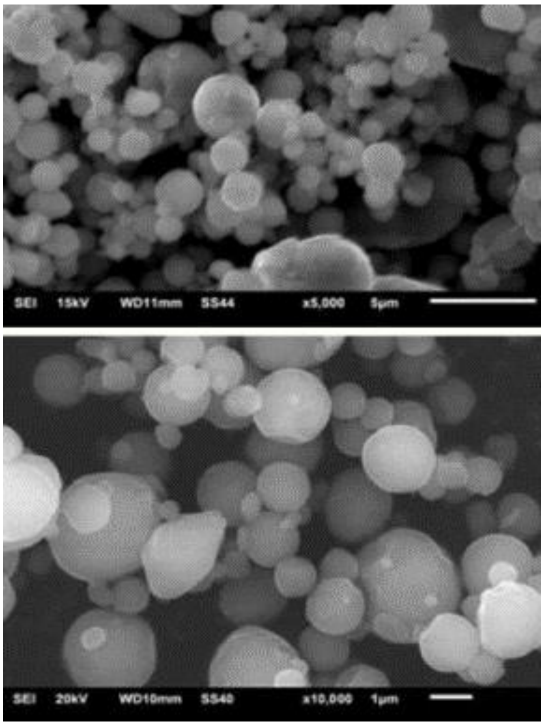

Figure 7. SEM images of nano-sized Al particles

All examined samples are a part of the EM group, yet they serve very diverse purposes in ammunition assemblies. Due to this specific cause, their energetic potentials vary significantly. EXPLO5 does not have the capability to differentiate particle size and consider it as a variable. However, it accepts real densities as input, which vary for compositions containing micro and nano-sized Al. Table 4 presents experimental and calculated data for pyrotechnic mixtures samples. As it can be seen, nano aluminum has greater energetic potential compared to micro aluminum. The disparity in the obtained values was anticipated due to the increased reactivity of nano compared to macro particles. Upon analyzing the findings for a particular pyrotechnic composition, it is clear that there is a change in all parameters when transitioning from micro to nano aluminum.

Table 4. Energetic potential of pyrotechnic mixtures

Sample label	Energetic potential [J·g^{-1}]			
	Density [g·cm^{-3}]	Thermo-chemical code calculation	Experimental value	Combustion efficiency [%]
PIR KAM M	0.5545	10156.1	8412.8	82.8
PIR KAM N	0.6562	10155.7	9114.9	89.8

Metal constitutes 1/3 of the concrete mixture, making it easy to track the changes when transitioning between different metal structures due to its significant mass fraction. The results show that using nanoparticle aluminum significantly impacts energetic potential. Micro aluminum yielded 8412 J·g^{-1}, while the composition with nano aluminum produced 9114 J·g^{-1} (an 8.34% increase). This substantial increase supports the future use of nano aluminum in pyrotechnic mixtures. In addition, the better combustion efficiency is clearly visible. For explosives with high filling masses that require significant amounts of metal powder, it is not economically viable to substitute all micro aluminum with nano Al. The research investigated the impact of nano aluminum on a bimodal mixture of Al. Table 5 shows that replacing only one-third of the total aluminum with nano-particle aluminum raised the energetic potential by 4.6%.

Table 5. Energetic potential of explosives

Sample label	Energetic potential [J·g^{-1}]			
	Density [g·cm^{-3}]	Thermo-chemical code calculation	Experimental value	Combustion efficiency [%]
PBX-M	1.684	9773.18	7015.00	71.8
PBX-N	1.682	9771.93	7339.50	75.1

Furthermore, there is a noticeable pattern of improved combustion efficiency when even a modest quantity of nano Al is added to the explosive composition. Metal powders are incorporated into explosives to enhance the total energy released during an explosion, with a maximum limit of 30%. The explosive charge containing the full quantity of nano aluminum demonstrated increased energy potential and combustion efficiency.

Composite solid rocket propellants analyzed in this research contain a very small amount of Al, so the transition from micro to nano size is not noticeable, which is shown in Table 8. But a minor rise in viscosity gradient, a notable increase in temperature sensitivity and an elevation in pressure exponent values were noted, with no impact on combustion stability when compared to the reference sample [1].

Table 8. Energetic potential of composite solid rocket propellants

Sample label	Energetic potential [J·g^{-1}]			
	Density [g·cm^{-3}]	Thermo-chemical code calculation	Experimental value	Combustion efficiency [%]
RP-M	1.557	5008.54	3801.5	75.9
RP-N	1.555	5006.36	3806.4	76.0

Figure 8 illustrates the graphical representation of the discrepancy between the theoretical and experimental values of the energetic potential. None of the samples achieved the energetic potential calculated by the software. However, a distinct pattern can be detected where the results are more closely aligned with the values when nano aluminum is included.

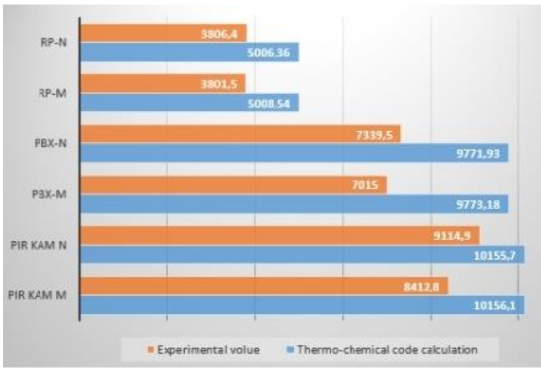

Figure 8. A graphic illustration of experimental and computational values of energetic potential

It had been expected that there would be a discrepancy between the values calculated by the software and those acquired empirically. Combustion efficiency (Figure 9) is an issue, particularly when dealing with high metal content, greater than 35%, typical of thermobaric explosives [27]. One of the reasons for that is the fact that aluminum has a high ignition temperature of 2200 K, which is normally needed for its proper burning. When aluminum burns, it generates heat and produces aluminum oxide (Al_2O_3). Nevertheless, achieving complete combustion of all the metal necessitates maintaining a high temperature in the environment [27-28]. This requirement can be effectively achieved by chemically supporting it through the burning of other oxidizer species.

Compositions such as thermobaric explosives need the combustion process to occur in an air atmosphere to allow metal particles to burn entirely after detonation. The IKA 2000 calorimeter analyzes energetic materials in an inert atmosphere. Compositions including powdered metals are unable to achieve maximal thermal values because combustion mechanisms are not fully completed due to the absence of oxygen.

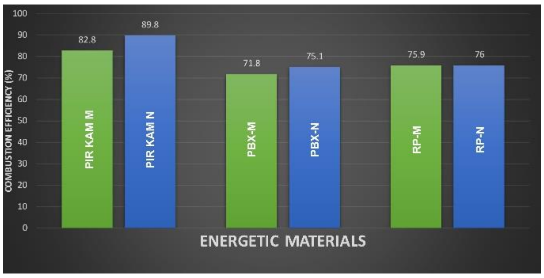

Figure 9. A graphic illustration of combustion efficiency for selected EM samples with micro and nano Al

Composite solid rocket propellants have metals in powder form in smaller amounts compared to TBE. Some studies have reported significantly greater combustion efficiency numbers (86-88%), although these experiments were conducted in an oxygen atmosphere [29] rather than in inert one, suggesting that lower combustion efficiency can be expected in an inert atmosphere. Pyrotechnic mixtures, regardless of particle size, show discrepancies between theoretical and experimental values when tested in an oxygen bomb [30].

Conclusion

The investigation's outcomes indicate a significant impact of nano aluminum on the energetic potential values. The pyrotechnic mixtures had the most significant effect, an 8.34% rise in energetic potential value is found with the presence of nano aluminum in the mixture, which contains up to 34% aluminum as stated in the study. Considering pyrotechnic mixes, it is important to note that the theoretical value is somewhat greater for the composition with micro (PIR KAM M) compared to nano Al (PIR KAM N) because of the actual measured densities of both compositions. Experimental data show that combustion efficiency rises as aluminum transitions from micro to nano size by 7%. The high combustion efficiency is a result of having that 2/3 of the composition act as an oxidant, ensuring that even in an inert atmosphere, there is sufficient oxygen to burn metallic fuel.

In explosives, replacing one-third of the micro-Al with nano Al in a bimodal mixture result in a 4.6% increase in energy potential. When analyzing the combustion efficiency of explosives with 26% Al, the experimental data suggests that the oxidizer (ammonium perchlorate) at a mass ratio of 10% may not provide enough oxygen to fully utilize Al as a fuel. It indicates that air, rather than inert gas, is also required to achieve optimal results. The impact of nano-particle Al on combustion efficiency is noticeable. The composition with micro-Al (PBX-M) has 71.8%, whereas the composition with a bimodal combination of micro and nano Al has 75.1%, resulting in an 3.3% increase.

The composite solid rocket propellants had the lowest impact due to the addition of only 1% nanoparticle aluminum in the mixture, resulting in a 1.3% rise in energetic potential for RP-N compared to the RP-M composition containing micro-Al. A relatively small amount of Al resulted in the combustion efficiency values of the two compositions being nearly the same, 75.9% for the sample RP-M and 76.0% for the sample RP-N. The results obtained for solid rocket propellants suggest that to analyze the impact of varying fuel component particles, like micro and nano Al, compositions with larger mass fractions of Al need to be created.

While there are multiple advantages of transitioning from micro to nano aluminum in EMs applications, there are significant challenges that must be acknowledged. The primary limiting factor in the serial production of nano Al compared to micro-Al is its significantly higher price, which is several times higher. This is particularly crucial as ammunition is a single-use article. Furthermore, there are other issues that still need to be addressed with the use of nano Al. Nanoparticles have a natural tendency to agglomerate. Phase separation of fuel and oxidizer nanoparticles is a significant issue that may impede consistent and dependable performance. An unresolved issue is their susceptibility to electrostatic discharge (ESD), collision, and friction because of their high sensitivity. Furthermore, stability and aging concerns need to be thoroughly investigated through rigorous experimental research. Despite remaining challenges in the implementation of nano Al, there are still numerous benefits - enhanced consistent mixing, lower activation energy for oxidation, shorter ignition delay of metal nanoparticles compared to traditional particles, increased thermal conductivity, quicker burning time, improved mechanical properties, and higher specific impulse.

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Teorijska i eksperimentalna razmatranja o uticaju smanjenja veličine aluminijuma na nano dimenziju na energetski potencijal energetskih materijala

Metalne čestice se već dugo koriste u energetskim materijalima kako bi uticale na poboljšanje nekih specifičnih performansi. Poslednjih godina, čestice metala koje se dodaju u energetske materijale su sve manje veličine. Posebno su interesnantne one nano razmere, jer vrlo pozitivno utiču na pojedine karakteristike. Ovi pozitivni aspekti su evidentni u smanjenju vremena iniciranja i sagorevanja, što rezultira poboljšanim sagorevanjem u sistemima sa ograničenom zapreminom, poboljšanim prenosom toplote i povećanim ukupnim oslobađanjem toplotne energije. Aluminijum je jedan od prvih metala koji je u vidu nanočestica korišćen u formulacijama energetskih materijala, sa masenim udelom u rasponu od nekoliko procenata do čak trećine ukupnog sastava. U ovom radu je analizirana mogućnost primene nanočestica aluminijuma na energetske karakteristike eksploziva, pirotehničkih smeša i raketnih goriva. Prikazane su razlike u energetskom potencijalu usled razlike veličine čestica aluminijuma (mikrometarske i nanometarske veličine). Za odabrane energetske materijale energetski potencijal pri sagorevanju u inertnoj atmosferi određen je metodom izoperiboličke kalorimetrije. Teorijske vrednosti energetskog potencijala određene su korišćenjem termohemijskog računarskog koda EXPLO5. Numeričko modeliranje vrlo je koristan način predviđanja ponašanja energetskih materijala koji proračunava brzo, ekonomično i na ekološki prihvatljiv način. Osim toga, efikasnost sagorevanja izabranih kompozicija može se proceniti upoređivanjem teoretskih i eksperimentalnih vrednosti.

Ključne reči: **nano aluminijum, eksplozivi, pirotehničke smeše, raketna goriva, energetski potencijal, kalorimetrija.**