Effect Of Particle Size On The Mechanical Sensitivity And Thermal Stability Aspects Of Pyrotechnic Flash Compositions

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Abstract: *The mechanical and thermal sensitivity of pyrotechnic flash compositions consisting of mixtures of potassium nitrate (KNO_3), sulphur (S) and aluminum (Al) with varying particle sizes of KNO_3 and Al indicated that, irrespective of the composition of the cracker mixture, all the compositions were found to be thermally and mechanically sensitive. Although the impact sensitivity results reflected the change in the surface area of the particle sizes, the changes were within a narrow range of limiting impact energy (LIE) (7.5–9.1 J). Further it was difficult to pinpoint a particular sieve fraction as sensitive since the response to explosion depended normally on the flash composition and the particle size but also on the density and the compactness of the chemicals*

DSC studies on the effect of the Al particle size showed that a decrease in the Al particle size led to a second exothermic activity. This behavior should be viewed with caution when considering safety aspects.

Keywords: *pyrotechnics, flash composition, fireworks, impact sensitiveness, friction sensitiveness, differential scanning calorimeter, potassium nitrate, sulphur, aluminum*

Introduction

In recent years frequent incidents have been reported involving fireworks during their processing, storage and transportation.[1] Large quantities of different categories or types of fireworks are manufactured and demand for them is rising continuously. In general, the composition of fireworks is basically a mixture of sulphur, phosphorus, chlorates, nitrates and pure aluminum metal powder. These mixtures are highly sensitive to temperature, mechanical impact, pressure and friction. Knowledge of the thermal stability, auto-ignition temperature, impact sensitivity, frictional sensitivity and electrostatic sensitivity of these mixtures is required to assess their hazard potential as well as to make suitable plans for safety during processing, storage, and transportation.[2,3]

Reported information on the sensitivity to thermal, mechanical and electrostatic hazards is scarce. Recently efforts have been made by Sivapirakasam *et al*. to study the thermal hazards and the impact sensitivity of various compositions of cracker mixtures.[4,5] The parameters that influence the mechanical and thermal sensitivity are particle size, purity, and moisture content of the chemicals and ambient conditions. Though there are a few reported studies on the effect of particle size on the mechanical sensitivity and thermal stability of some pyrotechnic mixtures,[6,7] pyrotechnic flash composition mixture consisting of KNO_3, S and Al has not yet been studied. This work attempts to report the effects of the particle sizes of KNO_3 (oxidizer) and Al (fuel) in pyrotechnic flash compositions on the mechanical sensitivity and thermal stability.

Particle sizes of the ingredients and their effects in pyrotechnics

Pyrotechnic compositions are sensitive to thermal and mechanical stimuli. The degree of the hazard depends upon the rate of availability of these energies in addition to the particle size of the components of the mixture, confinement and momentum (the force during impact). Each one of the factors has a significant role in enhancing the overall hazard potential of the mixture.

It has been reported[8] that the ease of ignition is greatly dependent on the particle size and surface area of the ingredients. Smaller particle sizes create large interfacial areas, and increased numbers of atoms at the particle interface which on ignition lead to a higher heat of reaction. These effects however are not actually consistent due to the results of various physical and chemical processes occurring concomitantly and in many instances competitively. This is particularly true where an endothermic phase change absorbs some of the heat of combustion.[9]

When the particles in a pyrotechnic mixture are small and jagged, less energy input (*via* impact, friction and thermal stimuli) is needed to produce hot spots. This is because energy is localized at the stress points. Also, dislocations, cracks and other discontinuities in the crystal structure are conducive to the formation of hot spots.[10] Ignition is achieved when the energy released from the initiating hot spots is sufficient in quantity and is efficiently captured by the adjacent layers or grains of blended chemical compositions.[11]

Experimental

Materials

The flash compositions were prepared from materials purchased from SD's fine chemicals, Mumbai. KNO_3 and S were of high purity (AnalaR) grade and Al was of LR grade.

Preparation of sieve fractions of KNO_3 and Al

The sieve fractions of KNO_3 and Al were prepared using the standard sieves obtained from Jayant Scientific Industries, Mumbai, India. Particles of KNO_3 passed through a 63 µm sieve mesh and collected in a 53 µm sieve mesh were termed as +53 µm sieved fractions. Similarly, five other sieve fractions of +63 µm, +75 µm, +90 µm, +105 µm, and +150 µm were prepared. The details of the sieved fractions separated for the study are summarized in Table 1. Similarly Al was separated into 5 sieved fractions and the details are given in Table 2.

In order to examine the effect of varying the particle size of KNO_3 on the sensitiveness of a flash composition, the sieved fraction of KNO_3 was mixed with the other two components *i.e.*, S, Al. The particles of S and Al were passed through a 100 mesh brass sieve. Similarly, in order to examine the effect of varying particle sizes of Al on the sensitiveness of a flash composition, the sieved fraction of Al was mixed with the other two components *i.e.* KNO_3 and S. The particles of KNO_3 and S were passed through a 100 mesh brass sieve. Tables 3, 4, 5 and 6 show the various flash compositions and the corresponding mixture fractions taken for studies.

Table 1 *Particle sizes of KNO_3.*

Sample No.	Particle Size of KNO_3
1	+ 53 µm (53 to 63 µm)
2	+63 µm (63 to 75 µm)
3	+75 µm (75 to 90 µm)
4	+90 µm (90 to 105 µm)
5	+105 µm (105 to 150 µm)
6	+150 µm (150 to 200 µm)

Table 2 *Particle sizes of Al.*

Sample No.	Particle Size of Al
1	+37 µm (37 to 45 µm)
2	+45 µm (45 to 53 µm)
3	+53 µm (53 to 63 µm)
4	+63 µm (63 to 90 µm)
5	+90 µm (90 to 105 µm)

Measurement of impact sensitivity

Impact sensitivity measurements on flash compositions with varying KNO_3 and Al particle sizes were carried out according to the procedure outlined in the United Nations (UN) Recommendations on the transport of dangerous goods.[12] The design and principle of the equipment were similar to those of a drop fall hammer of BAM standards. The details of the equipment employed in this study are presented elsewhere.[4]

Measurement of friction sensitivity

Friction sensitivity measurements on flash compositions with varying KNO_3 and Al particle sizes were carried out by BAM (friction tester) according to the procedure outlined in the United Nations (UN) Recommendations on the transport of dangerous goods.[13]

Thermal studies

A Differential Scanning Calorimetry (DSC) module 2910 model from TA Instruments was used for thermal stability measurements for the flash compositions with different KNO_3 and Al particle sizes. The studies were conducted with a sample size of 2 mg under pure nitrogen gas at a flow rate of 100 µl min^{-1}. The equipment and the experimental conditions employed have been reported in detail elsewhere.[5]

Results and Discussion

Effect of KNO_3 particle size on impact sensitivity

Impact sensitivity testing results for 3 different flash composition mixture ratios consisting of KNO_3, S and Al with varying KNO_3 particle sizes in the range of 53–150 µm are shown in Figure 1. The plot shows that all the mixture compositions studied were impact sensitive and Limiting Impact Energy (LIE) was in the range of 7.5 to 9.1 J. Hence, these mixtures could be grouped as category III explosives according to the classification of Andreiev-Beliave.[14,15] From Figure 1 it can be seen that an increase in the particle size of KNO_3 decreased the sensitivity to impact initially up to 63 µm and then increased it. This trend was due to the fact that impact sensitivity depends not only on the flash composition and the particle size but also on the particle shape, density and compactness of the chemicals.

Effect of Al particle size on impact sensitivity

Figure 2 shows the impact on the flash composition mixtures with varying Al particle sizes. The flash composition mixtures with the lowest particle sizes showed high sensitivity compared with those with higher particle sizes. This trend could be attributed to the increase in surface area as the Al particle size decreased. All the mixtures studied were impact sensitive and varied within a narrow range of LIE (8.5–11.5 J) and hence the flash composition mixtures could be grouped as category III explosives as per the classification of Andreiev-Beliave.[14,15]

Effect of KNO_3 particle size on friction sensitivity

Figure 3 shows the effect of KNO_3 particle size on the friction sensitivity of various flash composition mixtures consisting of KNO_3, S and Al. The friction sensitivity decreased with increasing KNO_3 particle sizes. The varying particle size of KNO_3 set the lowest friction load of 144 N for the flash composition studied. KNO_3 being an oxidizer, the effect of particle size was appreciable.

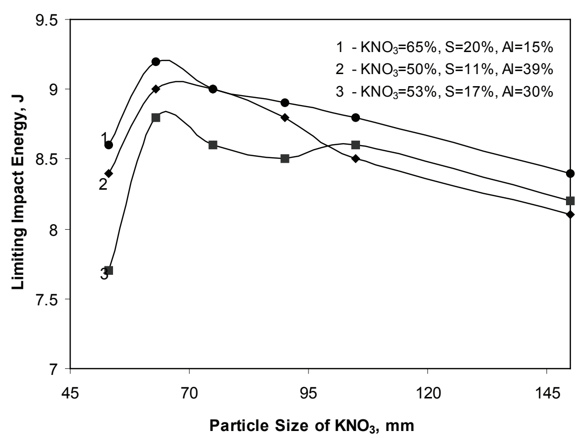

Figure 1 *Effect of KNO_3 particle size of flash compositions on the impact sensitivity.*

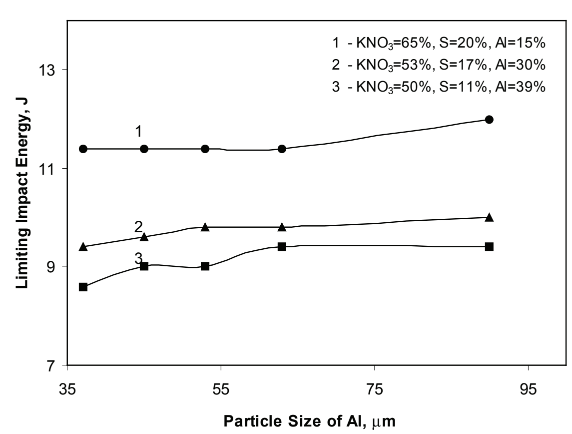

Figure 2 *Effect of Al particle size of flash compositions on the impact sensitivity.*

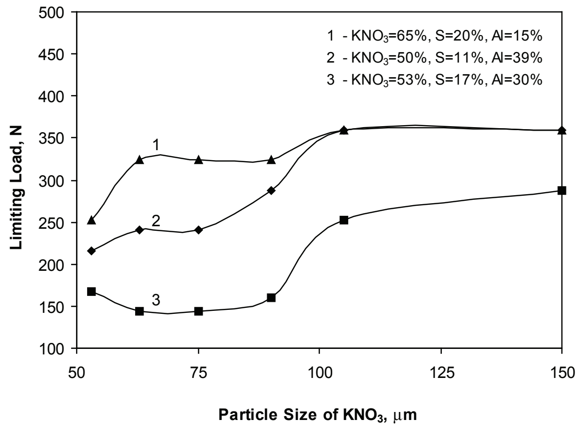

Figure 3 *Effect of KNO_3 particle size of flash compositions on the friction sensitivity.*

Effect of particle size of Al on friction sensitivity

The effect of Al particle size on the friction sensitivity of flash composition is presented in Figure 4. Although these mixtures were shown to be sensitive to friction there was no appreciable change with the change in Al particle size.

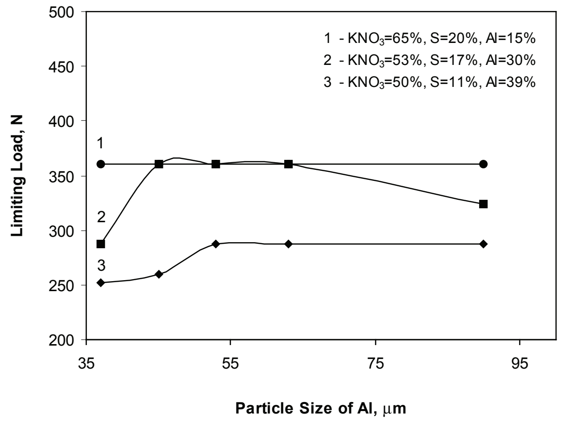

Figure 4 *Effect of Al particle size of flash compositions on the friction sensitivity.*

Effect of KNO_3 particle size on thermal stability

Table 5 shows the influence of KNO_3 particle size on the thermal decomposition of a pyrotechnic mixture. This is followed using DSC plots stacked according to the varying particle sizes (Figures 5, 6 and 7). In general, for all the flash compositions, the exothermic peak recorded between 299 °C

Table 3 *Effect of KNO_3 particle size for the various flash composition mixtures on mechanical sensitivity.*

Composition (wt%)	Particle Size (µm)	Limiting Impact Energy (J)	Frictional Limiting Load (N)
	53	8.4	216
KNO_3 = 50%	63	9.0	240
S = 11%	75	9.0	240
Al = 39%	90	8.8	288
	105	8.4	360
	150	8.0	360
	53	7.7	168
KNO_3 = 53%	63	8.8	144
S = 17%	75	8.6	144
Al = 30%	90	8.5	160
	105	8.6	252
	150	8.2	288
	53	8.6	252
KNO_3 = 65%	63	9.3	324
S = 20%	75	9.0	324
Al = 15%	90	8.9	324
	105	8.8	360
	150	8.6	360

Table 4 *Effect of Al particle size for the various flash composition mixtures on mechanical sensitivity.*

Composition (wt%)	Particle Size (μm)	Limiting Impact Energy (J)	Frictional Limiting Load (N)
	37	8.6	252
KNO_3 = 50%	45	9.0	260
S = 11%	53	9.0	288
Al = 39%	63	9.4	288
	90	9.4	288
	37	9.4	288
KNO_3 = 53%	45	9.6	360
S = 17%	53	9.8	360
Al = 30%	63	9.8	360
	90	10	324
	37	11.3	360
KNO_3 = 65%	45	11.3	360
S = 20%	53	11.3	360
Al = 15%	63	11.3	360
	90	11.9	360

Table 5 *Effect of KNO_3 particle size for the various flash composition mixtures on thermal sensitivity.*

Composition (wt%)	Particle Size of KNO_3 (μm)	Onset Temperature (ºC)	Peak Maximum Temperature (ºC)	Heat of Reaction (J g^{-1})	End Temperature (ºC)
	53	300.16	316.57	30.25	320.21
	63	319.66	316.57	48.93	329.90
KNO_3 = 50%	75	306.78	320.84	85.39	324.06
S = 11%	90	227.67	299.03.	697.8	314.31
Al = 39%	105	312.88	324.15	73.51	327.07
	150	310.47	320.36	41.44	324.28
	53	224.08	315.56	778.9	318.56
	63	312.2	326.12	290.6	330.52
KNO_3 = 53%	75	310.38	324.74	116.2	327.06
S =17%	90	313.79	328.80	232.6	331.13
Al = 30%		*331.84	*333.04	*28.16	*338.31
	105	315.53	326.22	78.03	329.57
	150	314.63	325.59	71.02	328.48
	53	314.38	327.20	336.7	330.64
KNO_3 = 65%	63	314.98	323.78	101.2	326.48
S = 20%	75	313.03	326.82	223.2	329.8
Al = 15%	90	314.3	326.63	146.2	329.65
	105	315.42	322.27	56.65	330.42
	150	314.27	328.63	195.3	330.92

* represents the DSC data for the second exothermic activity

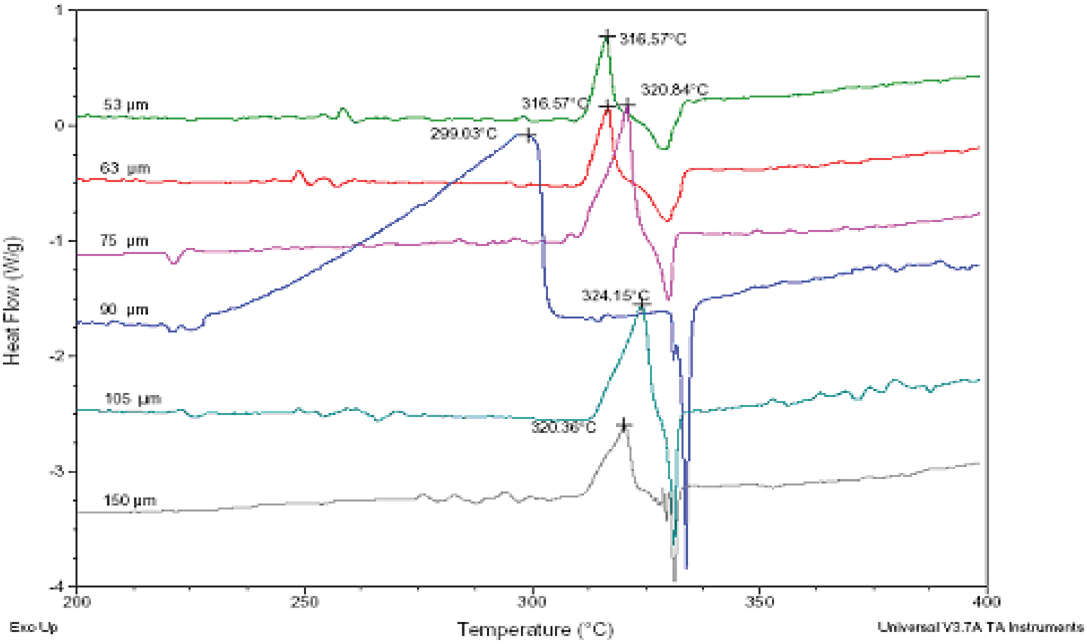

Figure 5 *DSC Plots showing the influence of KNO_3 particle size on thermal decomposition of a flash composition (KNO_3 = 50%, S = 11%, Al = 39%).*

and 333 °C was due to the prominent exothermic decomposition process. The concomitant endothermic peak with a peak maximum of 331 °C was due to melting (phase change) of unreacted KNO_3. Though the degree of exothermic activity was different with varying particle size, there was no specific trend or influence of particle size on the thermal decomposition.

A close examination of the DSC plot revealed that the decomposition process was a result of the physical and chemical processes occurring concomitantly. It appeared that the initial reaction process occurred in the solid phase (220 °C to 330 °C), until the endothermic melting of KNO_3, and hence the decomposition reaction could not proceed further at this temperature. The possibility of the existence of sulphur was remote and hence the decomposition process ended at this point.

The DSC plots (Figure 5) for the flash compositions consisting of 50% KNO_3, 11% S and 39% Al with varying particle sizes show a number of features in common, except for the one carried out with KNO_3 of 90 µm particle size. The 90 µm particle size mixture showed an early onset of thermal decomposition (227 °C) and the heat released was quite high as compared to the DSC plots of the remaining mixtures of varying particle sizes. Perhaps this oddity is because the flash compositions depend also on the particle shape and compactness of the chemical components. Though the changes in particle size in the mixture composition had a definite influence on the thermal decomposition, it was difficult to conclude which particle size and composition was the most ideal for flash composition manufacturing.

The influence of KNO_3 particle size on the thermal decomposition of a pyrotechnic mixture consisting of 53% KNO_3, 17% S and 30% Al was studied using DSC and the resulting plots were stacked according to the varying particle sizes (Figure 6). In case of the 53–63 µm sieve fraction, there was an earlier onset temperature for decomposition with the release of a high heat of reaction.

The influence of KNO_3 particle size on the thermal decomposition of a pyrotechnic mixture consisting of 65% KNO_3, 20% S and 15% Al was also studied using DSC and the resulting plots were stacked according to increasing particle size (Figure 7). This showed similar features to what was seen in the earlier compositions. Though a change was observed on increased KNO_3 particle sizes, the effect was not very significant.

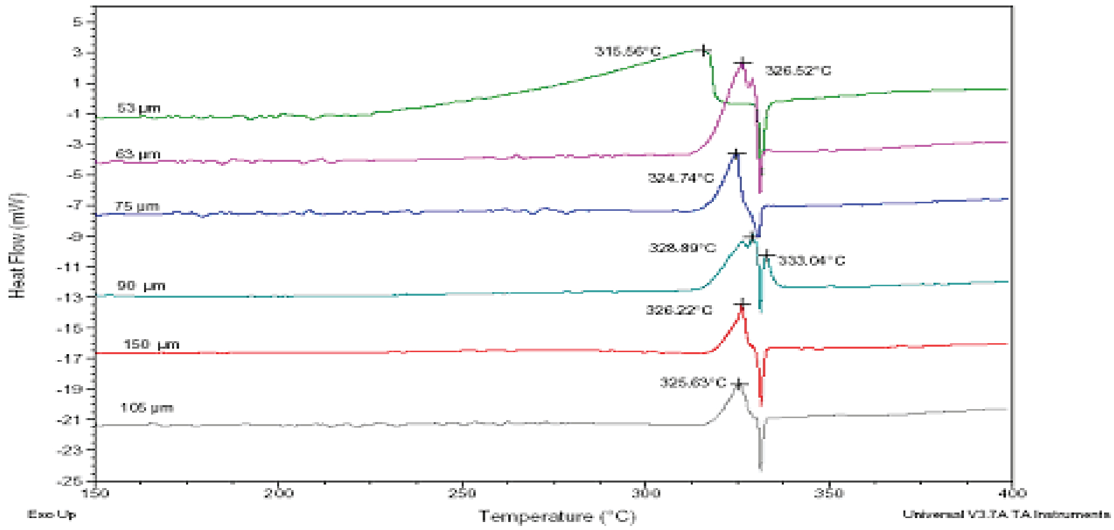

Figure 6 *DSC plots showing the influence of KNO_3 particle size on thermal decomposition of a flash composition (KNO_3 = 53%, S = 17%, Al = 30%).*

Effect of Al particle size on the thermal stability

Table 6 shows the influence of Al particle size on the thermal decomposition of the pyrotechnic mixture. The resulting plots were stacked according to the varying particle sizes (Figures 8, 9, 10). The onset of decomposition was generally found to be around 320 ºC for all particle sizes and the first process ended around 330 ºC. Here, in most cases, a second exotherm was observed immediately next to the endotherm at 331 ºC. The endothermic process recorded at the peak maximum temperature of 331 ºC was the melting of KNO_3 [reference Merck index]. DSC plots

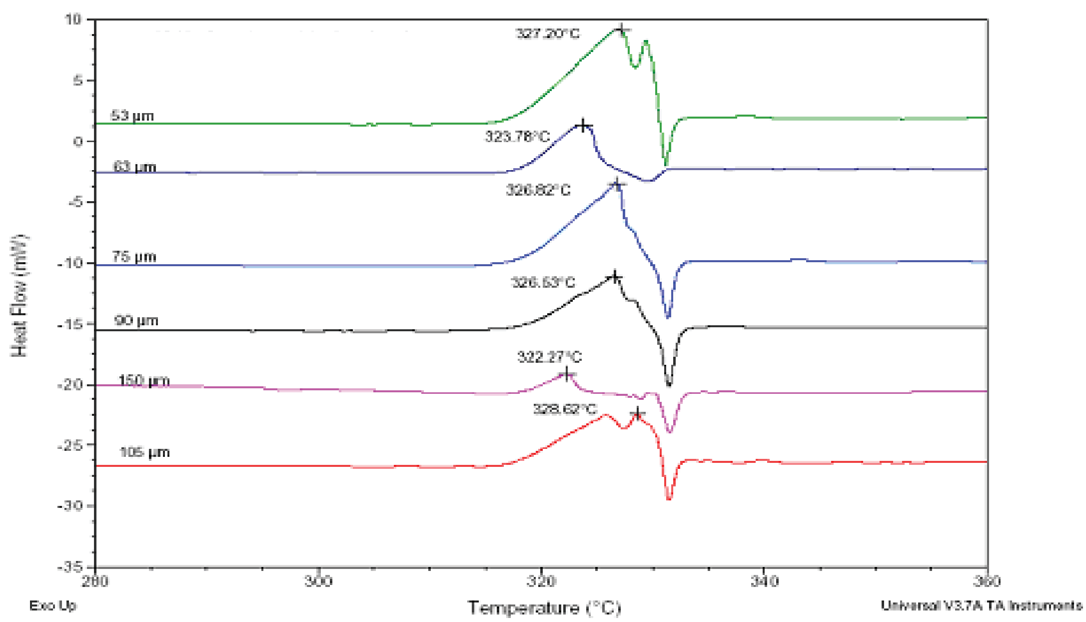

Figure 7 *DSC plots showing the influence of KNO_3 particle size on thermal decomposition of a flash composition (KNO_3 = 65%, S = 20%, Al = 15%).*

Table 6 *Effect of particle size of Al for the various mixtures of flash compositions on thermal sensitivity.*

Composition (wt%)	Particle Size of Al (μm)	Onset Temperature (ºC)	Peak Maximum Temperature (ºC)	Heat of Reaction (J g^{-1})	End Temperature (ºC)
KNO_3 = 50% S = 11% Al = 39%	37	220.03	332.75	263.2	331.8
	45	319.26	325.68	42.24	329.93
	53	319.82	326.65	27.84	330.77
		*331.54	*332.28	*11.03	*338.74
	63	320.62	325.84	47.65	329.91
	90	315.9	322.95	34.15	329.65
KNO_3 =53% S =17% Al =30%	37	318.71	326.82	76.7	330.95
		*332.25	*333.45	*12.5	*337.49
	45	312.10	329.77	105.5	330.87
		*332.15	*333.04	*81.43	*345.84
	53	316.78	328.84	101.5	330.82
		*317.28	*333.73	*23.43	*338.64
	63	319.91	326.24	66.5	330.9
	90	321.54	326.07	37.46	330.81
		*331.67	*332.68	*17.75	*339.99
KNO_3 =65% S =20% Al = 15%	37	317.69	326.35	79.87	329.91
	45	311.23	332.75	1007	353.77
	53	315.68	325.76	88.6	330.91
		*332.21	*334.29	*103.9	*353.54
	63	324.6	330.41	44.88	331.71
		*332.66	*338.45	*240.6	*363.17
	90	325	331.48	4.84	332.25
		*334.95	*343.07	*81.81	*353.72

* represents the DSC data for the second exothermic activity

also revealed that the decomposition process was the result of physical and chemical processes occurring concomitantly. The effect of particle size seemed to be inconsistent. It appeared that the initial reaction was taking place in the solid phase (220–330 ºC), until the endothermic melting of KNO_3. The concomitant reaction after KNO_3 melting appears to proceed in the liquid phase.

Figure 8 shows the DSC plot for flash compositions consisting of 50% KNO_3, 11% S and 39% Al with varying Al particle sizes. The second concomitant reaction could not be observed, because the first reaction could not provide enough enthalpy for the second reaction to proceed due to absorbance of the heat by the endothermic phase change. However, in some cases the second exothermic reaction could proceed. From Table 6, it was observed that the onset temperature increased with increase in particle size and remained constant. The decrease in the Al particle size increased the heat of reaction.

The influence of Al particle size on the thermal decomposition of pyrotechnic mixture consisting of 53% KNO_3, 17% S and 30% Al was studied using DSC and the resulting plots were stacked according to increasing particle size (Figure 9). In all the DSC runs with different particle sizes, the endothermic melting of potassium nitrate with concomitant second exothermic activity followed the first prominent exothermic peak.

The influence of Al particle size on the thermal decomposition of pyrotechnic mixture consisting

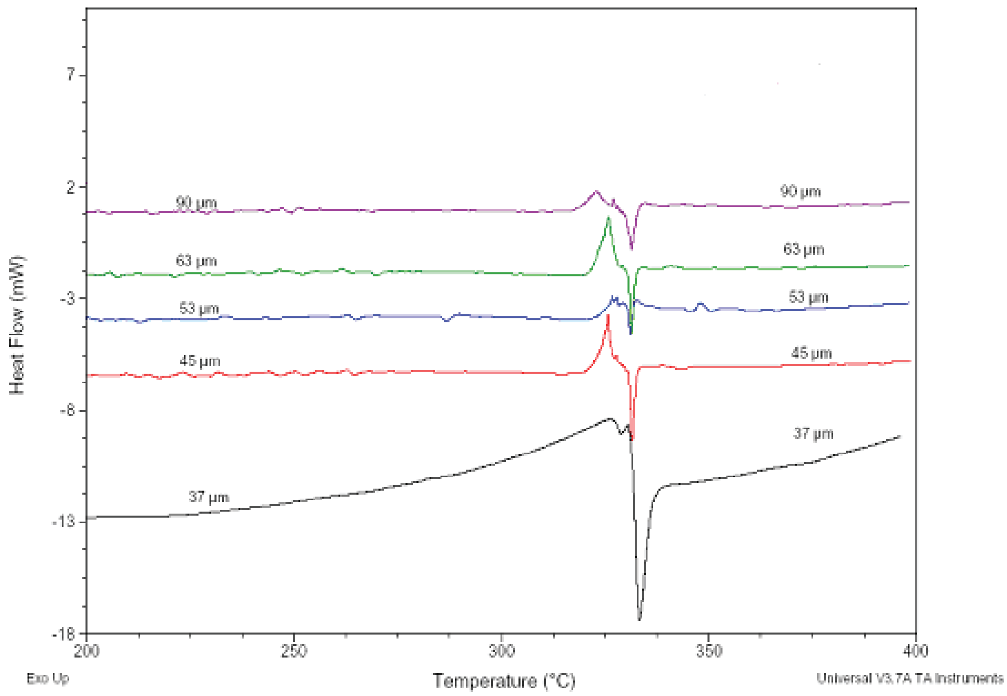

Figure 8 *DSC plots showing the influence of Al particle size on thermal decomposition of a flash composition (KNO_3 = 50%, S = 11%, Al = 39%).*

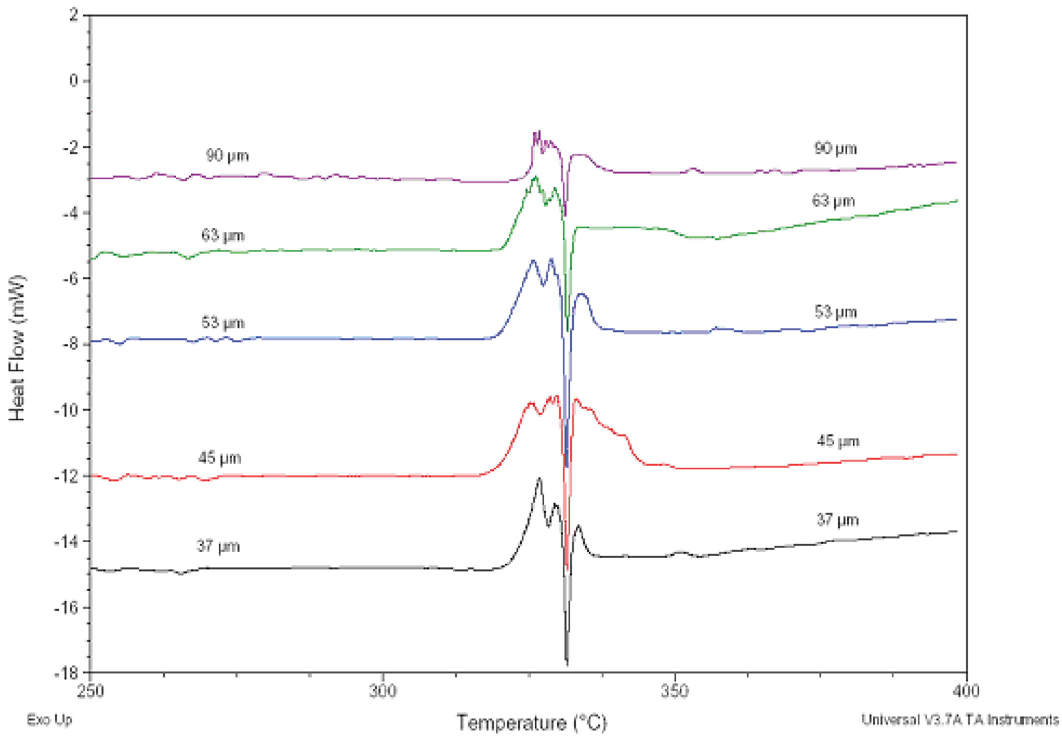

Figure 9 *DSC plots showing the influence of Al particle size on thermal decomposition of a flash composition (KNO_3 = 53%, S = 17%, Al = 30%).*

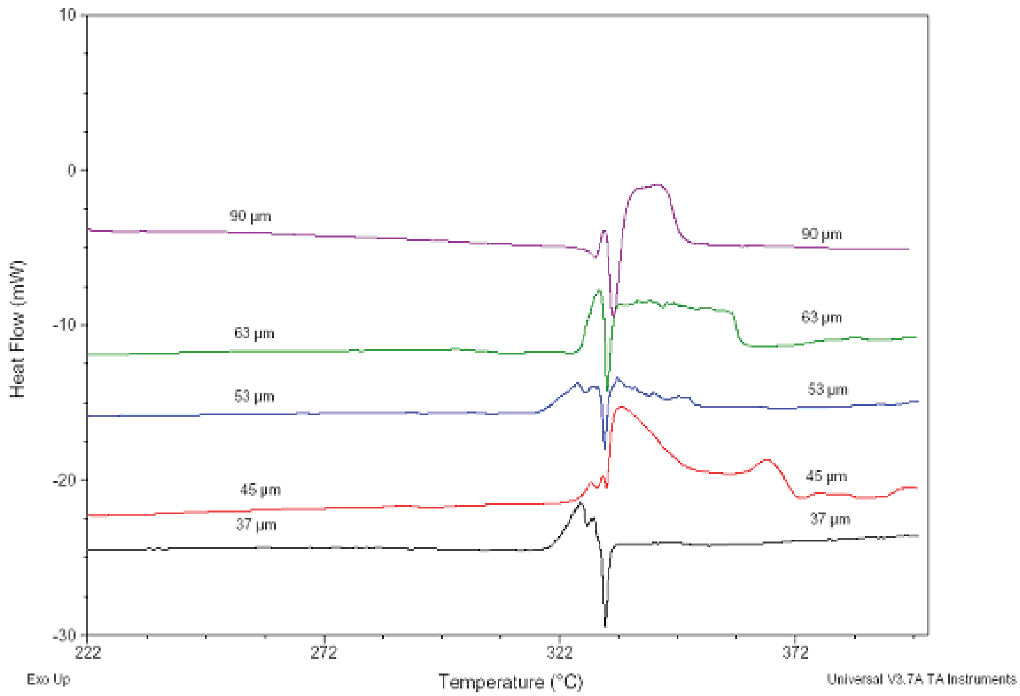

Figure 10 *DSC plots showing the influence of Al particle size on thermal decomposition of a flash composition (KNO_3 = 65%, S = 20%, Al = 15%).*

of 65% KNO_3, 20% S and 15% Al was studied using DSC and the resulting plots were stacked according to increasing particle size (Figure 10). The trend of the curve was similar to the two previous compositions except for the one with particle size of 45 µm. It was very interesting to note that the concentration of KNO_3 was the highest in the mixture and that all of them were involved in the thermal decomposition processes. As KNO_3 was the oxidizer in the flash composition a maximum amount of heat release was expected. The results are complex and it is difficult to interpret the role of each component in promoting the decomposition reaction.

Effect of Particle Size of KNO_3 (oxidizer) and Al (fuel) on Process Safety of Flash Composition

The mechanical sensitivity analysis showed that the decrease in particle size of KNO_3 had an adverse effect on sensitivity (both impact and friction) to a greater extent than the Al particle size had. However, the flash compositions studied could be categorized as class III explosives sensitive to impact.

The DSC studies showed that the decrease in particle size of KNO_3 and Al set the lowest onsets at around 220 ºC for the first exothermic activity. The chance of thermal hazard during the processing of flash compositions below the onset temperature of DSC, though remote, had to be so confirmed through the adiabatic calorimetric test. Given a margin of 100 ºC, it would be stated that below 120 ºC, the flash composition mixture would be stable provided there was no source of ignition or mechanical (impact/friction) effects of the flash composition.

The DSC studies on the effect of Al particle size showed that the decrease in Al particle size leads to a second exothermic activity. This behavior needs to be viewed with caution from the point of view of safety. The heat content from the first exothermic activity itself is enough to undergo good flash reaction by the mixture,[5] whereas the heat release due to the second exothermic activity is undesirable for the flash composition as this may lead to several cascading explosions. However, in order to improve the quality of cracking of flash composition, firework manufacturers generally tend to use finely divided Al powder in greater quantities than necessary. DSC studies revealed that the use of finer Al powders in a flash composition would only lead to secondary exothermic activity culminating in accident situations of higher magnitude. This secondary exothermic activity can lead to secondary explosions in any hazardous situation during processing and storage of flash compositions as the quantities are going to be high.

Conclusions

Mechanical and thermal studies on the sensitivity of pyrotechnic flash compositions with varying KNO_3 and Al particle sizes indicated that all the flash compositions studied were found to be sensitive. As the KNO_3 particle size increased, the sensitiveness to impact initially decreased up to 63 µm and then increased. This was due to the fact that impact sensitivity depended not only on the flash composition and particle size but also on the particle shape, density and compactness of the chemicals. The friction sensitivity decreased with the increase in KNO_3 particle size. The lowest Al particle size exhibited high impact sensitivity compared with the higher particle sizes. But there was no appreciable effect on friction sensitivity with change in Al particle size.

The DSC studies on the effect of Al particle size showed that the decrease in Al particle size led to a second exothermic activity. Hence, the use of finer Al powders in a flash composition would only lead to secondary exothermic activity. This secondary exothermic activity can lead to secondary explosions in any hazardous situation during processing and storage of flash compositions.

Acknowledgements

S. P. Sivapirakasam is grateful to the management and Principal, Mepco Schlenk Engineering College, Sivakasi, for their constant encouragement. The author is also grateful to TIFAC, Department of Science and Technology, Government of India, New Delhi, for offering facilities to carry out this research. The authors are thankful to the Director, CLRI for their kind permission to carry out this study in CLRI.

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