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Studies on characterization of size and shape of pulverizer ground ammonium perchlorate using laser based analyzers

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ABSTRACT

Most of composite propellant compositions contain solid loading up to 86 %. The main solid ingredients of composite propellant is ammonium perchlorate which is used in multi-modal form. The different particle sizes of AP are obtained using pulverizer, air classifier mill and fluid energy mill. However, pulverizer is most commonly used to get particle sizes in different range. In the present study, pulverizer has been utilized to grind AP by varying operating parameters such as pulverizer speed and feed rate. The ground AP was characterized using laser based particle size analyzer (CILAS make) and dynamic shape analyzer, DSA-10 of Ankersmid make. The data on shape infer that as size of ground AP decreases, shape factor decreases and particles become more irregular in shape. Also, based on the different grinding parameters of pulverizer, an empirical equation has been developed and used for the prediction of particle size. The experimental results indicate that the values of ground AP particle sizes are very close to predicted ones.

KEYWORDS

Ammonium perchlorate;
Pulverizer;
Particle analysers;
Average particle size;
Particle shape;
Shape factor.

INTRODUCTION

Composite propellants (CP) are the most important class of solid rocket propellants. Basically, a composite propellant contains an oxidizer mainly ammonium perchlorate (AP) (60-80%), a binder such as hydroxyl terminated polybutadiene (HTPB); (10-15%) and a metallic fuel like aluminium powder (15-20%). Ammonium perchlorate is the most important ingredient of solid rocket propellants. Its variation in particle size as well as coarse to fine ratio are responsible for the processibility and ballistic properties of the compositions such as burn-rate, pressure index and temperature sensitivity along with mechanical properties[1,2].

Generally, burn-rate is considered to be the single most important property governing the ballistic performance of a solid motor. The burn-rate, in turn depends upon particle characteristics of oxidizer, metal fuel and burn-rate catalyst[3-8]. The coarse/fine combination of AP affects the burn rate more prominently than other ingredients used in composite propellants.

Further to this, ballistic properties are also affected by particle shape. If particles of oblong shape are mixed in a propellant to a maximum packing level, it is quite likely that the particles develop some sort of preferred direction of alignment in packets of propellant block. This would give rise to different burning rates along different directions of flame propagation[9]. The volume packing of filler particles directly determines mechanical and rheological properties of the propellant. The ideal packing network assumes the fine AP particles occupying the void spaces in the coarse AP packing network; in turn the aluminium (P) particles thread their way in between the voids created by fine AP packing. This results in better packing network leading to lowering viscosity and improved mechanical properties.

Role of particle size measurement

The particles of coarse ammonium perchlorate procured from trade do not have consistent size and shape. Therefore, before using it in a composite propellant composition, it is essential to determine their size and shape. The particle size measurement is of two types i.e., primary measurement and secondary measurement. Primary measurements are direct, as carried out by microscopy while secondary are derived from the effects of some well studied physical phenomenon like settling, diffraction, or permeatry[10]. As a result, against the plethora of measurement techniques, there exists a different figure of particle sizes such as martin, ferets, surface, stokes, sieve diameters, etc. Other factors to be borne in mind before measurement include the end use of powder, sensitivity of the analyzer for the particle size envisaged. The industrial powders with large polydispersity cannot be adequately described by average particle size alone but distribution of various constituent fractions. Three standard distributions namely Normal, Log-Normal and Rosin-Rammler-Bennett are used to simulate the size distribution of powders and thus provide an accumulated view of particle size measurements[11].

Conventionally, the particle size of ammonium perchlorate is determined using sieve analysis. However, by sieving it is not possible to characterize powders below 40µm. Moreover, conventional methods are also handicapped by being manual in nature and hence prone to human error. So the focus of particle characterization has shifted from the usual time consuming and less accurate conventional methods to the laser based techniques.

Role of particle shape measurement

The need now a days is not only for the measurement of size and size distribution, but also for the measurement of shape and shape distributions. Particle shape is a fundamental powder property affecting powder packing, and thus bulk density, porosity, permeability, cohesion, flow ability, caking behavior, attrition, interaction with fluids, etc.

The shape of particle can be measured using dynamic shape analyzer. There exist shape parameters like roundness, chunkiness, irregularity, compactness, statistical shape indices, Fourier coefficients, fractal dimensions, aspect ratio, etc.[12-14]. However, the most important shape parameter is shape factor obtained by Digital Dynamic Shape Analyzer, which is based on image analysis technique. The shape factor is defined as follows:-

Shape factor = $4.\pi.A/P^2$

Where, A- Projected area of the particle, P- Perimeter of the projected particle area, (For perfect spherical particle Shape factor =1)

The lowering in the viscosity is mostly governed by size and shape of particles. As the particles become more spherical, packing efficiency increases which is responsible for lowering the viscosity. Thus, by selecting suitable size, shape and proportion of solid ingredients, it is possible to increase solid loading of the propellant compositions. The increase in solid loading gives better performance of rocket motor, which is our main aim. Thus, in order to cope up with the demand of AP in large quantity in different sizes (35-90µm), it is essential to characterize size and shape of the ground AP for better processibility.

In the following section, we report the effect of different operating parameters such as mill speed and feed rate of pulverizer on particle size and shape of AP using particle size and shape analyzers and its comparison to predicted values.

EXPERIMENTAL

Material

Ammonium perchlorate, procured from Pandian Chemicals Ltd. (PCL), cuddalore having average particle size 290µm prepared by anodic oxidation of sodium chloride and double decomposition reaction with ammonium chloride, was used as grinding material.

The pulverizer, model 1-SH procured from REICO, Pune, having grinding capacity in the range of 100-170 kg/hr, was used for grinding of ammonium perchlorate.

Characterization

The particle size of AP was determined by laser based particle size analyser CILAS, France, model-1064, in a non-aqueous medium. Shape factor of ammonium perchlorate was determined by Dynamic Shape Analyzer, DSA-10 of Ankersmid, Netherlands.

Procedure

All the experimental grindings were carried out by taking 5 kg of ammonium perchlorate in a fully assembled REICO pulverizer (1-SH). The mill diameter from hammer tip to hammer tip was 205 mm. The gap between hammer tip and mill case was 2 mm, and between hammer tip and hold down plate (screen) was 1.3 mm. The screen, which was used for grinding, had an opening of 0.5 mm. Ammonium Perchlorate from the hopper was charged continuously into the mill with the help of a screw feeder. It was pulverized by the collision against hammers and the lining plate and was discharged through the screen[15,16]. The product, thus obtained, was sampled by coning and quartering method. The average particle size and shape were characterized. Further to this, grindings were also carried out at different sets of pulverizer mill rpm and feed rate using AP of different surface moistures. Feed rate was measured by collecting the ground material for 1 minute from the feed screw and it was changed by varying the tension of feeder motor belt.

RESULTS & DISCUSSION

Effect of pulverizer mill rpm on average particle size

The effect of pulverizer mill rpm on ammonium perchlorate grinding is presented in TABLE 1 and figure 1. The data infer that on increasing the mill rpm from 660 to 1552 there is a drastic decrease in the particle size of ammonium perchlorate. This can also be correlated that on increasing the mill rpm there is an increase in stress events, which is responsible for decrease in the particle size.

The data on particle size of ground ammonium perchlorate were further compared with the sizes calculated from the following derived empirical equation relating average particle size to mill parameters.

$$P = K . P_F . (A)^{-1.031} . (B)^{0.1619}$$

Where, P- Particle size of Product in μm, P_F - Particle size of feed (290μm), A - Mill speed (rpm), B - Feed rate (Kg/hr), K - 120

In the above equation K is a mill factor, whose value depends upon the feed size, gap between the liner plate and hammer tip and also upon the condition of hammer tips. For the system under study with size of feed material 290μm, value of K was found to be 120.

Effect of feed rate on average particle size

To study the effect of feed rate on the particle size, grinding of ammonium perchlorate was carried out by keeping mill rpm constant at 1035 and data obtained are presented in TABLE 2 and figure 2. The less reduction in particle size on increasing the Feed screw rpm is due to the fact that as the feed rpm (feed rate) increases, more particles are available in a control volume. Although probability of impact will increase yet impact of a particular particle will decrease. The effect

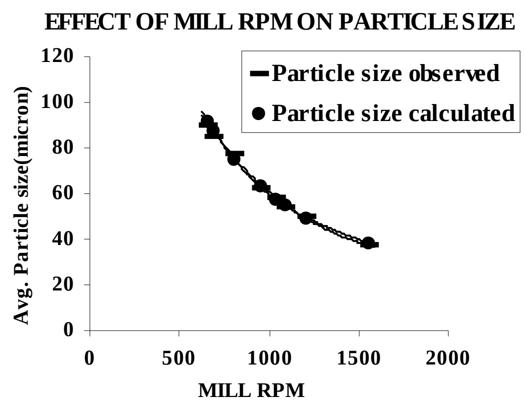

Figure 1: Effect of pulverizer mill rpm on average particle size

TABLE 1: Effect of pulverizer mill rpm on average particle size

Sr.no.	Mill rpm	Feed rate (Kg/hr)	Surface moisture of feed AP(%)	Particle size observed (μm)	Particle size calculated(μm)	Deviation in particle size (μm)	% Deviation
1.	661	107	0.03	90.25	91.87	-1.62	-1.79
2.	690	107	0.03	85.03	87.76	-2.73	-3.21
3.	805	107	0.03	77.72	74.86	2.86	3.68
4.	948	107	0.03	62.60	63.25	-0.65	-1.04
5.	1035	107	0.03	58.68	57.77	0.91	1.55
6.	1092	107	0.03	54.55	54.67	-0.12	-0.22
7.	1206	107	0.03	49.77	49.35	0.42	0.84
8.	1552	107	0.03	37.13	38.05	-0.92	-2.48

Full Paper

TABLE 2: Effect of feed rate on average particle size

Sr. no.	Mill rpm	Feed rate (Kg/hr)	Surface moisture of feed AP(%)	Particle size observed (μm)	Particle size calculated (μm)	Deviation in particle size (μm)	% Deviation
1.	1035	100	0.03	58.18	57.14	1.04	1.79
2.	1035	116	0.03	59.01	58.53	0.48	0.81
3.	1035	129	0.03	62.34	59.55	2.79	4.47
4.	1035	142	0.03	63.10	60.48	2.62	4.15
5.	1035	164	0.03	63.90	61.91	1.99	3.11

TABLE 3: Effect of surface moisture of feed AP coarse on average particle size

Sr. no.	Mill rpm	Feed rate (Kg/hr)	Surface moisture of feed AP(%)	Particle size observed (μm)	Particle size calculated(μm)	Deviation in particle size(μm)	% Deviation
1.	1035	107	0.02	58.34	57.77	-0.34	0.58
2.	1035	107	0.03	58.68	57.77	0.91	1.55
3.	1035	107	0.04	58.70	57.77	0.93	1.58
4.	1035	107	0.05	58.06	57.77	0.29	0.50
5.	1035	107	0.06	58.85	57.77	1.08	1.83
6.	1035	107	0.07	58.82	57.77	1.05	1.78
7.	1035	107	0.08	58.96	57.77	1.19	2.02

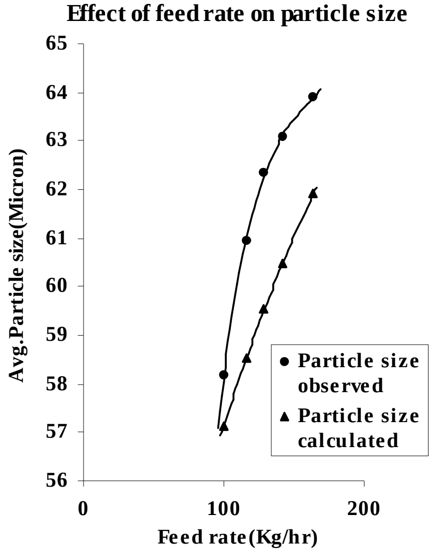

Figure 2: Effect of feed rate on average particle size

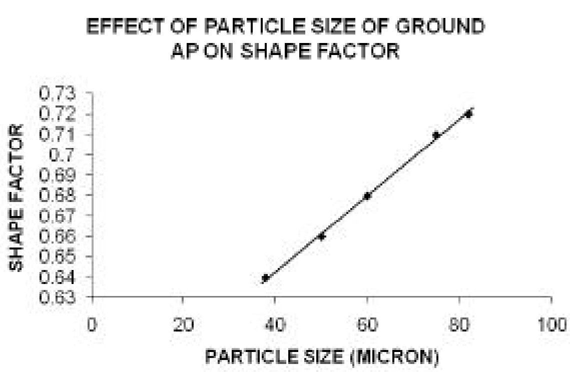

Figure 3: Effect of particle size of ground AP on shape factor

of feed rate on particle size is not as prominent as of mill rpm. The above data are also compared by using empirical equation and values suggest that these are in a good agreement with the observed values.

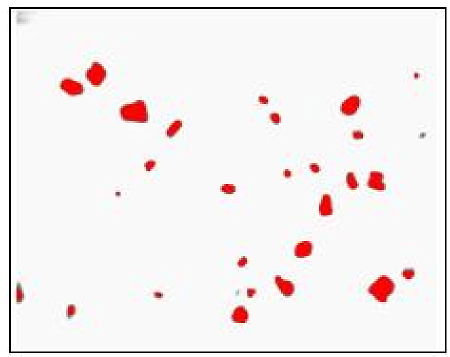

Figure 4: Photograph of ground AP by shape analyser

Effect of particle size of ground AP on shape factor

In continuation to this work further, ammonium perchlorate having average particle size 290μm was ground using a fully assembled REICO pulverizer grinding mill. The mill parameters were set to get particles having average size of 35μm, 50μm, 60μm, 70μm and 80 μm as measured by CILAS particle size analyzer. All ground AP powders were analyzed for shape using DSA-10. The variation in shape factor with particle size is shown in figure 3. The figure reveals that as particle size decreases, shape factor decreases. It indicates that ground particle becomes more irregular as it becomes finer. Furthermore, shape of ground ammonium perchlorate (figure 4) also indicates that irregular size of fine particles.

Effect of surface moisture of feed AP on average particle size

TABLE 4 : Effect of surface moisture of AP on feed rate

Sr. no.	Surface moisture of feed AP(%)	Feed rate (Kg/hr)
1.	0.02	107.0
2.	0.03	107.0
3.	0.04	107.0
4.	0.05	106.5
5.	0.06	106.0
6.	0.07	105.5
7.	0.08	105.0

Due to hygroscopic nature of AP, the effect of surface moisture of feed on grinding was also studied by keeping mill rpm and feed rate constant at 1035 rpm and 107 kg/hr, respectively, and data obtained are presented in TABLE 3. It is clear from the TABLE that the feed rate fairly remains constant upto 0.05 % surface moisture, thereafter it decreases as shown in TABLE 4. The average particle size remains almost constant with respect to surface moisture i.e. it does not change much in the range of 0.02 %- 0.08 % surface moisture of feed AP coarse.

CONCLUSION

The characterization of size and shape of ground AP have been carried out successfully using size and shape analyzers, respectively. The grinding of ammonium perchlorate was established by changing different operating parameters. Based on the above data an empirical equation was also derived. The experimentally obtained particle size data from particle size analyzer were compared with predicted ones, which suggest a good agreement in their values. The developed equation may find application during grinding of ammonium perchlorate for different particle size distribution using pulverizer. Further, the data on shape factor of ground AP infer that as size of ground AP decreases, shape factor also decreases as particle becomes more irregular in shape. Thus, characterization techniques are important in maintaining and improving the quality of finished product in terms of compaction, density, mechanical properties and burn rate and also help in smooth processing of propellant compositions.

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Abbreviations

AP : ammonium perchlorate
HTPB : hydroxy terminated polybutadiene
rpm : revolutions per minute
μm : micro meter

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