Development and Experimental Investigation on Delay Time Consistency of Modified Si/PbO/Pb_3O_4/FG Pyrotechnic Delay Composition

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Abstract—**In the present study, experimental investigation was carried out on the delay time consistency of modified Si/PbO/Pb_3O_4/FG pyrotechnic delay composition in a delay tube. Where Si is the fuel, PbO/Pb_3O_4 are oxidizers and Fish Glue (FG) is the binder. Ingredient mixing and loading pressure were studied. Results revealed that homogenous mixing of the delay composition is a very critical parameter for controlling the time consistency of pyrotechnic delay composition. The delay time accuracy was improved from 25% to about 7.42% by ensuring homogenous mixing of the ingredients. Results also show that loading pressure ranged from 30,000 to 65,000 psi did not affect much the delay time of this pyrotechnic composition and the burning rate.**

Keywords-Delay Composition; Loading pressure; Time consistency; Delay time; Burning rate

I. INTRODUCTION

Pyrotechnic is a general term for a mixture that burns at a selected, reproducible rate, providing a predetermined time delay between ignition and delivery of main effects [1, 2]. The modern practice requires pyrotechnic delays to function very accurately. This has led to an intensive research into the mechanism and combustion control of both gas producing and gasless pyrotechnic mixtures. Moreover, it has narrowed the field of investigation to the relatively few combustible materials that can be considered. Pyrotechnic compositions are mixtures of ingredients, which usually are not themselves explosive, and are designed to burn but not to detonate. Typical burning rate of pyrotechnics can vary from below 1 mm/sec to over 1,000 mm/sec. They are commonly used as delay devices in sophisticated missile systems to provide the required delay time before performing certain functions and to detonate the warhead after the predetermined delay time. The end result of any weapon or missile system is greatly dependent on accurate and consistent delay time. Propagation or burning of a pyrotechnic delay device depends on a number of factors such as confinement, particle size, fuel percentage, diameter, thickness, conductivity and geometry of the delay body, nature and ratio of ingredients, initial temperature, ambient temperature, ambient pressure, consolidation pressure, moisture, terminal charge, ignition composition, delay body material, storage condition, charge increment, additives, intimate contacts of ingredients etc. [3-8]. Repeatability and accuracy of any pyrotechnic delay device depends on design and development of the pyrotechnic delay composition which in turn depends on mixing, ingredient particle size, grain size and type, internal diameter and length of the delay tube. The main problem in pyrotechnic delay compositions is the accuracy ranging from ±10% to ±20% of the average value over the normally military operating temperature of -40^{o}C to + 60^{o}C [10]. In some pyrotechnics the time delay increased up to 25% from mean at -54°C [11-13]. The main objectives of this research were: (i) The development of a modified fast pyrotechnic delay composition comprising Si as fuel, PbO/Pb_3O_4 as oxidizers and fish glue as a binder. (ii) The experimental investigation of the effects of mixing and loading pressure on delay time consistency of this pyrotechnic delay composition in a pyrotechnic delay device.

II. EXPERIMENTAL PART

A. Materials

Analytical grade Fuel Silicon Powder and Oxidizers Lead (II) Oxide (PbO) and Lead Oxide Pb_3O_4 purchased from Sigma Aldrich Company and commercial grade fish glue binder were used during this research work. The purity of these ingredients was greater than 99%. The particle size of Si, PbO and Pb_3O_4 were ≤44 µm, 1~2 µm and < 10 µm respectively. Distillation was used for the preparation of binder solution.

B. Preparation of Pyrotechnic Delay Composition

Two methods were used for the preparation of the pyrotechnic delay composition.

1) (3-D) Automatic Tumbler Mixing Machine

The different chemicals (fuels and oxidizer) were individually dried out in a heating oven at 80°C for two hours to remove the moisture. The required quantities of the different ingredients were weighed according to the required percentages and blended thoroughly. The ingredients were mixed in the 3-D automatic tumbler mixing machine for 3 hours. A binder solution of 0.3% fish glue was prepared in distilled water. The binder solution was mixed in the already mixed composition to form a homogenous paste by using the spatula in agate container. Then the composition was semi dried in the drying oven at 80 °C. Semi dried composition was broken in to lumps by spatula in an agate container carefully. The composition was passed carefully through -212 mesh sieves to get grain size of ≤ 65 μm. Then the grains dried out for four hours at 80 °C to remove the water/moisture. The finished composition was stored in container and placed it in desiccator.

2) Manual Mixing Mortar and Pestle:

The individual chemicals (fuels and oxidizer) were dried in a heating oven at 80°C for two hours to remove the moisture. The chemicals were weighted according to the required percentages. Small batches of 5 gram were prepared and the chemicals were mixed in mortar and pestle for 30 minute to get homogenous mixture. Then a binder solution of 0.3% Fish Glue was prepared in distilled water. The binder solution was mixed in the already mixed composition to form the homogenous paste by using spatula in agate container. Then the composition (paste) semi dried in the drying oven at 80 °C. The semi dried composition was broken into lumps by spatula in an agate container carefully. The composition passed carefully through -212 mesh sieves by using Haver test shaker EML 200 to get grain size of ≤ 65 μm. [14, 15]. Then the grains dried for eight hours at 80 °C to remove the water/moisture. The finished composition was stored in container and placed in desiccator for 24 hours to become stable.

3) Safety During Manual Preparation of Delay Composition:

This delay composition is very sensitive to friction, especially during manual mixing/grinding of the ingredients in the mortar and pestle and at the time of grain formation when the composition is passed through a particular sieve to get the desired grain size. The following safety precautions are very essential in order to avoid injuries:

- Ensure the exposure of minimum number of operators during processing steps
- As this is a manual process, the quantity of pyrotechnic composition for each batch was kept to 5 grams in order to minimize the risks.
- Use specially designed Mortar and Pestle
- Use bullet proof screen while dry mixing the composition
- Use goggle and gloves while handling and preparing the composition

4) Filling of delay body:

Stainless steel delay elements were made by drilling 0.157 inch diameter holes through stainless steel rods. The tube length was kept at 0.787 inches. The finished composition was then loaded into stainless steel delay tube in five equal increments. Each increment was accurately weighted separately and pressed in a delay tube one by one. The surface of the delay column was kept flushed with the end of the delay tube. Excess delay composition was removed by sliding the end of the delay tube. A hydraulic press machine installed with a calibrated gauge was used to consolidate the delay composition in the delay tube. At compaction loads from 10,000 to 65,000 psi, the pyrotechnic mixture ignited and burned in a satisfactory manner. Consequently, for most of the tubes prepared in this way, the pressure was set at approximately 30,000 psi. A dwell time of about 2 s was used before relieving the stress. A mechanically initiate percussion assembled in the holder was then installed in the delay tube. A rubber O-ring was used to prevent the composition from environmental effect. The other end of the delay body was crimped.

C. Testing and Measurement:

The percussion cap was hit with a striking pin. The firing mechanism is an electromechanical switch. The delay time starts when the pin hits the percussion and stops when the Light Dependent Resistor (Sensor) detects the flame of the delay composition when it is completely burnt. In order to protect the senor from the slag a Perspex disc was installed in front of it. The delay time was measured with a digital Oscilloscope. Channel 01 of the oscilloscope was connected with the firing switch and Channel 02 was connected to the detector. A minimum of five measurements for each composition was kept. The mean delay time and mean burning rate were measured. Maximum variation from mean and standard deviation were calculated.

III. RESULTS AND DISCUSSION

$PbO/Pb_3O_4/Si/FG$ is a gasless delay mixture. The propagative burning of pressed column of this gasless delay composition is a combustion reaction in which PbO, Pb_3O_4 and Si react to give solid products. Some of the heat produced during the chemical reactions is lost through the delay body to the surrounding and the remaining heat is transferred to the unreacted mixture through conduction that sustains propagation. The variation in the inner diameter and length of the delay tube was kept at minimum. The ingredients and the percentages used during preparation of this modified delay composition are shown in Table I.

TABLE I. INGREDIENTS AND THEIR PERCENTAGE

S#	Ingredients	Percentage
1	Silicon	18.0
2	PbO	21.0
3	Pb_3O_4	60.7
4	Fish Glue	0.30

A. Effect of Ingredient Mixing on Delay Time

1) Test Results of Composition Mixed in (3-D) Automatic Tumbler Mixing Machine

The delay composition was prepared in a 3-D Automatic Tumbler Mixing Machine and then loaded in a delay tube of 0.157 in internal diameter. The length of the column was 0.787 in. The burning rate, delay time along with maximum variation in delay time and burning rate from mean are presented in Table II. All the tests were conducted at temperature range between 18~24 C°. Results are summarized in Table II. As shown, the mean delay time and burning rate are 0.569 sec and 1.395 in/sec. The maximum variation in delay time and burning rate are 23% and 24.3% respectively. These variations in delay time and burning rate are very high. It means that the delay composition prepared in the 3-D automatic tumbler mixing machine did not produce consistent delay time. The reason could be the non-uniform mixing of the composition ingredients.

TABLE II. TEST RESULTS OF DELAY MIXTURE MIXED IN (3-D) AUTOMATIC TUMBLER MIXING MACHINE

Sample No	Delay time (sec)	Burning rate (inch/sec)
1	0.588	1.339
2	0.626	1.258
3	0.536	1.469
4	0.493	1.597
5	0.600	1.312
Mean	0.569	1.395
Max variation from mean	23%	24.3%

The delay composition was then subjected to different loading pressures in the delay column in order to determine the effect of loading pressure on the delay composition in the delay tube. For this reason different loading pressures of 10,000, 12,000, 14,000, 20,000, 24,000 and 40,000 psi were applied to consolidate the composition in the delay column. Pressing the delay composition in the column increases compaction and hence increases density. Results in Figure 1 show that the burning rate and hence the delay time was not much effected by changing the applied loading pressure. The maximum variation in delay time from mean at different loading pressures ranged from 15% to 25%. This variation in delay time though not very large is still very high considering our goal. The conclusion being that the time consistency of this delay mixture could not be achieved by varying the loading pressure. Figure 2 also shows the mean delay time and mean burning rate at different loading pressures. The burning time and burning rate varied from 0.569 to 0.715 sec and 1.011 to 1.384 inch/sec respectively. The delay time and burning rate are inversely proportional to each other, when the delay time reduced the burning rate increased and vice versa. These results reveal that mixing through tumbler mixing machine is useful where large amounts are required to be prepared and where delay time consistency is not required.

2) Test Results of Composition Manually Mixed in Mortar and Pestle.

The ingredients of the pyrotechnic composition were mixed in mortar and pestle, the mixing procedure been discussed in Section II.B.2. The composition was then loaded in the delay body with internal diameter and length of 0.157 and 0.787 in respectively. The composition was then consolidated at 30,000 psi by using oil operated hydraulic press with calibrated pressure gauge. Dwell time of two sec was applied for each increment.

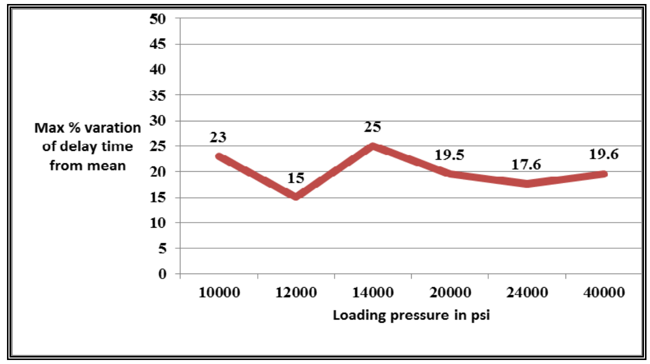

Fig. 1. Plot of Maximum percent variation of delay time from mean against applied loading pressure

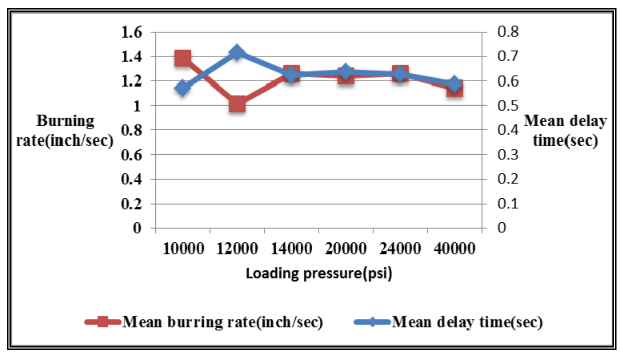

Fig. 2. Plot of burning rate and delay time against applied loading pressure

Results in Figures 3 and 4 show that, the variation in delay time of each sample range from 0.162% to 4.376%. Results also show that burning rate of the delay mixture varies from 1.222 in/sec to 1.335 in/sec. The mean delay time and burning rate are 0.617 sec and 1.276 in/sec respectively. The standard deviation in delay time and burning rate are 1.316 and 0.0269 respectively. The maximum variation in delay time reduced from 25% to 8.75%. The intimate contact of the fuel and oxidizers resulted in the reduction in delay time variation. The intimate contact was ensured through manual dry mixing in mortar and pestle followed by wet mixing. Additionally, the laboratory operating conditions i.e. controlling dry mixing time, wet mixing, drying time, stabilizing the composition for a specific time, controlling the weighing of the individual ingredients, controlling the weighing of delay composition, using calibrated measurement equipment were also ensured to be the same each time. Homogeneity and intimate contacts of ingredients played a key role in time reproducibility of this pyrotechnic delay composition. Intimate ingredient contact was ensured by dry mixing of fine and uniform particle sizes of the fuel and oxidizers followed by wet mixing with the binder solution. Dry mixing of the ingredients is not a very safe method as compared to automatic mixing, therefore safety precautions were ensured during this critical operation. The composition was made slurry which was then stirred thoroughly to ensure even distribution of the fuel and oxidizers. The fuels and oxidizers have different densities and if binder is not used then the fuel and oxidizer separate due to their density difference. The main reason of adding fish glue as binder is to protect the fuel and oxidizer from environment effects such as humidity. Binder also increases cohesion between particles of fuels and oxidizers to protect them from being segregated due to their density difference. The grains also provided ease of loading of the composition in the cartridge body.

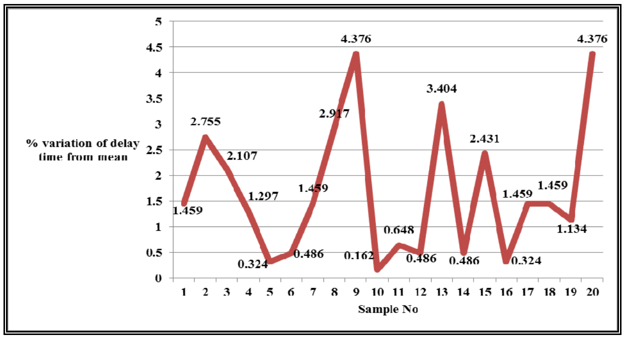

Fig. 3. Plot of percent variation of delay time from mean for each sample

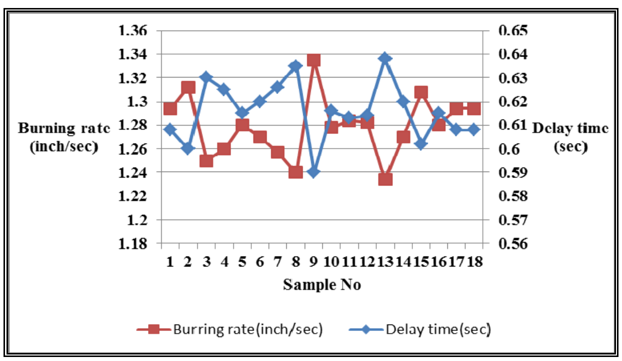

Fig. 4. Plot of burning rate and delay time for each sample

3) Effect of Loading Pressure on Delay Composition (Manually Mixed in Mortar and Pestle)

The data presented in Table III, show that the maximum variation in delay time is from 7.42% to 9.38% by varying the loading pressure from 20,000 to 65,000 psi. It means that if the composition is homogenously mixed, then this pyrotechnic delay composition is not much effected by the loading pressure and hence the burning rate is relatively insensitive to loading pressure. For varying loading pressure from 20,000 to 65,000 psi, only about 2% variation in mean delay time was recorded. Results in Table III also show that even at 65,000 psi, the composition did not become insensitive, ignited by the standard percussion primer and functioned successfully.

IV. CONCLUSIONS

The result analysis showed that ingredient mixing along with thorough controlling of the laboratory operating conditions are critical parameters for controlling the delay time variation and burning rate of pyrotechnic delay composition in a delay device provided that the variation in internal bore size and length of the delay tube is controlled. The manual mixing is much better than mixing in automatic tumbler mixer because the tumbler mixer did not produce intimate contact of the ingredients. The delay time and burning rate of this pyrotechnic delay composition was not much effected by the varying of the loading pressure. Only about 2% variation in mean delay time was recorded when the loading pressure varied from 30,000 to 65,000 psi. These experimental results can be applied as design guidelines for the developing of any pyrotechnic delay composition.

TABLE III. TEST RESULTS OF DELAY TIME AT DIFFERENT LOADING PRESSURES

S #	Mean delay time (sec)	Maximum % Variation in delay time	Mean burring rate (inch/sec)	Loading pressure (psi)
1	0.635	8.66	1.240	20000
2	0.629	9.38	1.252	24000
3	0.617	8.75	1.276	30000
4	0.634	7.42	1.242	60000
5	0.618	7.93	1.274	65000

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