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SHORT COMMUNICATION

Prediction of Particle Size of Ammonium Perchlorate during Pulverisation

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ABSTRACT

Ammonium perchlorate has been pulverised by an impact mill (air classifier mill) to study the influence of different operating parameters, viz., effect of mill speed, classifier speed, feed rate, and damper opening (suction rate) on the particle size. Further based on the different grinding parameters, an empirical equation has been developed and used for the prediction of particle size. The experimental results indicate that the values are very close to the predicted ones. In addition, particle size distribution has also been studied by applying different model equations and it has been found that Rosin-Rammler model is the most suitable model for this operation.

Keywords: Ammonium perchlorate, grinding, air classifier mill, composite propellant, average particle size, modelling equation, burn rate

NOMENCLATURE

ACM	Air classifier mill
FRAL	Feed rotary air-lock
HTPB	Hydroxy-terminated polybutadiene
RRB	Rosin-Rammler-Bennet
$F(x)$	Cumulative undersize weight fraction
B	Steepness constant
X	Particle size
x_m	Mode of distribution
σ_g	Ratio of 84 per cent size to 50 per cent size
n'	Uniformity index of distribution

1. INTRODUCTION

Composite propellants are the most important class of solid rocket propellants. Basically, a composite propellant contains an oxidiser [mainly ammonium perchlorate (60-80 %)], a binder such as hydroxy-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, 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

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turn depends upon particle characteristics of the oxidiser, metal fuel, and burn rate catalyst[3-6]. The coarse/fine combination of ammonium perchlorate affects the burn rate more prominently than the other ingredients used in composite propellants. The viscosity of propellant slurry is reduced due to proper packing achieved by incorporating multimodal oxidiser sizes, ie, 300 μm, 60 μm, and 9 μm in the composition.

Further, to cope up with the demand of ammonium perchlorate in large quantity in different sizes (35-70 μm) on a modern and flexible process, it is essential to estimate the effect of changes in the operational parameters on the process.

To get the required particle size (35-70 μm) and effects of different operating parameters such as mill speed, classifier speed, airflow rate, grinding of ammonium perchlorate in air classifier mill has been studied in detail. Based on different operating parameters, an empirical equation has been developed and utilised as a tool for predicting the particle size. Furthermore, the particle size distribution of ammonium perchlorate has also been tried to fit by different model equations[7]. The effect of different operating parameters of air classifier mill has been reported on particle size. Also, their comparison with predicted particle size distribution along with the different model equations have been reported for their suitability to ammonium perchlorate system.

2. EXPERIMENTAL PROCEDURE

2.1 Materials & Equipment

Ammonium perchlorate procured from the Ammonium Perchlorate Experimental Plant (APEP), Alwaye, having average particle size 300 μm as prepared by anodic oxidation of sodium chloride and double decomposition reaction with ammonium chloride, was used as grinding material.

The air classifier mill (ACM), model ACM-10 procured from the REICO, Pune, having grinding capacity in the range 140-300 kg/h, was used for grinding ammonium perchlorate.

2.2 Procedure

All the experimental grindings were carried out by taking ammonium perchlorate (25 kg) in a fully assembled REICO ACM-10 (Fig.1). The product size range that could be achieved by it was 30 μm to 70 μm. Also, it is not suitable for grinding of hard materials like metal fuels, metal oxides, etc. It is an impact mill consisting of a rotor in the form of a disc having 18 vertically-mounted pins on the periphery and stator in the form of a corrugated ring surrounding the rotor. The classifier is of impeller wheel type.

Ammonium perchlorate was fed to the grinding zone by a feed rotary air-lock (FRAL) valve. The

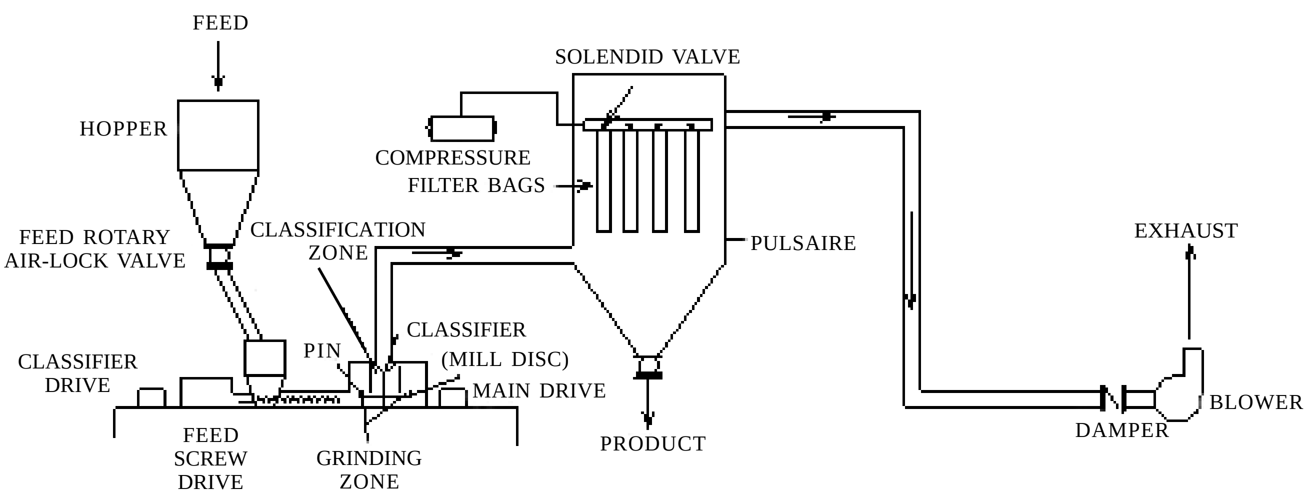

Figure 1. Schematic diagram of air classifier mill

particles of ammonium perchlorate were stressed by the grinding pins of the rotor disc causing their disintegration. The ground particles were then transported to the classification zone by the negative pressure created by a blower placed at the end of the system. A shroud ring separated the grinding zone from the classification zone.

The particles were classified based on the settling velocity, which depends upon particle density, shape, and size. In this zone, particles were subjected to mass force (gravity, inertial, centrifugal) and drag force of the air. So, the lighter particles followed the air and the heavier or larger particles were rejected and transported by the internal circulation back to the grinding zone, and were stressed again[8]. The lighter particles (product) were pneumatically conveyed to pulsair (collection system). The particles were then separated from the air at the surface of filter bags activated by reverse jet of air. The product thus formed was collected from the bottom of the pulsair through a rotary air-lock valve.

The product, thus obtained, was sampled by coning and quartering method. The average particle size was measured by sieve analysis using British Standard sieves.

3. EQUATIONS FOR PARTICLE SIZE DISTRIBUTION

The particle size distribution data can be represented in many ways. Although various models have emerged, an old approach using selection and breakage function developed by Schumann and Epstein in 1960 was used for designing size reduction operations[9]. The other models like log-normal, Rosin-Rammler-Bennet (RRB), and Gaudin-Schumann models are being used extensively for this purpose. The particle size distribution data of ammonium perchlorate obtained by varying different parameters are represented by the following mathematical functions[7]:

3.1 Rosin-Rammler-Bennet Function

The particle size distribution can be expressed by the following equation:

$$\ln\ln\,[1 / \{1-F(x)\}] = n'.\ln(x / x'_R)$$

where $F(x)$ is the cumulative undersize weight fraction x is the particle size, and x'_R and n' are the parameters of function.

By plotting left side of equation against $\ln(x)$, a straight line is obtained with a slope n', which is termed as uniformity index of distribution. The small values of n' indicate scattered distribution, whereas large values imply uniform distribution.

3.2 Gaudin-Schumann Function

The particle size distribution can be expressed by the following equation:

$$F(x) = (x / x'_G)^n$$

where x'_G and n are the parameters of the function $F(x)$.

3.3 Log-normal Distribution Function

This is commonly used for the analysis of comminution and is represented by the following equation:

$$d\,F(x) = \varepsilon\,\exp\,(-b.\,\ln^2\,(x / x_m))\,dx$$

where b is the steepness constant, which is equal to $1/(2\ln^2\sigma_g)$ and σ_g,is the ratio of the 84 per cent size to the 50 per cent size

$$\varepsilon = (b/\pi)^{1/2}\,\exp\,(-1/4b)/x_m$$

where x_m represents the mode of the distribution, which is equal to $c.x_{50}$

$$c = \exp\,(-1 / 2b)$$

4. RESULTS & DISCUSSION

Ammonium perchlorate was ground at 25 kg level in an ACM to study the effect of different operating parameters on particle size such as mill speed, classifier speed, feed rate, and airflow rate (damper opening) by varying one parameter only at a time and keeping the other parameters constant. The experimentally obtained average particle size of ammonium perchlorate was further compared with the particle size calculated from an empirical equation developed for the system.

Table 1. Effect of mill revolutions per minute on average particle size

FRAL rpm	Damper opening fraction	Mill rpm	Classifier rpm	Average particle size (observed)	Average particle size (calculated)	Deviation in particle size	Deviation
(A)	(B)	(C)	(D)	(μm)	(μm)	(μm)	(%)
			(a)				
5	0.75	3000	1200	49.86	45.36	4.50	9.02
5	0.75	3500	1200	43.23	39.79	3.44	7.96
5	0.75	4000	1200	38.54	35.52	3.02	7.84
5	0.75	4500	1200	34.63	32.14	2.49	7.19
5	0.75	5000	1200	30.78	29.38	1.40	4.54
			(b)				
5	0.75	3000	1000	59.91	54.93	4.98	8.31
5	0.75	3500	1000	52.28	48.18	4.10	7.84
5	0.75	4000	1000	46.63	43.01	3.62	7.76
5	0.75	4500	1000	42.15	38.92	3.23	7.66
5	0.75	5000	1000	38.48	35.58	2.90	7.53

4.1 Effect of Mill Speed on Average Particle Size

The effect of mill rpm on ammonium perchlorate grinding is presented in Table 1 and Fig. 2. In these trials, the grinder feed rate and pneumatic conveying rate were kept constant by controlling FRAL valve and line damper opening. Two sets of experiments were carried out at two different classifier rpm, viz., 1200 rpm and 1000 rpm, while FRAL valve and damper opening were at optimum. It is clear from Table 1 that at 3000 mill rpm, the value of an average particle size is 49.86 μm, whereas at 4000 mill rpm, it is 38.54 μm. Further, at 5000 rpm, it is about 30.78 μm. The above data infer that on increasing the mill rpm from 3000 to 5000, there is a drastic change 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, thus total speed increases, which finally decreases the particle size.

The above data of average particle size of ammonium perchlorate were further compared with the sizes calculated from the following derived empirical equation relating to an average particle size to four parameters:

$$P = 4.41 \times 10^{7} \cdot (A.B)^{0.35} \cdot C^{-0.85} \cdot D^{-1.05}$$

where

A	FRAL rpm (5-9)
B	Damper opening (25-100)
C	Mill rpm (3000-5000)
D	Classifier rpm (700-1200)
P	Average particle size

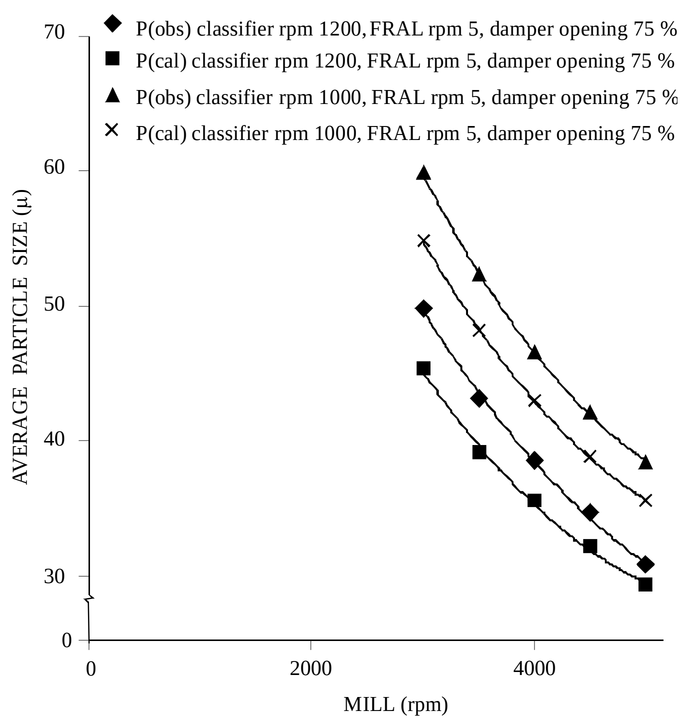

Figure 2. E ect of mill rpm on average particle size

Table 2. Effect of classifier revolution per minute on average particle size

FRAL rpm	Damper opening fraction	Mill rpm	Classifier rpm	Average particle size (observed)	Average particle size (calculated)	Deviation in particle size	Deviation
(A)	(B)	(C)	(D)	(μm)	(μm)	(μm)	(%)
			(a)				
5	0.75	4000	700	54.25	62.55	-8.30	-15.30
5	0.75	4000	850	50.40	51.02	-0.62	-1.23
5	0.75	4000	1000	49.80	43.01	6.79	13.63
5	0.75	4000	1100	43.04	38.92	4.12	9.57
5	0.75	4000	1200	38.54	35.52	3.02	7.84
			(b)				
5	0.75	5000	700	48.87	51.74	-2.87	-5.87
5	0.75	5000	850	42.35	42.20	0.15	0.35
5	0.75	5000	1000	38.48	35.58	2.89	7.53
5	0.75	5000	1100	33.31	32.19	1.12	3.36
5	0.75	5000	1200	30.78	29.38	1.40	4.55

Thus, it is clear from Table 1 that at low mill rpm the particle size difference between the observed and the predicted values are in the range 8-9 per cent, while on increasing the mill revolutions per minute the difference becomes less and at optimum revolutions per minute, ie 5000, it is very close to the observed values.

4.2 Effect of Classifier Revolutions per Minute on Average Particle Size

The effect of classifier revolutions per minute on particle size of ammonim perchlorate is presented in Table 2 and Fig. 3. The effect of classifier revolutions per minute was studied at two mill revolutions per minute ie at 4000 and 5000 by keeping other parameters constant. It is clear from Table 2 that on increasing the classifier mill revolutions per minute from 700 to 1200 (max), there is a noticeably decrease in the particle size of ammonium perchlorate. The data suggest that both mill revolutions per minute as well as classifier rpm are playing a vital role during the grinding of ammonium perchlorate and are interdependent. The predicted values of particle size of ammonium perchlorate are also very close at higher values of mill rpm and classifier rpm. Thus, the difference in values are in the range 1-15 per cent at 4000 mill rpm and 0–7 per cent at 5000 mill rpm.

Table 3. Effect of FRAL rpm on average particle size

FRAL rpm	Damper opening fraction	Mill rpm	Classifier rpm	Average particle size (observed)	Average particle size (calculated)	Deviation in particle size	Deviation
(A)	(B)	(C)	(D)	(μm)	(μm)	(μm)	(%)
5	0.5	5000	1000	31.98	30.87	1.11	3.47
6	0.5	5000	1000	33.81	32.91	0.90	2.66
7	0.5	5000	1000	35.98	34.73	1.25	3.47
8	0.5	5000	1000	37.13	36.39	0.74	1.99
9	0.5	5000	1000	38.28	37.93	0.35	0.91

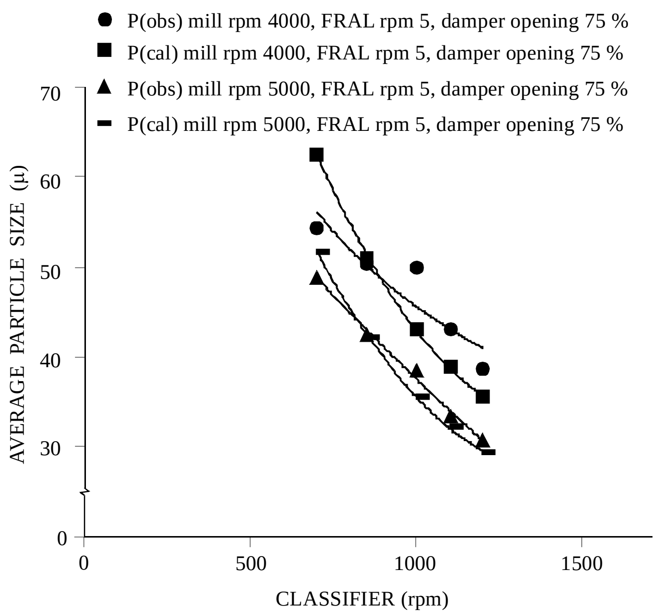

Figure 3. Effect of classifier revolutions per minute on average particle size of ammonium perchlorate.

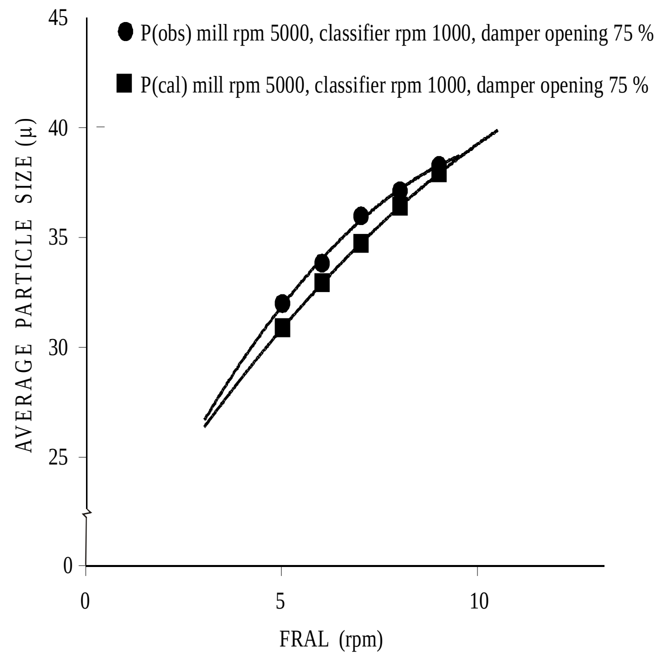

Figure 4. Effect of FRAL revolutions per minute on average particle size of ammonium perchlorate.

4.3 Effect of FRAL Revolutions per Minute on Average Particle Size

To study the effect of FRAL rpm on the particle size, grinding of ammonium perchlorate was carried out by keeping damper opening, mill rpm, and classifier rpm constant and data obtained are presented in Table 3 and Fig. 4. The data reveals that on increasing the FRAL rpm there is a reverse trend in particle size in comparison to mill rpm and classifier rpm variations. The data infer that on increasing the FRAL rpm, there is less reduction in particle size. Thus, at FRAL rpm 5, the particle size observed is 31.98 μm and 35.98 μm at FRAL rpm 7 while at the FRAL rpm 9, the value is 38.28 μm. The less reduction in particle size on increasing the FRAL rpm is due to the fact that as the FRAL 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 above data were also compared using an empirical equation and the values thus obtained suggest that these are in good agreement with the observed values.

4.4 Effect of Damper Opening on Particle Size

To study the effect of damper opening on particle size, grinding of ammonium perchlorate was carried out by keeping FRAL rpm, mill rpm and classifier rpm constant, and data thus obtained are presented in Table 4 and Fig. 5. It is clear from Table 4 that at lower damper opening, ie, low airflow rate, the particles are smaller and on increasing the damper opening, there is less reduction in particle size because of increase in the drag force on the particles

Table 4. Effect of damper opening on average particle size

FRAL rpm	Damper opening fraction	Mill rpm	Classifier rpm	Average particle size (observed)	Average particle size (calculated)	Deviation in particle size	Deviation
(A)	(B)	(C)	(D)	(μm)	(μm)	(μm)	(%)
5	0.25	4000	700	38.09	42.58	-4.49	-11.79
5	0.50	4000	700	47.16	54.28	-7.12	-15.10
5	0.75	4000	700	54.25	62.55	-8.30	-15.30
5	1.00	4000	700	57.58	69.18	-11.60	-20.14

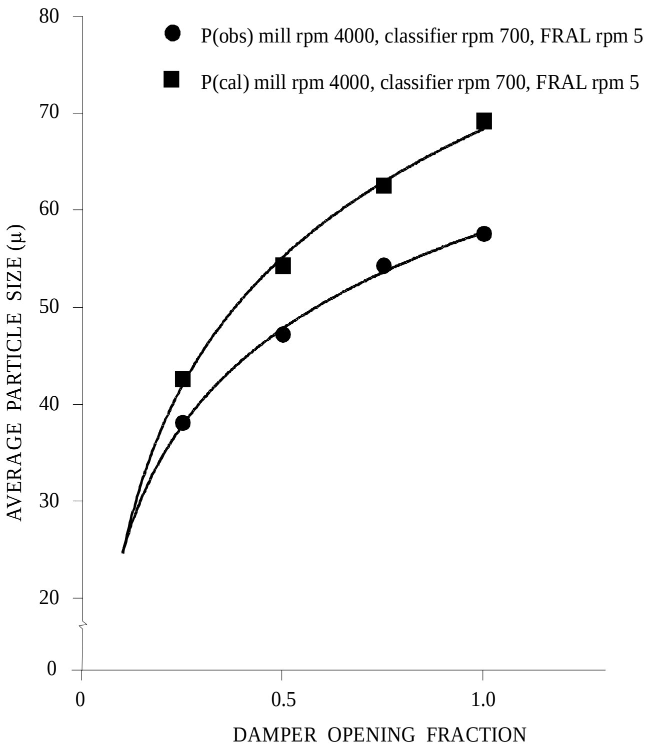

Figure 5. Effect of damper opening on average particle size of ammonium perchlorate.

(at constant centrifugal force). Thus at 50 per cent damper opening, the average particle size is 47.16 μm while at 75 per cent damper opening, it is 54.25 μm. The maximum value obtained at 100 per cent damper opening is 57.58 μm. The above data were also compared with the predicted data and it was infered that in the present study, predicted values are on a higher side than the observed values.

5. ASPECTS OF SIZE DISTRIBUTION EQUATIONS

The three models, viz., Rosin-Rammler-Bennet (RRB), Gaudin-Schumann function, and log-normal distribution function were employed to find the suitability of models for the ammonium perchlorate grinding system. In this system, based on the equation, model graphs were plotted and coefficient of determination or coefficient of regression, ie R^2 was determined. Higher the value of R^2, better the suitability of the model to a particular system. Thus, the graphs were plotted of ln ln $(1/(1-F(x)))$ versus ln (x), ln $F(x)$ versus ln (x), and $F(x)$ versus ln (x) using RRB, Gaudin-Schumann, and log-normal distribution on equation, respectively, and presented in Fig. 6. The value of coefficient determination (R^2) for RRB equation was found to be 0.9824 while in the case of Gaudin-Schumann function, the value of R^2 was 0.8685. Further, in

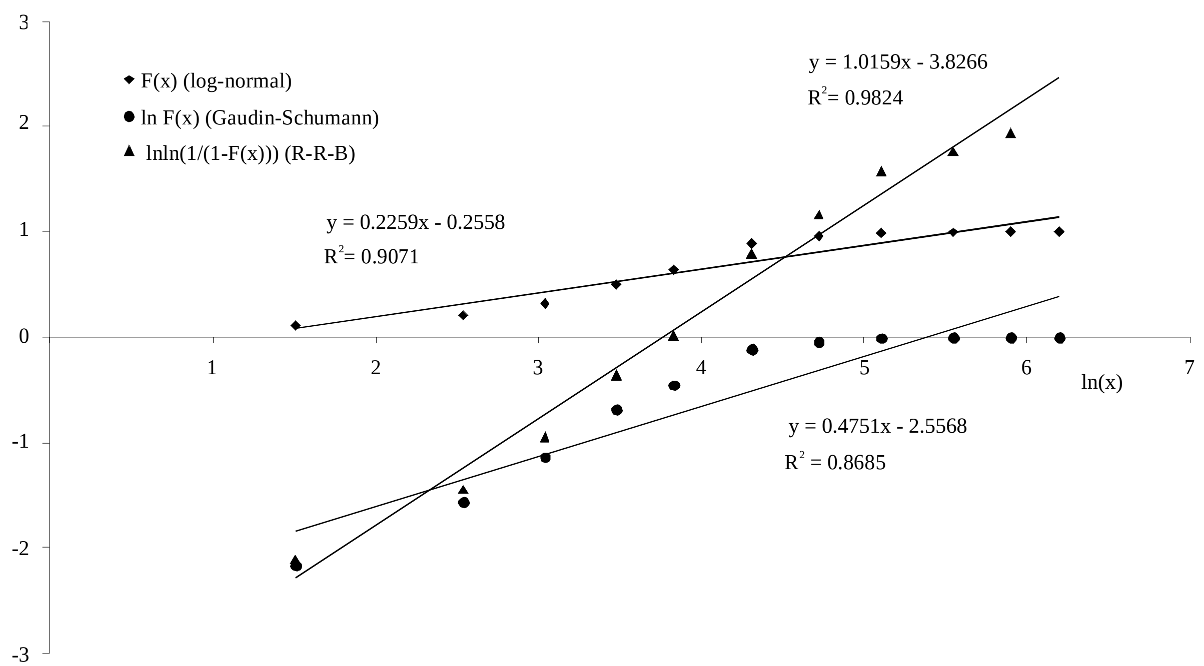

Figure 6. Log-normal, Gaudin-Schumann & Rosin-Rammler-Bennett plots for air classifier mill ground powder

the log-normal distribution function, the value of R^2 was 0.9071. The above data indicate that R^2 is maximum (0.9824) in case of RRB function, whereas lowest (0.8685) in the case of Gaudin-Schumann function. Thus, it may be concluded that RRB model equation is the best fit for ammonium perchlorate grinding system.

The aim of study was to explore the utility of an empirical equation to get the required particle size of ammonium perchlorate without carrying out many trials for composite propellant manufacture where ground ammonium perchlorate is used in bulk quantity. The predicted data indicate that experimentally obtained particle sizes of ammonium perchlorate are in good agreement.

6. CONCLUSION

The grinding of ammonium perchlorate was established by changing the different operating parameters. Based on the above data, an empirical equation has also been derived. The experimentally obtained particle size of ammonium perchlorate were compared with the predicted ones which suggest a very good agreement in their values. Thus, the developed equation may find application during the grinding of ammonium perchlorate for different particle size distribution. Further, three different model equations were also employed and Rosin-Rammler-Bennett equation was found the best fit for ammonium perchlorate grinding system.

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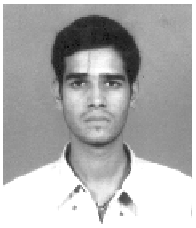
Contributors



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