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An alternative approach to improve burning rate characteristics and processing parameters of composite propellant

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ABSTRACT

Modification in burn rate of composite solid propellant has become a necessity of a mission. Burn rate can be modified by various mean viz. (i) tailoring the ammonium perchlorate (AP) particle size, (ii) use of nano-sized particles and (iii) incorporating burn rate modifiers. Literature discusses about various burn rate modifiers. Out of these, Iron oxide (Fe_2O_3)/IO is the well known burn rate enhancer. Here a systematic study was carried out by undertaking experiments at varying levels of IO in composite propellant compositions. This paper attempts to understand the effect of IO content and its specific surface area on burn rate characteristics of composite propellant. This study also attempts to find out alternative to ultrafine AP and nano burn rate enhancer to account for high burning rate of composite propellant along with reduced slurry viscosity and longer pot life for easy processing. As ultrafine AP and nano burn rate enhancers have their limitations in terms of (i) high end of mix (EOM) propellant slurry viscosity, (ii) difficulty in propellant processing, (iii) less reproducibility in attaining the similar particle size in each batch results in lesser repeatability in ballistic properties of propellant, (iv) hazards involved in size reduction, (v) limitations in handling, storage and shelf life and also (vi) the higher cost of nano particles. Therefore an extensive experimental study was performed to achieve this objective. In this study, we incorporated two different grades of IO(A) and IO (B) in composite propellant compositions. The average particle size of both grades of IO i.e., A and B are of ~1 μm. But the specific surface area of IO (B) was about 15 times more than IO (A). The large difference in specific surface area of both IO was due to difference in the manufacturing process. During the manufacturing of IO, the calcination temperature plays very important role in deciding the specific surface area. High specific surface area is obtained if calcination is done at very high temperature (>1773 K). Burning rate measurements were carried out. It was observed that IO is a good burn rate enhancer. Initially, the burn rate increased with the increase in % of IO. But after that only a marginal enhancement in burn rate was observed. It was noticed that though both grades of IO are effective burn rate enhancer but IO(B) was 30% more effective than IO(A). Also it was found that IO(B) is an alternative to ultrafine AP and nano burn rate modifiers. This IO(B) was further used to develop the propellant compositions with high burn rate without incorporating ultrafine AP and nano particles. Viscosity measurement and mechanical properties determination revealed that both the IOs did not much adversely alter the processing characteristics and mechanical properties of propellant.

1. Introduction

Composite propellant is the most important class of solid rocket propellant. It is most commonly used in strategic, tactical and rocketry system to deliver the required thrust to the missile/rocket. Design and operation of a rocket motor depend upon burn rate of the propellant. The knowledge of burn rate thus becomes an important condition for a successful design of a solid rocket motor. As the burn rate is exponentially dependent on the pressure, hence the combustion chamber pressure and pressure index have been found to be the most important design parameters. All the same, the designed burn rate is not always available with the selected propellant, at a selected combustion chamber pressure. Due to varied range of applications, tailoring of burn rate characteristics of a composite solid propellant is most sought after, to suit the mission requirements [1]. The burning rate of composite solid propellant is dependent on several parameters like: solid loading,

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oxidizer, oxidizer's particle shape, oxidizer's particle size distribution and burn rate modifiers etc. [2–6].

The modification in burning rate of composite propellant are usually achieved by right selection of particle size and percentage of ammonium perchlorate (AP) which is the most common oxidizer for composite propellant. However the designed mechanical strength and processing characteristics get affected by these. Embedding metal wire or staples in the propellant also allows modification of burn rate. But the logistics and the other complexities have desisted selection of this method as an attractive method of modification. The burn rate of a composite propellant are routinely being modified by addition of small amount of burn rate modifiers to propellant composition. In fact, this method has been found to be the best and the most effective method.

For increasing the burn rate, Iron oxide (Fe_2O_3), Copper oxide (CuO), Copper chromate ($Cu_2Cr_2O_5$) and Manganese dioxide (MnO_2) are commonly used. Iron oxide is the most common burn rate enhancer [7–17]. Lots of research has been carried out to understand the mechanism of burn rate modifier so as to better tailor the burn rate of composite propellant [18–19]. However, upon perusal of literature, it becomes clear that the catalytic effectiveness of the various metal oxides is often contradictory among the various studies conducted with composite propellant. A major contributing factor in explaining this variation is undoubtedly, the poor defined and often varying, propellant composition, AP particle size distribution, morphology and particle size of the transition metal oxides. To have the significant enhancement in burn rate, nano size transition metal oxides, aluminium powder [20–28] are also being used in composite propellant. But their incorporation has limitations in terms of availability, handling, storage, cost, processing difficulty and also hazards.

This paper reports a series of experimental studies performed on composite propellant with iron oxide (IO) of two different trade source. Both the IO were having similar particle size but different physical parameters. In current study, the authors limited themselves to aluminized composite propellant. The burning rate characteristics of AP/Al/HTPB composite propellant containing various content of IO were investigated. An effort was made to obtain detailed experimental data on the burning rate characteristics to understand the role of physical parameters especially specific surface area of IO. A new approach to account for the enhancement in burn rate with reduced EOM viscosity and longer pot life was suggested in the current study.

The main aim of this paper was to find out an alternative mean of enhancement in the burn rate of composite propellant without adversely affecting the processing characteristics, mechanical properties and also cost consideration. For this purpose experimental studies were carried out, which were further presented in this paper. Based on experimental investigations a new propellant composition was developed to fulfil high burn rate requirement without use of ultrafine AP and nano-sized burn rate enhancer. At every stage of development, our objective was to remove a variable (i.e., ultrafine AP) from the composition. To our knowledge this is the first systematic study where the aim was not to just study iron oxide as burn rate enhancer but to find out its suitable source which could be as effective as nano burn rate modifiers. At the same time its incorporation should remove ultrafine AP from propellant composition to improve processabilty, reproducibility, cost effectiveness, availability, and safe processing.

2. Experimental

2.1. Materials

Ammonium perchlorate (AP) was procured from M/s Pandian Chemicals (purity>99%) and used as bimodal distribution having average particle size 300 and 60 µm and as trimodal distribution with average particle size 300, 60 and 6 µm. Aluminum powder of average particle size 15±3 µm, was procured from Metal Powder Company, Madurai (India). Iron oxide (Fe_2O_3) was procured form Aldrich chemicals and BASF chemicals. In this study these IOs are referred as IO (A) and IO (B) respectively. The binder consisted of hydroxyterminated polybutadiene (HTPB, purity 99%, OH value 40–50, moisture 0.15%) purchased from ANABOND, Di-octyl adipate (DOA, ester content 99%, saponification value 303±3, moisture 0.5%), purchased from Subhash Chemicals, n-butanediol(nBD), purchased from M/s Spectrochem Chemicals, Trimethylolpropane (TMP), purchased from M/s Chemsworth Industries, Pyrogallol and toluene diisocyanate (TDI, purity 99%, RI at 30 °C is 1.565–1.567), purchased from Bayers Chemicals.

Table 1
Physical parameters of iron oxide.

Parameter	Fe_2O_3 (IO)	
	A (Aldrich)	B (BASF)
Fe%	68.2	65.5
Specific Surface area m^2/g	4.49	71.25
Tap Density g/cm^3	0.803	0.135
Bulk Density g/cm^3	0.646	0.107
Average Particle size, µm	0.68	1.43

Table 2
Propellant composition.

Composition No.	Wt%				
	Binder	AP	Al	IO(A)	IO (B)
C1(Ref.comp.)	16	67	17	–	–
C2	16	67	17	0.5	–
C3	16	67	17	1.0	–
C4	16	67	17	1.5	–
C5	16	67	17	–	0.5
C6	16	67	17	–	1.0
C7	16	67	17	–	1.5

2.2. Propellant formulation

Both the IO samples were analysed first for Fe-content, specific surface area, average particle size and bulk density. Physical parameters of IO are shown in Table 1.

To examine the effect of IO(A) and IO(B) on the burning rate characteristics, seven numbers of composite propellant compositions were formulated. The binder consisted of HTPB as hydrocarbon fuel, DOA as plasticizer as a processing aid, nBD as chain extender and TMP and pyrogallol as crosslinkers. Chain extender and cross linkers were used to impart required mechanical properties to propellant. Binder was cured with TDI. AP as solid oxidizer and Al as metallic fuel were used in propellant compositions. AP, Al and Binder composition were taken uniform in all propellant formulations. The average particle sizes of AP were of 300 µm and 60 µm and they were used in the weight% ratio of 3:2. The detailed propellant compositions are listed in Table 2.

The IO (A and B) were added in formulation in parts. IO was added as 0.0 to 1.5% at an increment of 0.5%. All the solid ingredients (AP, Al, IO) were first stored in an oven at 333 K for 24 h to remove the moisture before using them into propellant compositions. The requisite quantity of ingredients was weighed properly and mixed in 15 kg batch size in a vertical planetary mixer using standard procedure for composite propellant processing. Vacuum was applied to remove trapped gases from the propellant slurry during mixing. The temperature of slurry was maintained to 328 K during mixing by circulating hot water in mixer bowl

jacket. This decreases the viscosity of propellant slurry and helps in the removal of trapped gases to ensure the propellant to be free from voids and flaws. The propellant slurry was cast under vacuum [29] as tubular grain and cured for 120 h at 333±2 K in water jacketed oven.

2.3. Characterization method

Specific surface area was determined by BET. Average particle size of AP (60 µm) was determined by sieve analysis method and for ultrafine AP (6 µm), it was determined by laser based CILAS particle size analyser; Model 1064L, France by Wet method. Bulk density was measured by Tap Density Test Apparatus. % Fe was calculated by chemical analysis method and also determined by using ICP-AES technique, instrument Ultima 2000 Horiba Yvon, France, in presence of Argon gas as a plasma generator. The AP received from the trade was of average 300 µm. The reduction of AP particle size was done in Pulverizing mill (Hammer Mill) and fluid energy mill to obtain average particle size of 60 µm and 6 µm, respectively. The end of mix (EOM) viscosity and viscosity build up of propellant slurry was measured in Brookfield viscometer, model HBT dial type, by inserting a T-C spindle at a rotating speed of 2.5 rpm at predetermined temperature. The density of cured propellant was measured with a Mettler density kit, which works on the Archimedes principle with toluene as fluid. The mechanical properties of cured propellant samples were evaluated using dumbbells using ASTM Standard D638 at crosshead speed 50 mm/min 300 K on a Housefield testing machine.

2.4. Burning rate determination

The cured propellants were cut into 6 mm × 6 mm × 150 mm strands for further measurements of burning rate. Propellant strands are conditioned at a temperature of 300 K for 24 h in an oven, before conducting the experiments. The experimental investigation of solid strand burning rate (SSBR) was performed using acoustic emission technique [30–31]. The methodology involved combustion of propellant strands with nichrome ignition wire in a nitrogen pressurized steel bomb. Perturbations caused by deflagration of strands were sensed by a piezoelectric transducer (200 KHz) in conjunction with an oscilloscope through water medium. The burning rates were computed from the time that was recorded for the trial conducted for each sample. SSBR was determined at five different pressure (P) i.e., 3, 5, 7, 9 and 11 MPa. Experiments were repeated to obtain five burning rate readings at each pressure, which established the repeatability of the burning rate values. The calculated average accuracy of burn rate measurement in acoustic emission technique was ±2%. Results of entire burning rate measurements were corresponding to an initial temperature 300 K. The pressure index (n-value) was calculated using SSBR data using Vielle's law

$$r = aP_c^n$$

Where

r = Burn rate of propellant
a = Temperature coefficient
P_c = Chamber pressure
n = Pressure index

3. Result and discussion

3.1. Burn rate

Seven different samples of propellant compositions based on HTPB/AP/Al/IO(A)/IO(B) were formulated. Burn rate (BR) was determined at five different pressures for all the compositions. Five tests were conducted at each pressure and averages of these tests were plotted as Burn Rate(r) v/s Pressure (P) graphs. Propellant composition C1 i.e., without IO was considered as reference composition. Burn rate of other compositions with IO (A) and IO (B) were compared with reference composition.

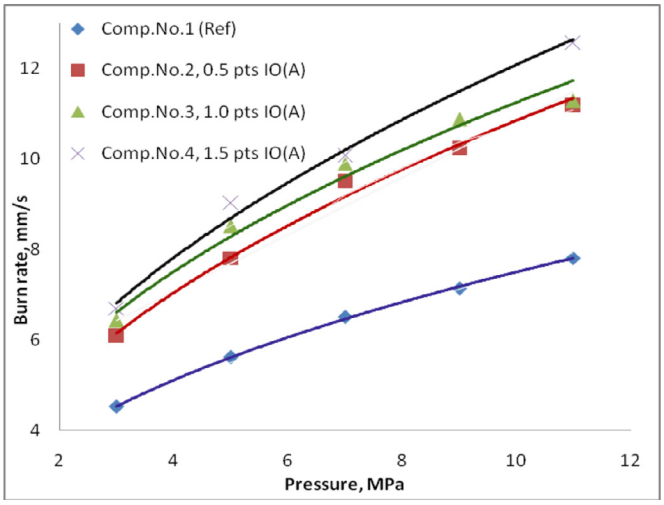

Fig. 1. Burn rate v/s pressure for propellant with IO(A).

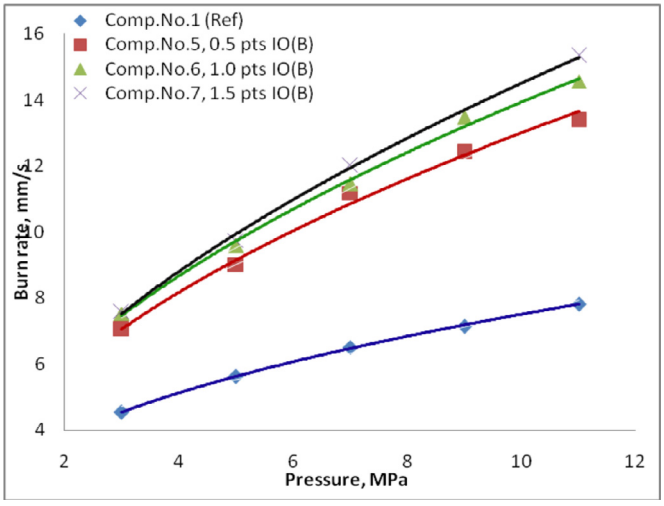

Fig. 2. Burn rate v/s pressure for propellant with IO(B).

Figure 1 showed the comparison of burn rate data for compositions with IO (A) i.e., C2, C3 and C4 with reference composition(C1). Similarly burn rate v/s pressure graphs for the compositions with IO (B) i.e., C5, C6 and C7 were compared with reference composition (C1) in Fig. 2.

These results indicated that the burn rate of propellant enhanced with the incorporation of IO. It happens since the presence of IO reduces the AP decomposition temperature and increases the gas phase reactions [18–19,32]. IO also enhances AP condensed phase reactions and reduces binder melt layer on the burning surface. Which help in accelerating the deflagration rates of AP hence the burning rate of propellant [32].

It was also seen from Figs. 1 and 2 that burn rate of propellant increased with increasing content of IO at each pressure. It was found that burn rate enhancement was significant with 0.5% IO. But after that burn rate increased marginally with further increase in IO level. Figures 1 and 2 also showed that at lower pressure (i.e., at 3 MPa), the enhancement in burn rate with further increased % IO after 0.5% was negligible. But it became significant as pressure

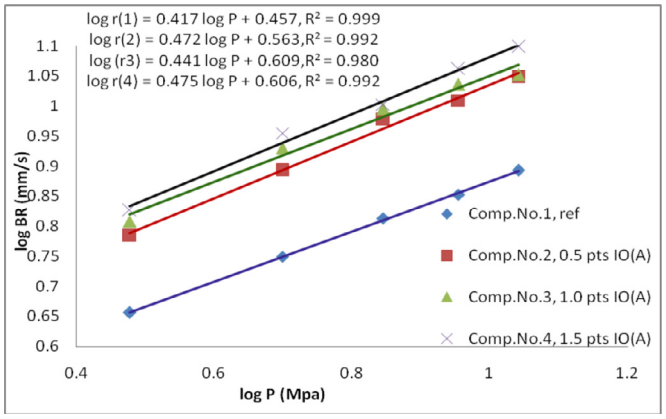

Fig. 3. Pressure Index for propellant with IO(A).

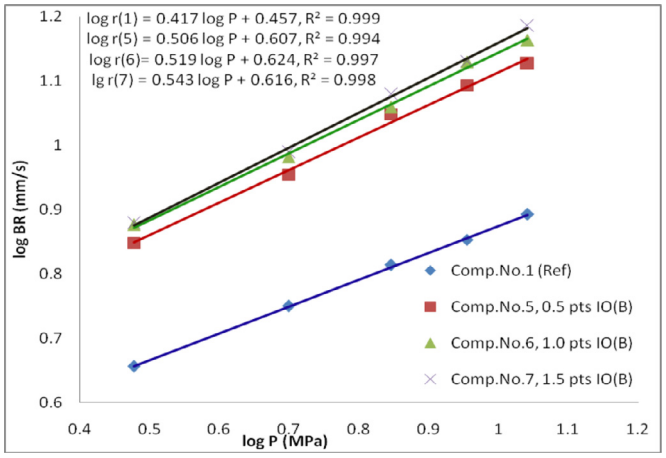

Fig. 4. Pressure Index for propellant with IO(B).

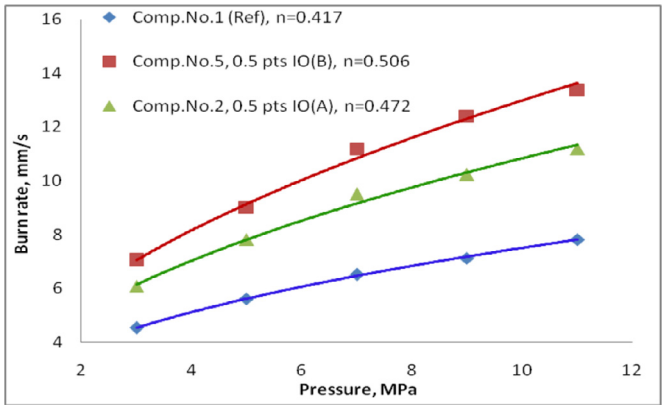

Fig. 5. Variation of burn rate with pressure for propellant with IO(A) and IO(B).

increased. This might be due to the increased n-value with IO as indicated from Figs. 3 and 4. Similar trend is observed for both the IO.

Burn rate of C2 i.e., with IO (A) and C5 i.e., with IO (B) was compared with reference C1, as shown in Fig. 5. It was noticed that though both the IO were effective burn rate enhancer but IO (B) was found to be more effective than IO (A). Burn rate enhancement was about 30% more with the propellants containing IO (B). It was because IO (B) having about 15 times more specific surface area than the IO (A). Due to which it provided more surface active sites where AP decomposition products (NH_3 and $HClO_4$) could adsorb and then further react. Hence accelerated gas phase reactions which increased the heat transfer and hence burn rate.

Though both the IO were of ~1 µm average particle size but the higher specific surface of IO(B) was due to its calcinations at very high temperature >1700 K during manufacturing. High specific surface area can also be achieved if nano-sized particles are used. But these nano-materials have their own limitations in terms of their higher cost, processing difficulty and hazards associated with them. Also, the physical and chemical properties of a material change drastically as move from micron to nano size.

3.2. Pressure index

Pressure index (*n*-value) was calculated from log BR v/s log P graphs (Figs. 4 and 5) and summarized in Table 3.

It was noticed that the presence of IO enhanced the burn rate pressure index. Ishitha and Ramakrishna also observed the enhancement in regression rate of AP and burning rate pressure index [32]. As composite propellant comprises of both premixed flame and diffusion flame. The dominance of each of these flames determines the burn rate pressure index of the propellant. When the binder melt is less, the relative burning surface area of AP particles is more and also, they are not separated by large distance due to which certain amount of premixing is possible. This will lead to a higher *n*-value.

Another reason for higher *n*-value for propellant with IO is the higher thermal conductivity of these propellants. An increase in thermal conductivity of the propellant enhances the thermal penetration thickness. Thus, a higher thermal conductivity value for propellant on addition of IO, could depict that the available heat at the surface is less and the smaller binders melt flow over the surface, hence corresponding higher n-value observed in propellant having IO [32].

3.3. Slurry viscosity

Propellant slurry EOM viscosity and viscosity build up for all the seven compositions were also measured and presented in Table 4. Results indicated that EOM viscosity and viscosity build up were not much affected by IO(A) and IO(B). Small variation in viscosity data might be attributed to error tolerances in measurements, instrument's sensitivity, process parameters variation, AP particle size distribution. Also it could be the reason that ultrafine particles of IO might have occupied the interstitial sites during the packing of AP (300 µm) and AP (60 µm) hence not much affecting the slurry viscosity.

3.4. Mechanical properties

Physical and mechanical properties of all the propellant samples were measured and summarized in Table 5. Density of all the propellant samples was in the range of 1.72–1.74 g/cc. Small enhancement in propellant density with increment of IO level was attributed to the higher density of IO. Mechanical properties data revealed that IO modified the mechanical properties only to small extent. Mechanical properties were found to be in the acceptable range of variation. Hence IO did not adversely alter the mechanical properties to remarkable extent.

3.5. IO(B), an alternative to ultrafine AP

Ultrafine AP is widely used in propellant composition with high burn rate. But it has certain limitations like: reproducibility in attaining the same particle size at every time, difficulty in processing in terms of high propellant slurry EOM viscosity and shorter pot

Table 3
Pressure index and thermal conductivity of propellant with IO (A) and IO (B).

Composition No.	IO(A)	*n*	Thermal Conductivity, W/m K	Composition	IO(B)	*n*	Thermal Conductivity, W/m K
C1	0.0%	0.417	0.53	C1	0.0%	0.417	0.53
C2	0.5%	0.472	0.56	C5	0.5%	0.506	0.58
C3	1.0%	0.441	0.56	C6	1.0%	0.519	0.59
C4	1.5%	0.475	0.57	C7	1.5%	0.543	0.62

Table 4
Propellant slurry viscosity.

Comp.No.	IO (A)	Viscosity at 317 K, Pa s		Comp. No	IO (B)	Viscosity at 317 K, Pa s	
		EOM	After 1 h			EOM	After 1 h
C1	Nil	416	672	C1	Nil	416	672
C2	0.5	384	512	C5	0.5	352	480
C3	1.0	448	608	C6	1.0	320	512
C4	1.5	352	448	C7	1.5	448	640

Table 5
Physical and mechanical properties.

Comp No.	C1	C2	C3	C4	C5	C6	C7
IO (A) wt%	Nil	0.5	1.0	1.5	Nil	Nil	Nil
IO (B) wt%	Nil	Nil	Nil	Nil	0.5	1.0	1.5
Density g/cc	1.723	1.729	1.733	1.741	1.729	1.731	1.740
T.S MPa	0.45	0.56	0.59	0.57	0.65	0.56	0.51
Elongation %	59.8	53.6	51.7	50.5	54.1	55.9	56.7
E-Mod MPa	2.8	2.7	3.3	3.1	2.5	2.9	3.6

Table 6
Comparison of two iron oxide.

Parameter	Propellant composition	
	C8 [HTPB based binder +IO(A)+ Al+ AP(300, 60 & 6 µm)]	C9 [HTPB based binder +IO(B)+ Al+ AP(300 & 60 µm)]
B.R.at 5 MPa (mm/s)	14±0.2	14±0.2
EOM viscosity at 40 °C (**Pa s**)	1088	400
n-value	0.454	0.469

life. Moreover, the process of AP size reduction to ultrafine is very hazardous.

Experiments were conducted to get the desired high burn rate of propellant with easy and safe propellant processing. Propellant composition was developed with IO (B). This study was used in the experiments where the desired high burn rate (i.e., 14±0.2 mm/s at 5 MPa) of propellant was achieved without using the ultra-fine AP. Table 6 mentioned the comparison of two propellant compositions having same burn rate. C8 was HTPB based composite propellant with 86% solid loading, comprising of Al, AP (300, 60 and 6 µm) and IO (A). Similar burn rate was achieved in **C9** wherein IO(B) was used in place of IO(A) without using ultrafine AP(6 µm).

In C9, same burn rate was achieved with lower EOM viscosity and longer pot life, which enabled the propellant slurry to cast in the rocket motor with very low web (6–10 mm), which would be otherwise not possible. High burn rate could also be achieved by using nano burn rate enhancer but their incorporation generally result in higher EOM viscosity, higher hazard, higher cost and lesser reproducibility. Further, as the slurry viscosity was low enough in C9, to accommodate more solids in the propellant formulation. Hence the C9 can further be modified by enhancing the solid loading, to get, high energetic, also it can be further modified to achieve higher burning rate with improved ballistic performance. Of course the efforts are being continued to further improvement.

4. Conclusion

Experiments were performed to study the effect of IO and its physical parameters on ballistic properties of composite propellant. Two IO (A and B) were used in propellant composition. From all these experiments it was concluded that burn rate of propellant enhanced with the incorporation of IO. Burn rate of propellant increased with increasing level of IO. It was concluded that burn rate enhancement was significant with 0.5% IO but after that only marginally enhancement in burn rate with further increase in IO. The trend was similar with both IO. But the burn rate enhancement is about 30% more with the propellants containing IO (B). This observation could be explained on the basis of the difference in their specific surface area. This was because IO (B) was having about 15 times more specific surface area than the IO (A). Higher specific surface of IO (B) was due to its calcination at very high temperature during manufacturing. The presence of IO also enhanced the burn rate pressure index. It was also concluded that the mechanical strength and processing characteristics did not get affect by IO. Based on these observations, it was proposed that calcinated IO i.e., IO (B) could be a good mean to enhance the burn rate of composite propellant significantly without use of ultrafine AP or nano burn rate enhancers. This study can be further used to improve solid loading and hence propellant performance. The use of IO (B) is cost effective also, as it can be used in place of nanoparticles which are of very high cost. This study was used to develop the propellant composition of high burn rate (14±0.2 mm/s at 5 MPa) and eliminate the incorporation of ultrafine AP. Hence improved the reproducibility in ballistics properties of composite propellant. Hence a new approach was suggested to develop propellant composition of high burn rate and high performance. Constant endeavor for further improvement is still continued.

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