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Research paper

Morphological Improvement of Aluminium Powder and Its Influence on Processing Parameters for Composite Solid Rocket Propellant

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Abstract: Slurry viscosity is one of the most crucial parameters in the flawless processing of composite solid rocket propellants. Composite solid rocket propellants are used in rockets and missiles for various mission requirements. Currently mission requirements have become very demanding with respect to propellant energetics and performance. Propellant performance and energy can be increased in many ways; increasing the solid loading of a propellant formulation is one of them. At present, formulations containing 86% solid loading are well established with respect to processing and their physical-mechanical-ballistic properties. However, increasing the solid loading beyond 86% becomes quite challenging since the viscosity of the propellant slurry increases many fold and processing becomes rather difficult. The objective of the present work was to process high solid loading composite propellants by reducing the overall slurry viscosity, and consequently attempts have been made by tailoring the morphology of the key solid ingredients. To date, the morphology of the ingredients has received little attention and substantial focussed efforts are required. Hence, in the present work the morphology of one of the key ingredients, fuel aluminium powder, was modified to create highly spherical particles, and formulations were prepared with these spherical particles with stepwise increased solid loading. In summary, it was found that the morphology of the aluminium particles has a profound effect on the viscosity, the viscosity being reduced by 40% in the case of conventional 86% solid loading formulations. This has helped to increase the solid loading substantially, to about 90%, and flawless high density propellants were easily realised.

Keywords: aluminium powder, propellant, viscosity, ballistic property, mechanical property, solid loading

1 Introduction

Solid rocket propellants have an important use in defence as well as space applications. Propellant formulations are generally composed of an oxidizer, a metal fuel and a polymeric binder. The metal fuel *e.g.* aluminium powder, is used in propellant formulations for improving the density, adiabatic flame temperature, characteristic velocity, specific impulse and combustion characteristics of the solid rocket propellant. Aluminium powder with various particle sizes, such as 15 and 6 µm, are used in these formulations for achieving a better packing density and good flow properties *i.e.* the viscosity of the propellant mix. The percentage of aluminium powder is varied from 18-22% in an aluminized propellant composition, and contributes to achieving a high specific impulse, but at the cost of a higher viscosity of the propellant mix [1]. The processing of a solid rocket propellant involves various unit operations, such as mixing, casting and curing. Ease of casting of the propellant in the narrow ports of the rocket motor demands a low viscous of the propellant slurry [2, 3]. There are numerous ways in which the viscosity of the slurry may be reduced, one of these being the introduction of spherical particles in the composition [4]. It has been proposed in the literature [5-7] that particles of spherical morphology should aid in achieving low values of the viscosity, as compared to irregular or elongated particles. This is also evident in yet another study, where by using of particles of sphericity 0.82 instead of particles of sphericity 0.76 the viscosity was reduced three fold for the same solid fraction. Mueller *et al.* [8] studied the flow properties of magmatic suspensions and highlighted that the flow properties depended strongly on both the shape and volume fraction of the suspended particles. They captured the effects of particle volume fraction and particle aspect ratio (representing the shape of the particles) on the viscosity of the suspensions with the help of the Maron-Pierce model, and investigated the impact of particle shape when modelling flows by considering particles of different aspect ratios. They concluded that the effect of particle shape should not be neglected. They studied the effects of suspensions of solid particles on the viscosity in a Newtonian liquid, in which the particle volume fraction and particle aspect ratio were varied systematically. The aspect ratio of a particle is the ratio of its longest diameter to its shortest perpendicular diameter. This is also used to describe the shape of a particle. Particles affect the flow of a suspension because additional energy is dissipated due to fluid–

particle and particle–particle interactions. The particle volume fraction can only be increased to a value of maximum packing fraction in the suspension, above which there is a dramatic, non-linear increase in viscosity. Above this packing fraction the suspension becomes jammed and its flow ceases. Thus, the reviewed literature suggests that the viscosity of a slurry could very well be decreased by modifying the morphology of the particles.

In the present work, an attempt was made to prepare spherically shaped aluminium powder by using an atomization process with quenching in gaseous nitrogen medium, keeping the same average particle size as in the case of an irregular grade [9-13]. Propellants being viscoelastic fluids show thixotropic nature, and preparation of less viscous slurries could be achieved by incorporating such spherical aluminium particles [14]. Various experiments were subsequently carried out increasing the volume fraction of the solid particles in order to study the flow behaviour as well as the ballistic properties of the propellant. The propellant mix prepared using this aluminium has improved the flow properties, which in turn gave better processibility of the propellant.

2 Materials and Methods

2.1 Morphological improvement of aluminium powder (ALP) and experimental design for propellant formulations

Aluminium ingots were used for preparing the fuel ALP, having an average particle size of 15 ±3 μm, by atomization technology. Developmental trials related to improving the morphology of the aluminium powder were carried out at the premises of industrial partners. Usually, molten aluminium is passed through an atomizer held at the top of the atomization chamber to form very fine droplets of molten aluminium. As these droplets pass through the length of the atomization chamber, they become quenched by the ambient air. The fine particles of aluminium obtained in this process are irregular in shape. Hence, in the developmental trials, the atomized molten aluminium droplets were quenched in the presence of nitrogen gas. The aluminium powder obtained using this modification were found to be spherical in shape, but their average particle size was the same as that of the irregular particles obtained by quenching in the presence of ambient air. The spherical aluminium particles obtained had a good size distribution for achieving better packing of the solid powders during the mixing process.

Thus, two types of powders – irregular ALP (I-ALP) and spherical ALP (S-ALP) were obtained for conducting processing experiments of propellant formulations (according to the design illustrated in Table 1) in order to understand the effect of powder morphology on the viscosity of the propellant formulations.

Table 1. Comparison of the important morphological, physical and rheological characteristics of aluminium powders

Measurement	I-ALP	S-ALP	Instrument
Sphericity Circularity Roundness	0.70-0.75 0.65-0.70 0.58-0.60	0.88-0.9 0.8-0.85 0.7-0.75	SEM/2D Image analysis
Tapped density [g/cm^3]	1.350	1.40	V-tap matic-II
Bulk density [g/cm^3]	0.933	0.935	

Other key ingredients used in the propellant formulations were the oxidizer ammonium perchlorate (AP), obtained from Pandian Chemicals, Tamilnadu, and a binder based on hydroxyl terminated polybutadiene (HTPB), obtained from Ms. Anabond Limited, Tamilnadu, curing agent cum cross-linker toluene di-isocyanate (TDI), obtained from Abhang Organics, Pune, and plasticizer di-octyl adipate (DOA), obtained from M/s Surabhi Industries, Pune.

AP of average particle size 300 ±20 μm (size distribution using sieve: 65 ±5% in the range 500-300 μm and 35 ±5% in the range 300-45 μm), HTPB (mol. wt.: 2500-2900, viscosity: 40-65 P, hydroxyl value: 40-45 mg KOH/g), DOA (density at 20 °C: 930 ±20 kg/m^3, acid value: <0.5), TDI (purity: 99% min., RI at 25 °C: 1.566-1.568) were mixed in specified proportions.

A two blade vertical planetary mixer was used to perform ingredient mixing with the binder to form a slurry, and the viscosity of the slurry was measured with the help of a T-spindle of a Brookfield viscometer (Brookfield-D220) at the end of the mixing process. This slurry was then cast into moulds using the vacuum casting technique and the moulds were cured. The cured propellant samples were evaluated for their physical-mechanical-ballistic properties. The mixing operation of the ingredients is a well defined process in which HTPB and DOA is poured into the mixing bowl at a temperature of 45 ±2 °C. After adding the liquid ingredients, mixing is carried out for 15 min at a blade rotation speed of 10-15 rpm. The powder ingredients, such as aluminium powder and AP, are then added one by one and mixing is continued for 15 min, respectively. Subsequently mixing under vacuum (<30 torr) is performed for 15 min before adding the curing agent cum cross-linker to the bowl. After addition of the cross-linker, mixing is continued for 30 min at a temperature of 40 ±2 °C and the slurry is withdrawn after complete mixing. Curing agent addition is made at lower temperatures to avoid instant curing of the slurry in the bowl. After complete mixing, a portion of the propellant slurry is used for viscosity measurement at 45 °C, and the rest is sent for casting followed by the curing process. Curing of the propellant slurry is performed at 60 °C for 120 h.

2.2 ALP characterization

The physical characteristics of the powders I-ALP and S-ALP were analysed using a scanning electron microscope (SEM) VEGA3-TESCAN at 500X magnification, and a Malvern 3000 Mastersizer V3.81 particle size analyser (based on the light scattering principle, with a Mie scattering model) in the wet mode using water as the dispersion medium. Their morphologies were studied by evaluating the circularity of the particles with a 2D image analyser based on the ISO 9276-6 standard. The tapped and bulk densities were measured using a Tapped density meter (V-tap matic-II). The results are listed in Table 1. The flow properties of the powders were investigated using a Powder-Pro A1 and a FT4 powder rheometer.

2.3 Propellant slurry characterization

The viscosity of the propellant slurry was measured using a T-C spindle of a Brookfield viscometer (Brookfield- D220).

2.4 Cured propellant characterization

The physical-mechanical-ballistic properties were evaluated for the cure propellant samples. The physical propertiesof the propellant e.g. density, was evaluated using a gas pycnometer (Thermo-scientific). The mechanical properties were measured with a Universal Testing Machine (Tinius-Oilsen, Model H25KF) and the ballistic properties, such as burning rate, were measured with an acoustic emission strand burner.

3 Results and Discussion

3.1 Powder morphology

Images of the two aluminium powders (I-ALP and S-ALP) investigated in this study are shown in Figures 1 and 2 respectively. Powder I-ALP consisted of non-spherical and elongated, irregularly shaped particles. In comparison, powder S-ALP had good particle sphericity and a small number of elongated particles. The particle size distribution of the aluminium powders is shown in Figure 3, and indicates a narrow particle size distribution in the spherical aluminium powder S-ALP.

Figure 1. SEM image of I-ALP

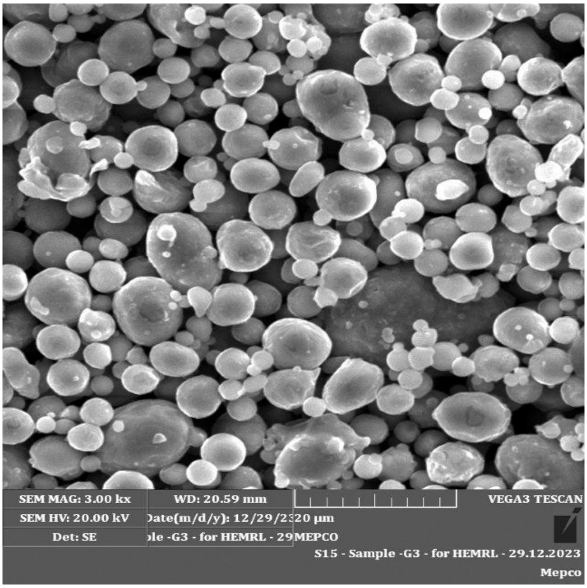

Figure 2. SEM image of S-ALP

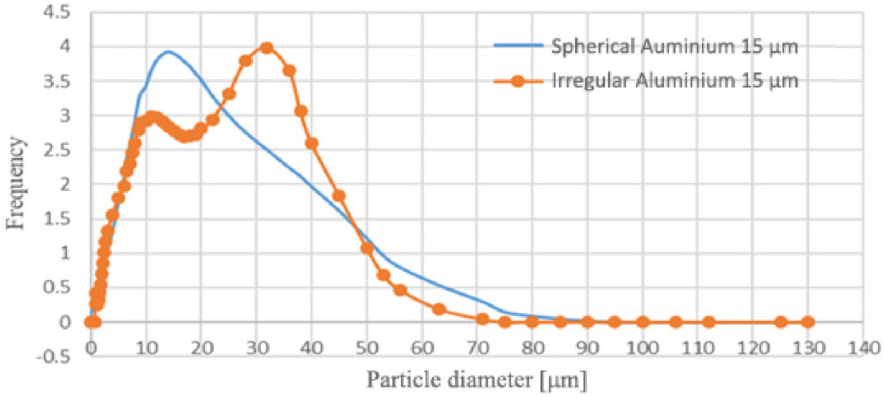

Figure 3. Particle size distribution

3.2 Processing of the propellant formulations

3.2.1 Type of formulation – low burning rate

3.2.1.a Viscosity effects

Formulations SRP01, SRP02 and SRP03, represented the low burning rate type of propellant, are shown in Table 2. The targeted burning rate was same for all of these formulations, in order to see the effect of the aluminium powders alone (I-ALP and S-ALP) on the respective viscosity values; all other ingredients with their individual wt.% were kept the same. Viscosity represents the flow behaviour of the propellant slurry. The viscosity data of these propellant formulations is presented in Table 3, Figures 4 and 5. It is very evident from Table 3, that the viscosity of formulation SRP02 containing S-ALP is 40% less than SRP01 (I-ALP) and the same trend is shown in the viscosity build-up values. To study the repeatability and its scale-up effect, another batch (SRP03) of propellant was prepared with a batch size of 60 kg and its viscosity values were compared with SRP01. The scale-up effects were found more promising as the viscosity was reduced by almost 50% as compared to the viscosity of propellant SRP01 specified in Table 3.

Table 2. Experimental design

Type of formulation	Formulation	Batch size [kg]	Solid loading [%]	Binder [wt.%]	Oxidiser [wt.%]	I-ALP [wt.%]	S-ALP [wt.%]	NCO/OH ratio
Low burning rate	SRP01	5	86	14	68	18	–	0.735
	SRP02					–	18	
	SRP03	60						
High burning rate	SRP04	5				18	–	
	SRP05					–	18	
High solid loading	SRP06		88	12			20	0.730
	SRP07		90	10	69		21	0.725

Table 3. Viscosity data of the propellant slurry

Viscosity [P]	SRP01	SRP02	SRP03
– End of mixing	8320 (at 41.8 °C)	5440 (at 42.2 °C)	4480 (at 41.2 °C)
– after the period at 45 °C [h]:			
– 1	9600	6400	5760
– 2	10560	7360	6400
– 3	11520	8960	8000
– 4	14400	11200	–

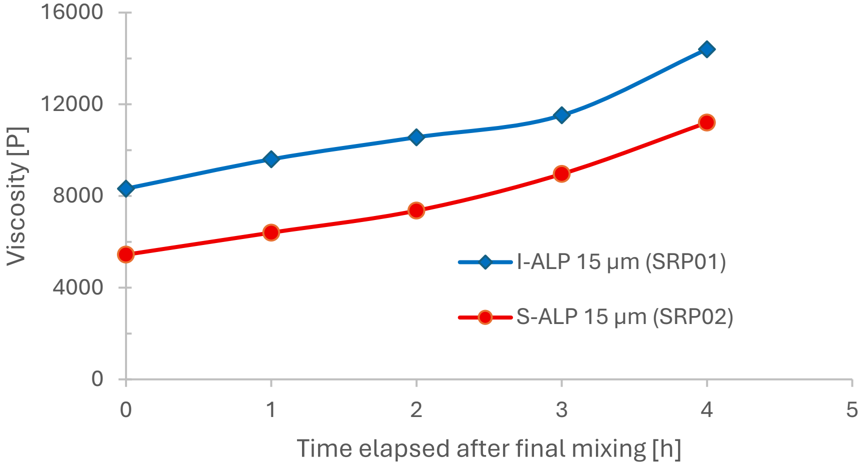

Figure 4. Viscosity comparison of SRP01 and SRP02

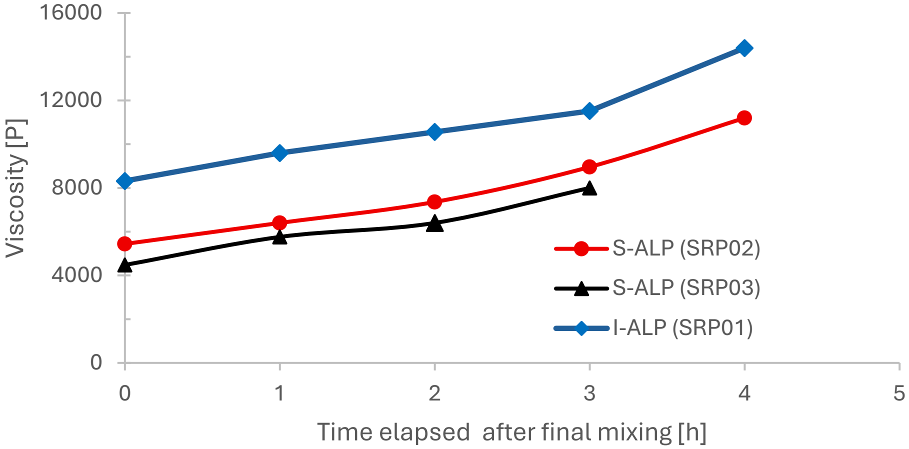

Figure 5. Viscosity comparison of SRP01, SRP02 and SRP03

3.2.1.b Evaluation of ballistic and mechanical properties

Characterization of solid propellants is crucial for rocket performance. This includes ballistic and mechanical property determination, such as burning rate of the propellant and tensile strength – elongation – modulus, respectively. These properties are listed in Table 4, which shows that the incorporation of S-ALP has not compromised on achieving the targeted ballistic and mechanical properties, and is meeting the specifications quite well.

Table 4. Ballistic and mechanical properties

Properties	SRP03	SRP02	SRP01
Strand burning rate (Spec: 6.7 ±0.2 mm/s)	6.66, 6.55, 6.62, 6.62, 6.59 (Average: 6.60)	6.90, 6.90, 6.82, 6.85, 6.89, 6.86 (Average: 6.87)	6.72, 6.81, 6.64, 6.69, 6.70, 6.78 (Average: 6.72)
Tensile strength (Spec: >5 kgf/cm^2)	7.06	7.62	7.09
% Elongation (Spec: >30%)	35.43	49.04	45.29
Young's modulus (Spec: 30-50 kgf/cm^2)	38.47	36.09	38.22

3.2.2 Type of formulation – high burning rate

3.2.2.a. Viscosity, ballistic and mechanical properties of propellant

The effects of the morphology of the S-ALP was also predominantly visible in the high burning rate formulation. It is once again evident from Table 5 that, by replacing I-ALP with S-ALP in the high burning rate composition, the viscosity has been reduced by 30% and the ballistic and mechanical properties are also following specification.

Table 5. Viscosity, ballistic and mechanical properties of propellants

Properties	SRP05	SRP04
Viscosity [P]:		
– End of Mixing	15680 at 49.1 °C	22700 at 44.1 °C
– after 1 h at 45 °C	20800	27200
Strand burn rate (Spec: 20 ±1 mm/s)	20.26, 20, 20.09, 19.98, 20.10, 20.30 (Average: 20.12)	20.62, 20.21, 20.78, 20.86 (Average: 20.61)
Tensile strength (Spec: >5 kgf/cm^2)	12.54	14.32
% Elongation (Spec: >25%)	32.16	26.57
Young's modulus (Spec: 30-50 kgf/cm^2)	43.9	47.7

3.3 Type of formulation – high solid loading

3.3.1. Evaluation of viscosity, ballistic properties and mechanical properties of the propellant batches composed of I-ALP and S-ALP

As was evident from Sections 3.1 and 3.2, the use of S-ALP instead of I-ALP was very promising in terms of viscosity reduction. This advantage was used further to stepwise increase the solid loading of the formulations. Thus two different modified propellant formulations were prepared with high solid loading (88% solids – SRP06) and (90% solid loading – SRP07) using S-ALP. The main objective of the high solid loading propellant was to obtain improved performance from a given rocket motor. The achieved properties of the propellants are presented in Table 6. By incorporating S-ALP into the compositions, it was possible to increase the percentage of solids in the compositions within the processable viscosity of the slurry, and there was a substantial gain in the density.

Table 6. Properties of high solid loading propellant

Properties	SRP06 (88% solid) Batch size: 10 kg	SRP07 (90% solid) Batch size: 10 kg	SRP03 (86% solid) Batch size: 60 kg
Viscosity [P]:			
– End of Mixing	6080 (at 42.8 °C)	16640 (at 45.1 °C)	4480 (at 41.2 °C)
– after the period at 45 °C [h]:			
– 1	7680	22400	5760
– 2	10240	25280	6400
– 3	12160	27520	8000
– 4	14720	30720	–
Density [g/cm³]	1.810	1.846	1.760

3.3.2 Calculation of the maximum packing fraction and model application for viscosity prediction

In the present study, the Maron-Pierce model has been used to calculate the maximum particle fraction, as given in Equation 1 [8].

$$k_r = \left(1 - \frac{\varphi}{\varphi_m}\right)^{-2} \tag{1}$$

where k_r is related to viscosity, φ is related to actual packing fraction of particles and φ_m is related to maximum packing fraction.

This model is applicable since the aspect ratios of I-ALP and S-ALP particles falls between $0.04 \leq rp \leq 22$. Furthermore, the viscosities, in this study, have been measured with the T-C spindle whose shear rate is ~1 s^{-1}. Hence the measured viscosities (normalized by the binder viscosity) are equated with the relative consistency k_r. By applying this model, the value of the maximum packing fraction for S-ALP was found to be 0.92, while that for I-ALP was 0.88. Thus, it is evident that S-ALP particles can be better accommodated in the slurry as compared to irregular particles. Considering these maximum packing fraction values, the modified Krieger-Dougherty model [11] was applied to calculate the viscosity values of high solid loading formulations as shown in Figure 6. The modified Krieger-Dougherty model (Equation 2) was used since it takes into account the effect of particle shape on the viscosities of the solid-liquid suspensions.

$$\eta = \eta_0 \left(1 - \frac{\varphi f}{f_c}\right)^{-2.6 f_c} \tag{2}$$

where η and η_0 are the viscosity at a particular and zero shear stress, f and f_c are the actual and maximum volume fraction of solid in the suspension.

This model predicted viscosities that were almost twice the observed values and hence a coefficient of 0.5 may be added to the model to predict viscosities closer to the observed ones.

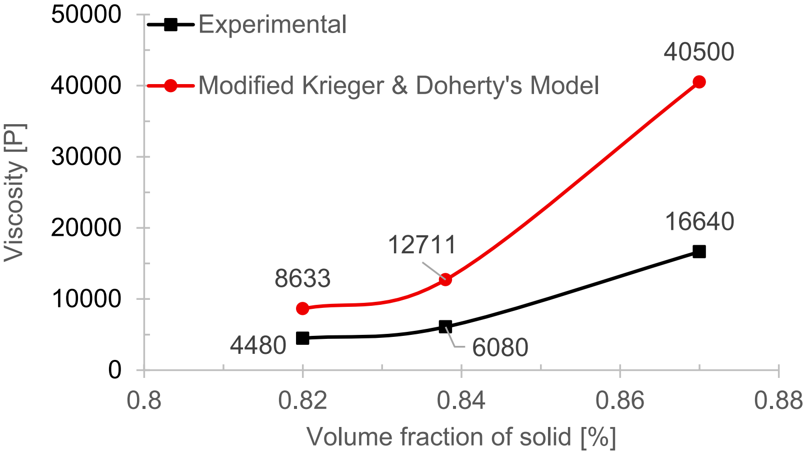

Figure 6. Comparison of experimental and theoretical viscosity results for SRP03, SRP06 and SRP07

4 Conclusions

Morphology improvement in the case of aluminium powder has contributed significantly to the reduction of the viscosity of propellant slurries. Even in the case of a high solid loading propellant, the achieved viscosities are less than the upper limits of castable viscosities (>20000 P). This helps to increase the propellant density of the composition by almost 5%, which is a substantial gain. The following are the conclusions from the present study:

- The effect of the sphericity of spherical aluminium powder, S-ALP, is greater than its particle size distribution on the viscosity values in spite of there being more fines contents in it. This is clearly understood from the viscosity data of the propellant slurry made by incorporating S-ALP. Hence, the effects of the morphology of the S-ALP overcome the size distribution due to less interlocking between the particles of S-ALP than is the case with the irregular one, I-ALP.

- The viscosity of the low burn rate propellant formulation (86% solid loading) containing S-ALP was 40% less than the formulation containing I-ALP.
- The effect of the morphology of the S-ALP was also predominantly visible in the high burn rate formulation (86% solid loading), since the viscosity had been reduced by 30% by replacing I-ALP with S-ALP.
- The incorporation of S-ALP into the compositions helps to increase the percentage of solid loading into the compositions. A formulation with 88% solid loading exhibited a quite low viscosity of 6080 P at 42.8 °C, while a 90% solid loading formulation exhibited a viscosity of 16640 P at 45.1 °C. These viscosity levels are less than the castable viscosity limit of 20000 P, leading to an enhancement of the energetic performance of the propellant formulation.
- By applying an available literature model, the value of the maximum packing fraction for S-ALP was found to be 0.92, while that for I-ALP was 0.88. This clearly indicates the benefit of adopting S-ALP in propellant compositions.

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Authorship contribution statement

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Pallav Jain:	foundations, methods, performing the experimental part
Brijesh Kumar:	methods, performing the experimental part
Suraj Nayak:	conception, foundations
Pramod Negi:	conception, foundations
Mukesh Jain:	other contribution to the publication
Nellutla Pandrinath Rao:	other contribution to the publication
Anil Prasad Dash:	other contribution to the publication

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