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# Thermal and sensitivity analysis of nano aluminium powder for firework application

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**Abstract** The performance of the fireworks is governed by the sound level it produces. As per Govt. of India notification, crackers sound level should not exceed 125 dB (AI) or 145 dB(C) pk. In this study, nAl powder was synthesised at different particle sizes of 113, 187 and 218 nm as a fuel. These powders are well mixed with the oxidizer ($KNO_3$) and the igniter (S), and the cake bomb was manufactured with various compositions and checked for their performance. The thermal analysis was performed by DSC and DTA, and the impact sensitivity was analysed for all compositions. The nano-sized chemicals showed high thermal energy content and high sensitivity for various compositions. Further it is observed that the nAl chemical for producing the optimum sound level in the cake bomb has been reduced to 62.5% when compared with μm powder.

**Keywords** nAl · Firework powders · Impact sensitivity · Noise level

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## Introduction

In firework industry, various metal/chemical powders are mixed in different compositions consisting of Al, S with oxidizing agents like $KNO_3$, $BaNO_3$ and $KClO_4$ to attain the explosivity. It is difficult to estimate the degree to which particle size of the fuel can affect burn rate. The explosivity can be determined through mechanical and thermal sensitivity analysis. Hence an attempt is made specifically with nano powders of fuel under different composition by considering metallurgical and safety aspect. Pyrotechnic mixtures are energetic compounds susceptible to explosive degradation on ignition, impact and friction and are obtained by mixing finely divided (reducing) metal powders with inorganic oxidizing agents that are capable of undergoing self-sustaining combustion [1]. The compositions have a wide range of applications utilizing the production of light, heat, sound or smoke [2]. In firework industry, different metal/chemical powders are mixed in different proportions to attain explosive behaviour. Particle size, particle morphology and metal content are the vital properties which decide the explosivity of any explosive composition and the safety of handling is decided by these properties. The explosivity can be determined through mechanical and thermal sensitivity of a material. Hence an attempt is made specifically with nano powders on metallurgical and safety aspects.

Fireworks are of two types namely light-producing and sound-producing fireworks. Magnesium powder is frequently employed as a fuel for high light output and it is replaced by another metallic fuel in combination with sulphur for high sound output [3]. In firework crackers, an in-depth study carried out on the white powder composition consisting of Al, sulphur with different oxidizing agents like $KNO_3$, $BaNO_3$, $KClO_4$ etc. At present different chemical compositions of powders are being carried out with micron level [4]. The performance of the fireworks is decided by the sound level it produces. As per Govt. of India notification, crackers sound level should not exceed 125 dB (AI) or 145 dB(C) pk at 4-m distance from the point of bursting. This has been framed in order to standardize the amount of chemicals in authorized atom bomb manufacturing.

Some research studies have been carried out in Aluminium powder with coal and the results show that DSC studies on the effect of Aluminium particle size led to multiple exothermic activities and increase of Onset temperature [5]. The decrease in the Aluminium particle size increases the heat of reaction. This emphasises the fact that Al powder addition has made the mixture to ignite more quickly.

Unfortunately no research has been conducted to control the sound level with the use of nano powders in this field. With nano-size aluminium (nAl) particles, the specific surface area increases which will cause easier ignition and increased burn rates. In this study, nAl powders of three particle sizes have been employed in a mixture with the oxidizer and the igniter.

## Literature review

Aluminium particles have long been employed as a fuel ingredient in various energetic materials to enhance the energy density and stability characteristics of propulsion systems [6, 7]. Most of previous studies were focused on micron-sized particles [8–11], with very limited efforts devoted to the particle behaviour at nano scales [12, 13]. Nano-sized sulphur and oxidizers were synthesised by the ball mill method and the size was determined using a particle size analyzer. Pyrotechnic mixtures of compositions using five different oxidizers in different particle sizes, mixed with sulphur (S), aluminium (Al) and boric acid ($H_3BO_3$), were used to produce sound producing cake-bomb firecrackers for analysis. Decreasing the particle size from micro to nano improves the efficiency of firecrackers using the oxidizers and the limiting impact energy (LIE) of pyrotechnic compositions falls in the range of 2.55–4.51 J [14].

Aluminium powder is commonly added to explosives, propellants and pyrotechnic compositions to improve their performance; nAl powders have increasingly gained attention because of their potential incorporation in explosive and propellant mixtures. The DSC–TG results show the oxidation of Al nano particles occurs at least in two steps [15, 16]. The dry ball milling is carried out in a horizontal jar containing balls, powder and a process-controlling agent such as stearic acid under an inert gas atmosphere with a few % of oxygen, which can prevent explosion. The size and shape of the flake powder produced depend on the ball milling conditions such as the ball size, the number of intermediate stops and the amount of stearic acid [17, 18].

The addition of nano-sized energetic Al particles can greatly enhance the propellant burning rate because the reaction of the particles can occur in close proximity to the propellant's burning surface. The mass-burning rate per unit area increased from 1.00 to 5.80 g $cm^{-2}$ s and the results show that addition of high energy oxidiser and nano-sized aluminium exhibited improved impact sensitivity at levels that can be considered acceptable for deployment. Therefore, the burning rates of aluminized propellants (containing nano-sized aluminium particles) can be substantially increased when oxidizer-rich crystals are added to the propellant formulation [19]. Pre-ignition reaction is unique to nano-Al mixtures and attributed to the significantly higher specific surface area of the nanometric Al particles which provide increased sites for I-sorption [20, 21].

# Experimental

## Preparation of nano powders

The aluminium metal powder of various grades is readily available at Sivakasi, Tamilnadu, India. The purity of the aluminium metal powder is 85% and the remaining is aluminium oxide. Aluminium nano powder has so far been prepared by many processes like wire explosion process, vapour condensation methods, chemical methods and mechanical attrition [22, 23]. In this study, Fritsch, GmbH, 'Pulverisette 6' planetary mono mill was used for preparing different particle sizes of nAl powder. After grinding the 50-μm sized aluminium powder for 2, 5 and 8 h in the ball mill, the particle sizes of the output powder were measured by the particle size analyser (Make: Zetasizer Nano ZS). The details are shown in Fig. 1.

Typical scanning electron micrograph (SEM) images (Model: SU1510, Make: Hitachi High-Technologies (Singapore) Pte. Ltd.,) of different samples are shown in Fig. 2. It is deduced that the aluminium particle size is reduced by the increase in milling time. Meanwhile all the SEM pictures show the particles with irregular size and shape distributed throughout the matrix. This is due to the agglomeration of particles at low particle sizes. Sample D1 represents the SEM picture of one of the firework chemical mixture with large size particles of $KNO_3$ and S along with nAl.

## Preparation of pyrotechnic mixtures

Table 1 shows the basic formulation of the pyrotechnic mixtures with chemicals. Composition was prepared by using the Chemicals of $KNO_3$, S and various nAl powders.

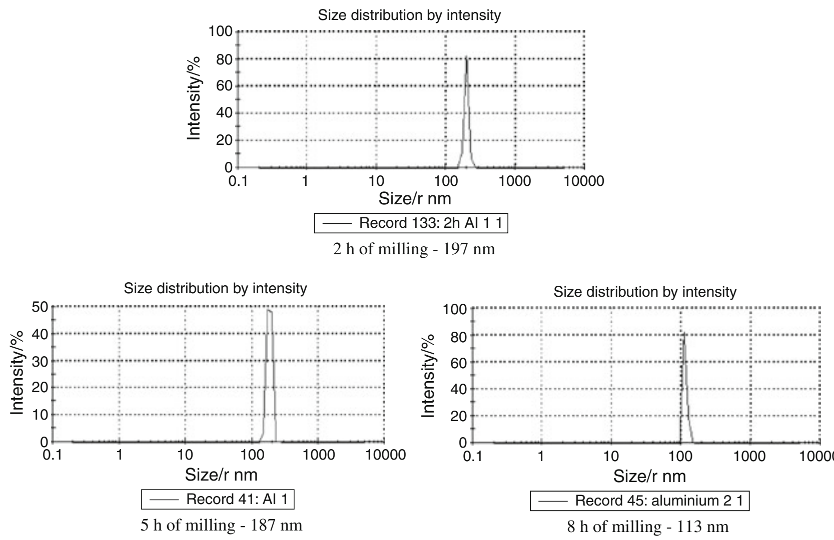



**Fig. 1** Particle size distribution of aluminium powder during various grinding time

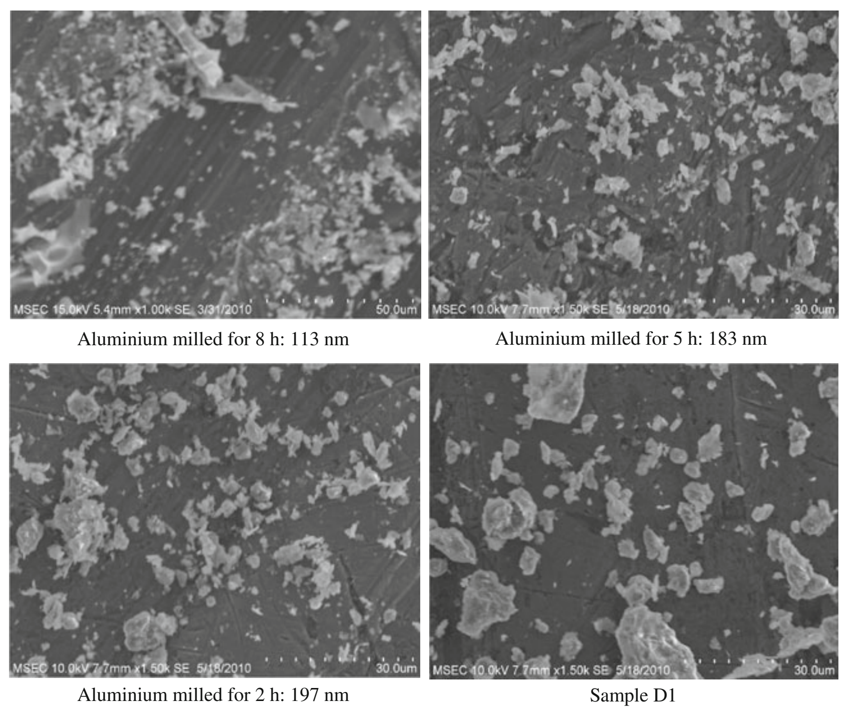



**Fig. 2** SEM images of nAl powders and sample D1

The purities of $KNO_3$ and S were 97.6 and 99.9%, respectively. Initially $KNO_3$ and S are sieved in the range of $-20+60$ and $-100+200$, respectively; here, the powder which can pass through 20 mesh is called $(-20)$ and the powder which is retained in 60 meshes $(+60)$. Then these powders are well mixed with nano aluminium powders of three different sizes, and sieved with 20 meshes. Then the compositions are mixed well and sieved with 20 meshes for 3–5 times to attain homogenous mixture. The homogeneous mixing was obtained using the mechanical aided mixture machine. The sieves used for this mixing meet the BSS standard. The number of mixtures required for an analysis was calculated with the help of Design of Experiment software.

## Cake bomb manufacturing

Cake bomb is one of the crackers manufactured in the firework industry which gives joy during festive celebrations. It is generally in cylindrical or square shape. It is made by filling the chemicals of $KNO_3$, S and Al on the Kraft paper (brown) with 240 GSM (gram per square metre) thickness. Gummed jute string of length 130–260 cm, and thin foil papers (cello paper) were used for making

**Table 1** Particle size and composition of various samples

| Sample no. | Composition/% | Particle size |
|---|---|---|
| Mass percentage of $KNO_3$:Al:S = 58:22:20 | | |
| A1 | 58.00 | −20 + 60 |
| | 22.00 | 197 nm |
| | 20.00 | −20 + 60 |
| A2 | 58.00 | −20 + 60 |
| | 22.00 | 183 nm |
| | 20.00 | −20 + 60 |
| A3 | 58.00 | −20 + 60 |
| | 22.00 | 113 nm |
| | 20.00 | −20 + 60 |
| B1 | 58.00 | −100 + 200 |
| | 22.00 | 197 nm |
| | 20.00 | −100 + 200 |
| B2 | 58.00 | −100 + 200 |
| | 22.00 | 183 nm |
| | 20.00 | −100 + 200 |
| B3 | 58.00 | −100 + 200 |
| | 22.00 | 113 nm |
| | 20.00 | −100 + 200 |
| Mass percentage of $KNO_3$:Al:S = 66:11:23 and 63:15:22 | | |
| C1 | 66.00 | −20 + 60 |
| | 11.00 | 197 nm |
| | 23.00 | −20 + 60 |
| C2 | 66.00 | −20 + 60 |
| | 11.00 | 183 nm |
| | 23.00 | −20 + 60 |
| C3 | 66.00 | −20 + 60 |
| | 11.00 | 113 nm |
| | 23.00 | −20 + 60 |
| D1 | 63.00 | −100 + 200 |
| | 15.00 | 197 nm |
| | 22.00 | −100 + 200 |
| D2 | 63.00 | −100 + 200 |
| | 15.00 | 183 nm |
| | 22.00 | −100 + 200 |
| D3 | 63.00 | −100 + 200 |
| | 15.00 | 113 nm |
| | 22.00 | −100 + 200 |
| Mass percentage of $KNO_3$:Al:S = 68:8:24 and 64:14:22 | | |
| E1 | 68.00 | −20 + 60 |
| | 8.00 | 197 nm |
| | 24.00 | −20 + 60 |
| E2 | 68.00 | −20 + 60 |
| | 8.00 | 183 nm |
| | 24.00 | −20 + 60 |
| E3 | 68.00 | −20 + 60 |
| | 8.00 | 113 nm |
| | 24.00 | −20 + 60 |
| F1 | 64.00 | −100 + 200 |
| | 14.00 | 197 nm |
| | 22.00 | −100 + 200 |
| F2 | 64.00 | −100 + 200 |
| | 14.00 | 183 nm |
| | 22.00 | −100 + 200 |
| F3 | 64.00 | −100 + 200 |
| | 14.00 | 113 nm |
| | 22.00 | −100 + 200 |
| Mass percentage of $KNO_3$:Al:S = 58:22:20 when Al is in µm | | |
| M1 | 58.00 | −20 + 60 |
| | 22.00 | 840 µm |
| | 20.00 | −20 + 60 |
| M2 | 58.00 | −100 + 200 |
| | 22.00 | 840 µm |
| | 20.00 | −100 + 200 |

firecrackers. Small size paper cases of 1.5 × 1.5 × 1.5 cm (3.4 $cm^3$) were used to prepare cake-bomb firecrackers similar to commercially available ones.

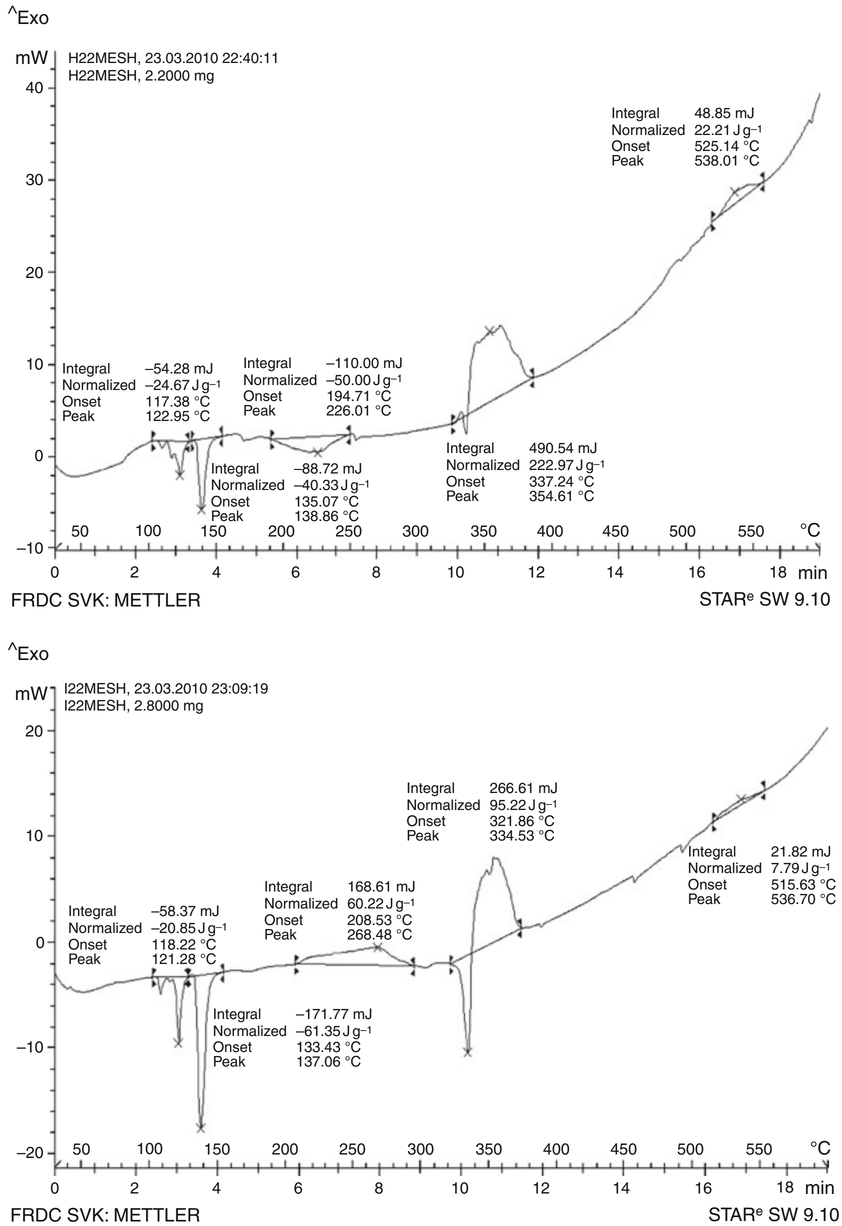
Cake-bomb firecrackers were manufactured manually by experienced technicians from the firework industries. The chemicals of different ratios were taken and sieved separately to remove the impurities and mixed thoroughly in a non conducting surface like newspaper, rubber mat etc. The mixture was again sieved four or five times to make it homogeneous and free from grits. The shells were taken and filled with the chemical mixtures. After some time, the gum coated jute string was wound around it tightly. It was dried in sun light for 2–3 h. Then the fuse wire was inserted and kept in place by charcoal powder.

## Thermal analysis

### *Differential scanning calorimetry*

All samples were analysed for their thermal properties in DSC (Make: Dupont, USA, Range: Ambient to 1000 °C, heating rate: 10–40 °C, Model: 2000). Figure 3 reveals that endothermic peak for melting of sulphur which occurs at 110 °C. Also decomposition starts around at 330–370 °C. For all the compositions irrespective of the particle sizes, the peaks appeared nearly at the same temperature. This shows that addition of nAl powder does not affect the decomposition temperature. However, particle size reduction affects the change of enthalpy during decomposition.

**Fig. 3** DSC graphs for the samples A3 and C3




*Thermogravimetry*

All samples were analysed for their thermal properties to study the mass loss during heating in TG/DTA (Make: Perkin Elmer, Range: Ambient to 1500 °C, heating rate: 10 °C, Balance type: Horizontal differential, Model: Diamond TG/DTA).

The activation energy, $E_a$, in kJ mol$^{-1}$ is expressed by the Kissinger relation [24]:

$$\ln\left(\frac{q}{T_{\max}^2}\right) = -\frac{E_a}{RT_{\max}} \quad (1)$$

where $q$, $T_{\max}$ and $R$ are the heat rate (K min$^{-1}$), the peak temperature (K) and the Gas constant (J mol$^{-1}$ K$^{-1}$). From Fig. 4, it is found that for the sample C3, the heat rate is 283 K min$^{-1}$, the peak temperature is 533 K and the gas constant is 8.314 J mol$^{-1}$ K$^{-1}$. By substituting the above values in Eq. 1, the activation energy for sample C3 is calculated as 30.63 kJ mol$^{-1}$. Some TG graphs are shown in the Fig. 4. The results of the thermal analysis are shown in Table 2.

Impact sensitivity analysis

Impact sensitivity of pyrotechnic mixture was tested using the BAM method by impact sensitivity tester, supplied by Electro Ceramic Private Limited, Pune, India [14, 15]. It consists of solenoid control and a release device. The release device holds the weight to be dropped. A cylindrical block of 2 kg is used to strike the sample from

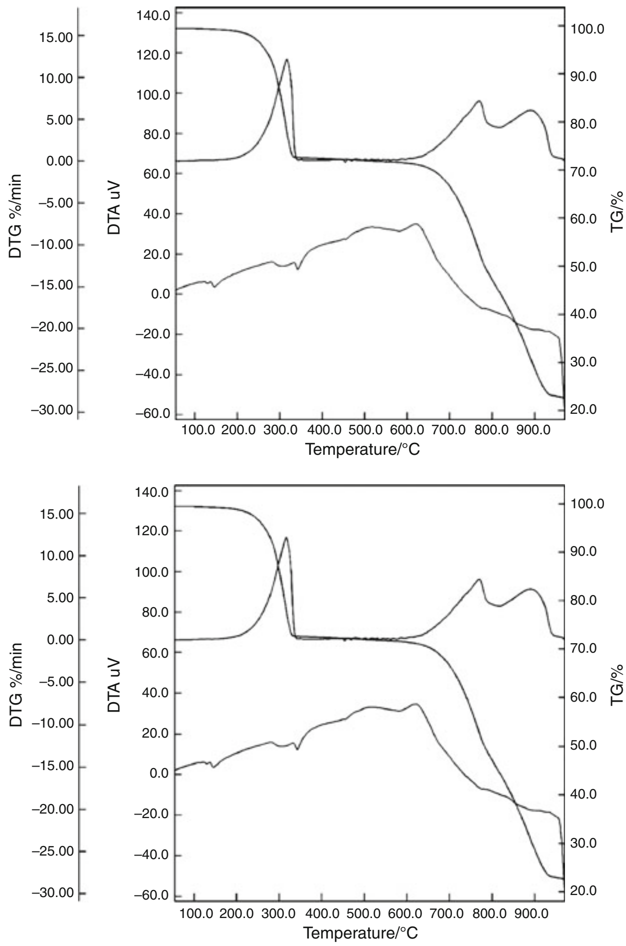



**Fig. 4** TG graphs for the samples E3 and C3

**Table 2** Thermal analysis of different pyrotechnic mixtures

| Sample no. | DSC | | | DTG | |
|---|---|---|---|---|---|
| | Onset temp./°C | Peak temp./°C | $\Delta H$/ J $g^{-1}$ | Peak temp./°C | Activation energy/kJ $mol^{-1}$ |
| A1 | 331.53 | 340.13 | 441.49 | 302.00 | 33.77 |
| A2 | 333.74 | 342.13 | 397.78 | 305.00 | 33.99 |
| A3 | 337.24 | 354.61 | 222.97 | 293.00 | 33.09 |
| B1 | 330.89 | 337.35 | 427.94 | 280.00 | 32.12 |
| B2 | 333.58 | 343.58 | 340.13 | 320.00 | 35.13 |
| B3 | 332.37 | 340.09 | 317.16 | 290.00 | 32.86 |
| C1 | 358.89 | 371.84 | 352.99 | 280.00 | 32.12 |
| C2 | 333.59 | 344.33 | 241.15 | 390.00 | 40.5 |
| C3 | 321.86 | 334.53 | 295.22 | 260.00 | 30.63 |
| D1 | 334.52 | 340.75 | 463.62 | 295.00 | 33.24 |
| D2 | 328.24 | 365.13 | 445.87 | 295.00 | 33.24 |
| D3 | 335.34 | 342.68 | 417.25 | 280.00 | 32.12 |
| E1 | 333.72 | 339.77 | 304.36 | 340.00 | 36.65 |
| E2 | 333.59 | 339.99 | 433.13 | 295.00 | 33.24 |
| E3 | 337.68 | 373.26 | 252.67 | 305.00 | 33.99 |
| F1 | 331.78 | 338.15 | 436.65 | 305.00 | 33.99 |
| F2 | 330.51 | 340.24 | 390.76 | 296.00 | 33.31 |
| F3 | 333.41 | 338.8 | 390.92 | 260.00 | 30.63 |
| M1 | 337.26 | 350.77 | 457.57 | 322.00 | 35.28 |
| M2 | 332.08 | 339.58 | 639.51 | 302.00 | 33.77 |

various heights. The design and principles of the equipment are similar to those of the BAM standard drop fall hammer equipment. Ten milligrammes of pyrotechnic mixture was taken and placed on the bottom anvil. The top anvil was placed above the mixture. Table 3 shows the results of the impact energy of the pyrotechnic composition of different powders.

The mechanical sensitivity measurement study shows that all the pyrotechnic flash composition mixtures with varying nAl powder were sensitive to impact. The LIE of the above compositions studied fell in the range of 2.6–4.6 J.

Noise level testing

Noise level test was carried out as per the rules of notification of PESO (Petroleum and Explosives Safety Organisation), formerly known as 'Department of Explosives', Govt. of India. The noise level was measured by four noise level monitors using Model no. 824L obtained from Larson and Davis, USA and the average value of the readings was taken as sound level data. The noise level was measured at 1.2 m elevation from the ground level of bursting at 4 m distance. The metres were placed in four places and each angle between them is 90°. A hard concrete surface of 5 m diametre was the site for the explosion, with no obstacle (like tree or any other structure) to carry out the noise level test.

As given in Table 4, three samples of cake bombs for each mixture were manufactured by varying the weight of the chemicals. The inner shell volume was taken to be constant. As the amounts of the chemical filled inside the crackers were varied, the net densities were different from sample to sample.

The results of all experiments performed by the author were evaluated with the use of different equipment based on the original experimental description. The results are same with negligible error. The repeatability of results is confirmed by conducting successive experiments. The description is so abstract that the readers cannot obtain anything new and concrete. If the reproducibility is discussed, more concrete description is needed.

## Results and discussion

From the thermal analysis, the impact sensitivity analysis and the noise level test, the activation energy, the impact

**Table 3** Impact energy of different powders

| Sample no. | Impact energy/J |
|---|---|
| A1 | 3.9 |
| A2 | 2.8 |
| A3 | 2.6 |
| B1 | 3.6 |
| B2 | 2.7 |
| B3 | 2.5 |
| C1 | 5.8 |
| C2 | 3.9 |
| C3 | 3.5 |
| D1 | 4.7 |
| D2 | 3.8 |
| D3 | 2.9 |
| E1 | 6.7 |
| E2 | 4.8 |
| E3 | 4.2 |
| F1 | 4.8 |
| F2 | 4.6 |
| F3 | 3.5 |
| M1 | 4.6 |
| M2 | 4.0 |

**Table 4** Comparison of noise level for pyrotechnic mixtures

| Sample no. | Chemicals in cake bomb/g | Noise level | |
|---|---|---|---|
| | | dB(AI) | $dB(C)_{peak}$ |
| A1 | 2.00 | 127.7 | 148.9 |
| | 1.75 | 125.8 | 147.3 |
| | 1.50 | 125.9 | 148.3 |
| A2 | 1.50 | 126.9 | 148.7 |
| | 1.25 | 126.9 | 148.0 |
| | 1.00 | 124.9 | 147.9 |
| A3 | 2.00 | 127.7 | 147.9 |
| | 1.75 | 127.3 | 145.9 |
| | 1.50 | 127.9 | 146.8 |
| B1 | 2.00 | 126.7 | 149.9 |
| | 1.75 | 126.6 | 148.3 |
| | 1.50 | 125.6 | 147.4 |
| B2 | 1.75 | 126.4 | 148.3 |
| | 1.50 | 126.5 | 146.3 |
| | 1.25 | 125.4 | 146.3 |
| B3 | 1.75 | 127.9 | 148.9 |
| | 1.50 | 127.9 | 148.3 |
| | 1.25 | 125.7 | 147.3 |
| C1 | 2.00 | 126.9 | 148.9 |
| | 1.75 | 126.8 | 148.0 |
| | 1.50 | 125.9 | 147.9 |
| C2 | 1.75 | 125.6 | 147.7 |
| | 1.50 | 124.6 | 144.8 |
| | 1.25 | 124.6 | 146.7 |
| C3 | 1.75 | 127.5 | 148.4 |
| | 1.50 | 125.7 | 146.0 |
| | 1.25 | 125.5 | 145.5 |
| D1 | 2.00 | 127.4 | 148.9 |
| | 1.75 | 126.9 | 148.9 |
| | 1.50 | 125.8 | 148.5 |
| D2 | 1.75 | 125.9 | 146.3 |
| | 1.50 | 125.6 | 146.0 |
| | 1.25 | 125.1 | 145.8 |
| D3 | 2.00 | 128.9 | 148.0 |
| | 1.75 | 127.9 | 147.8 |
| | 1.50 | 127.8 | 146.2 |
| E1 | 2.00 | 127.8 | 147.3 |
| | 1.75 | 126.9 | 145.3 |
| | 1.50 | 125.3 | 146.9 |
| E2 | 1.75 | 126.4 | 145.4 |
| | 1.50 | 126.8 | 147.3 |
| | 1.25 | 126.9 | 148.6 |
| E3 | 2.00 | 127.5 | 148.4 |
| | 1.75 | 127.1 | 148.2 |
| | 1.50 | 126.9 | 147.9 |
| F1 | 2.00 | 127.6 | 146.9 |
| | 1.75 | 126.2 | 146.5 |
| | 1.50 | 125.1 | 147.3 |
| F2 | 1.75 | 126.2 | 148.5 |
| | 1.50 | 125.5 | 147.4 |
| | 1.25 | 124.4 | 146.5 |
| F3 | 2.00 | 128.2 | 148.9 |
| | 1.75 | 127.9 | 147.4 |
| | 1.50 | 127.5 | 147.0 |
| M1 | 2.00 | 125.3 | 149.4 |
| | 1.50 | 120.5 | 138.1 |
| | 1.00 | 116.9 | 135.9 |
| M2 | 1.50 | 124.4 | 144.0 |
| | 1.25 | 119.6 | 138.9 |
| | 1.00 | 116.0 | 135.4 |

energy and the noise level were measured for the pyrotechnic samples containing nAl of the three particle sizes. These results are shown in Fig. 5.

The enthalpy change decreases monotonically in most cases, as shown in Fig. 5a. However, variation is high for the sample having the composition $KNO_3$:Al:S with

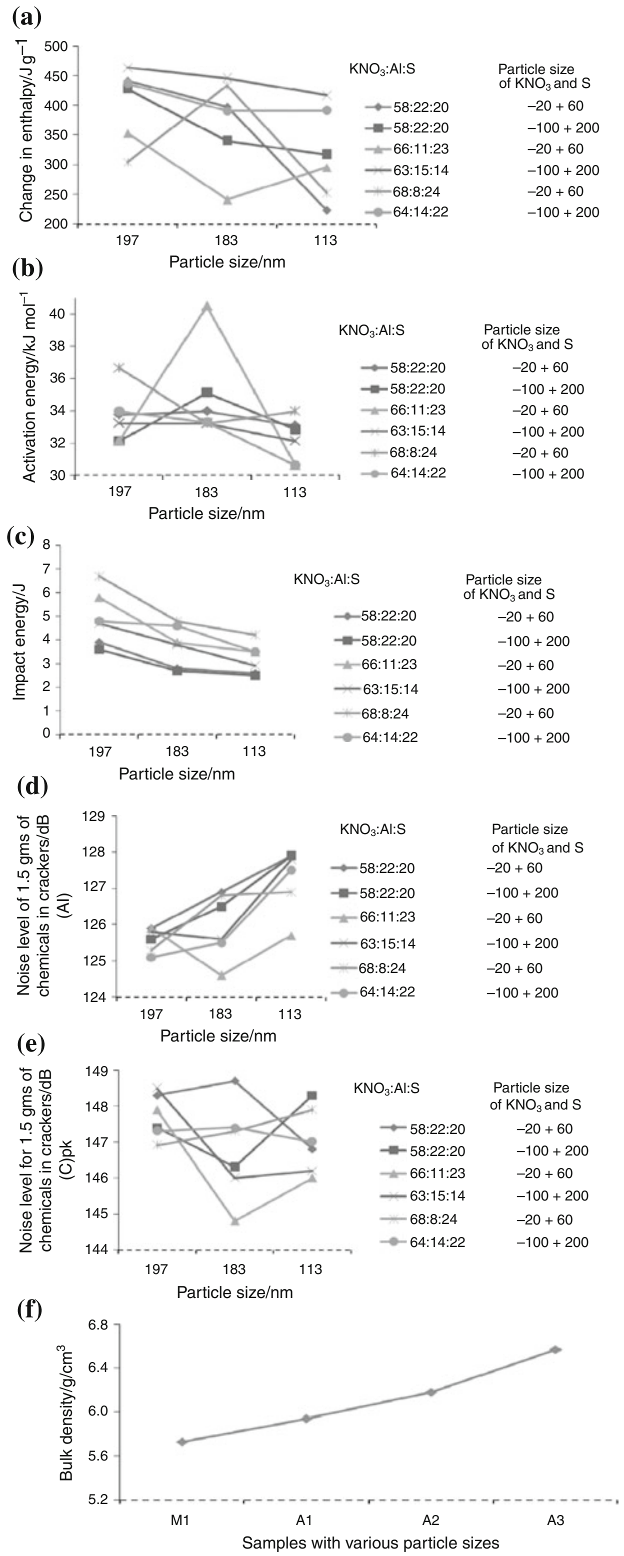



**Fig. 5** **a** Variation between change in enthalpy and nAl particle size. **b** Variation between activated energy and nAl particle size. **c** Variation between impact energy and nAl particle. **d** Variation between noise level in dB(AI) and nAl particle. **e** Variation between noise level in dB ($C_{pk}$) and nAl particle. **f** Variation between bulk density and particle size of Al powder

68:8:24, because of very low fuel content. The variations may be due to the inhomogeneous mixing and the variance in particle sizes.

From Fig. 5b, activation energy variation is not uniform (all the curves do not have similar patterns) for different mixtures. This may happen because of non uniform particle size distribution. Thus, it may be assumed that the activation energy is nearly constant independent of the particle size.

It is likely from Fig. 5c that the impact energies decrease monotonically with decreasing nAl powder sizes. This reveals that handling of nano powders will have risk and being utmost care. Further the composition of $KNO_3$:Al:S with ratio 68:8:24 performs better impact energy than other samples.

As shown in Fig. 5d and e, the noise level of the cake bomb increases with decreasing nAl powder size except for the case $KNO_3$:Al:S = 68:8:24. For the exception, this is presumably because of the relatively low content of nAl. Also from the above figures and Table 4, aluminium powdered cake bomb with nano particle size gives the threshold noise level of 125 dB(AI) and 145 dB(C pk) with 1.25 g instead of 2 g (62.5%) for the same composition when compared with aluminium particle size in µm. For example, sample cake bomb having the pyrotechnic mixture having µAl. (sample M1 produce 125.3 dB(AI) with 2 g of chemicals and mixture having nAl (sample A2) produce 126.9 dB(AI) with 1.25 g of chemicals.

Figure 5f reveals an obvious rise of bulk density when particle size decreases. A smaller particle can enter the vacant space created around irregularly shaped large particles without increasing the bulk volume.

## Conclusions

From the experimentation it is concluded as below:

- Onset temperature and peak temperature for the nano-sized powder has increased marginally during combustion of nano powders due to the high decomposition temperature and thus releasing more heat energy.
- Differential thermogravimetric result shows quick phase transition for nano powder and hence mass loss during the decomposition at an earlier stage.
- The nAl quantity of chemical for producing the optimum sound level in the cake bomb has reduced to 62.5% when compared to µm powder.
- The particle sizes of the oxidizer and igniter ($KNO_3$ and S) also influences the noise level of crackers considerably because they affect the burning rate.
- Moreover decreasing the particle size to nano level, the mechanical sensitivity has increased and may create the safety problem when handling.

- The result shows that magnificent combustion field and hence pollution will reduce to a great extent which contributes to growth and welfare of fireworks industry and society as a whole.

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