

Effect of Silicon Contents on Different Oxidizers Used in Delay Composition

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Summary: Study was undertaken on the mass consumption and burning time of ranges of Si/Pb₃O₄/FG and Si/PbO/FG pyrotechnic delay compositions by varying Silicon Content. Silicon is a fuel; PbO and Pb₃O₄ are oxidizers whereas Fish Glue is a binder. The mass consumption and burning time of both these compositions was determined with different silicon contents. The Silicon content was varied from 5 wt% to 55 wt% for Si/Pb₃O₄/FG delay mixture, whereas for Si/PbO/FG delay composition the Silicon content was varied from 5 wt% to 40 wt%. The mass consumption of Si/Pb₃O₄/FG composition increases and burning time decreases with increase in fuel content until the maximum mass consumption of 1.080 mg/ms and minimum burning time of 300 ms were recorded at 35 wt. % Silicon Content. Similarly, the mass consumption of Si/PbO/FG delay composition also increases and burning time decreases with increase in Silicon content but beyond 15 wt% Silicon content the delay mixture shows unpredictable burning propagation behavior. Study was also conducted on the effect of fast red lead delay mixture as first fire charge to improve the burning performance of Si/PbO/FG delay compositions.

Keywords: Delay Composition, Mass consumption, Burning time, Fuel, Oxidant, Fish glue.

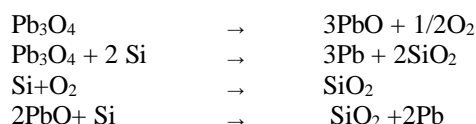
Introduction

Pyrotechnics delay composition comprises a fuel, oxidizer usually in combination with a binder and solvent to form slurry. Then, the slurry is dried to obtain a solid product. This solid product is then transformed into desired grains sizes. The burning time of a pyrotechnic delay composition is generally changed by varying fuel and oxidizer, the main ingredients of a delay composition. A pyrotechnic reaction is an exothermic. Pyrotechnic delay composition is a mixture that burns at an intended and reproducible rate, providing a pre-determined delay time between ignition and main effect. Time consistency is the most importance parameter while designing any pyrotechnic delay compositions. Different types of fuels like Si, B, Sb, W and Oxidants like PbO, Pb₃O₄, Ba₂CrO₄, KMnO₄, KClO₄, K₂Cr₂O₇, and Bi₂O₃ are used in pyrotechnic delay composition [1-4]. The fuel is selected as an energetic material which liberated a sufficient amount of heat when oxidized. Fuels can be categorized as metal, nonmetal or organic compound. Binder is added for granulation and it also may reduce sensitivity and improve stability of pyrotechnic delay composition.

Delay compositions are classified as fast burning, medium burning and slow burning according to their burning rate and type of fuel. Delay compositions are either gassy which produce large volume of gas on combustion greater than 20 cc/g and gasless delay composition which generate little gas on combustion, less than 5 cc/g [5-

6]. Quality Control of the ingredients and mixing of the compositions is very important together with stoichiometry which effect burning rate and other factors. Burning rate depends on particle size, confinement, impurities, material of delay tube, environmental conditions including temperature, ambient pressure and humidity, pressing load, binder, ingredients and their ratios etc. [7-13].

Si-Pb₃O₄ delay compositional is a fast burning delay composition. Lead oxide reacts with Silicon to give solid products SiO₂ and Pb [14, 15]. The reported reaction of Silicon with red lead oxide is given below.



To the best of our knowledge, very limited data is available on Si-PbO and Si-Pb₃O₄ delay compositions. Moreover not much study is available in the reported literature on the use of binder in these types of pyrotechnic delay compositions. Fish Glue has been used for the first time as binder in both these delay compositions. The aim of the present study was:

To develop Si-Pb₃O₄-FG and Si- PbO-FG pyrotechnic delay compositions and to carry out experimental investigation on the mass consumption

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and burning time of Si-Pb₃O₄-FG and Si- PbO-FG pyrotechnic delay compositions by variation the Silicon content.

1. Due to variation in Si content, density of the delay composition changes. So for same column length the charge weight cannot be kept constant. This study has been conducted to determine effect of varying the Si wt % on mass consumption and burning time of these delay compositions.
2. To enable the end-user to optimize the performance of these delay compositions for the indent applications.

Experimental

Materials

Analytical grade Silicon Powder, Lead (II) Oxide (PbO) and Lead Oxide Pb₃O₄ were purchased from Sigma Aldrich Company and commercial grade Fish Glue binder was used during this research work. The purity of these ingredients is $\geq 99\%$. The particle sizes of Silicon (Si), Lead (II) Oxide (PbO) and Lead Oxide Pb₃O₄ were $\leq 44\ \mu\text{m}$, $1\text{--}2\ \mu\text{m}$ and $< 10\ \mu\text{m}$ respectively.

Preparation of Pyrotechnic Delay Composition

Mixing of pyrotechnic delay ingredients

Before mixing the chemicals, the individual ingredients (fuels and oxidizer) were dried in a calibrated heating oven at $80\ ^\circ\text{C}$ for one hour to remove the moisture. These materials were dried in order to ensure accurate masses in delay mixture because different ingredients have different absorptivity. The Relative Humidity of the composition preparation room was maintained between 40 and 60 %. After weighing the chemicals with calibrated weighing balance, the ingredients were mixed in a (3-D) automatic Tumbler Mixing Machine. From the already mixed composition small batches of 5 g each were further processed by mixing the chemicals in Mortar and Pestle in a specially designed fuming hood for 30 min to further homogenize the compositions.

Grains formation of pyrotechnic delay compositions

Binders solution of 1.0 wt. % Fish Glue was prepared in distilled water followed by mixing the binder solution in the already mixed composition (Para 2.2.1). A homogenous paste was prepared by

using the spatula in agate container. Semi dried the composition (paste) in the Drying oven at $80\ ^\circ\text{C}$. To avoid the formation of lumps, the semi dried composition was broken by spatula in a special container carefully. The composition was sieved gently through 212 mesh sieves to get grains sizes of $\leq 65\ \mu\text{m}$. An automatically operated test shaker was used for preparation of grains of required particle sizes [16]. The grains were dried for four hours at $80\ ^\circ\text{C}$ to remove the water/moisture. Stored the finished composition in special container and placed it in desiccators for 24 h to stabilize the compositions.

Safety during mixing

Since these compositions are very sensitive to friction especially during the dry mixing and grains formation processes, therefore the following safety precautions were implemented during dry mixing and grain formation to avoid any accidental initiation of these compositions.

- Ensured minimum exposure of operators to the hazards of pyrotechnic mixture during processing steps.
- The quantity of pyrotechnic composition for each batch was kept 5 g to minimize the risks and to get intimate mixing.
- Used specially designed Mortar and Pestle.
- Used bullet proof screen while dry mixing the composition and passing the grain through sieves of required mesh sizes.
- Used Fire proof goggle and gloves while handling and preparing the composition.

Filling/Loading of composition in delay tube

The finished compositions were then loaded into stainless steel delay tube of 4.0 mm internal diameter. These increments were pressed in a delay tube one by one. A hydraulic press machine installed with a calibrated gauge was used to consolidate the delay composition in the delay tube. Pressed each increment at 40000 psi in the delay tube

Testing and data recording:

Testing and data recording system consists of:

- Electromechanical firing switch
- Power supply
- Digital Oscilloscope
- Detector
- Light Dependent Resistor (Sensor)

The percussion cap of this mechanically initiated delay device was hit with a striking pin. The firing mechanism consists of an electromechanical switch. The delay time started when the pin hit the percussion and stopped when the Light Dependent Resistor (Sensor) detected the flame of the delay composition when it completely burnt. In order to protect the sensor from the slag Perspex disc was installed in front of it. The delay time was measured with digital Oscilloscope. The sensitivity of the Oscilloscope used was in microsecond while the measured results were in millisecond in order to ensure accuracy in delay time measurement. Channel 01 of oscilloscope was connected with the firing switch and Channel 02 of the oscilloscope was connected to the detector. Schematic diagram of testing and data recording is shown in Fig. 1

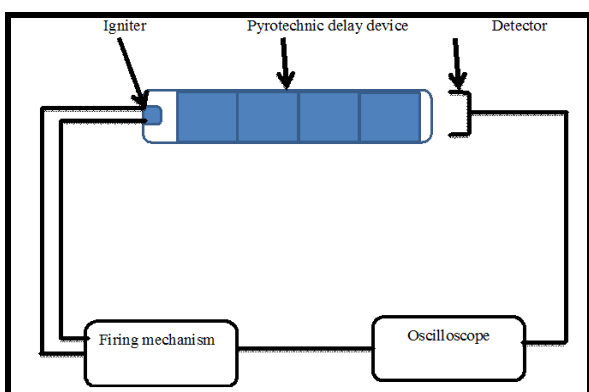


Fig. 1: Firing and delay measurement system of pyrotechnic delay device

Different conditions that influence the experiments

The burning performance and measurement results of pyrotechnic delay composition are affected by different laboratory operating conditions. The following conditions were controlled during composition preparations

- Both dry and wet mixing time was controlled
- Accurate weight of the individual ingredients and delay compositions was controlled and ensured to be the same every time through calibrated weighing balance.
- Relative humidity effect the performance of delay composition, therefore the Relative Humidity of the composition preparation was maintained between 40 and 60 %.
- The stabilization of the composition was ensured for specific time
- Calibrated pressure gauge was used during pressing the delay composition

- Variation in diameter of the delay body was controlled
- The accuracy of the measurement instruments is very important especially in short delay devices. Short pyrotechnic delay devices are in milliseconds ranges, therefore, the resolution of these measurement instruments was Nano second ranges to avoid motion blur.

Results and Discussion

Effect of Silicon content on Si/Pb₃O₄/FG delay mixtures

The mass consumption and burning time of Si/Pb₃O₄/FG delay mixture at different Silicon contents is shown in Table-1 and Fig 2. The Silicon content was varied from 05 wt.% to 55 wt.% in these delay mixture. The ingredients of the different delay mixtures are shown in Table-1. A total of nine delay mixtures with different Silicon contents were prepared. Five tests were conducted for each mixture in stainless steel tube of 4 mm internal diameter, the results were averaged. The loading pressure was kept 40000 psi to consolidate the delay mixture in the delay column. The mean values of mass consumption and burning rate were measured. The mean standard deviations of charge weight, burning time and mass consumption were also calculated. Fish Glue has been used as a binder in these delay compositions. Fish Glue as binder binds fuel and oxidizer together in the form of free flowing granules to provide ease of loading in the delay column. If binder is not used in a delay mixture, fuel and oxidizer segregates during storage due to the difference in their densities. Binder also protects the pyrotechnic compositions from environmental conditions. Moreover, binder protects the metal fuel from reacting with atmospheric oxygen.

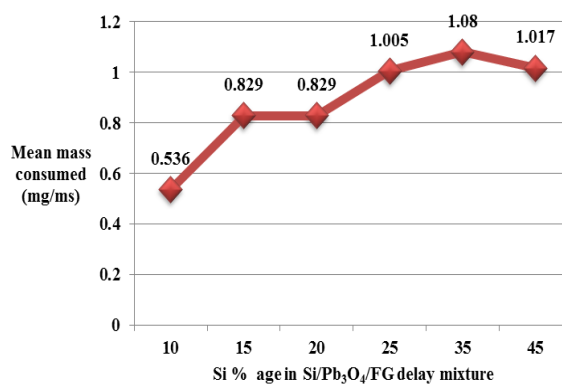


Fig 2 Effect of Silicon content on mass consumption of Si/Pb₃O₄/FG delay mixture

Table-1: Test results of Si/Pb₃O₄/ FG delay mixtures at different Silicon contents.

Pyrotechnic mixture	MCW	MBT	MCC	MSDCW	MSDBT	MSDCC
5.0% Si, 1.0 %FG, 94.0 % Pb ₃ O ₄	476	NR	NA	NA	NA	NA
10.0% Si, 1.0 %FG, 89.0 % Pb ₃ O ₄	355	663	0.536	5.51	6.52	0.022
15.0% Si, 1.0 %FG, 84.0 % Pb ₃ O ₄	378	456	0.829	3.97	4.83	0.009
20.0% Si, 1.0 %FG, 79.0 % Pb ₃ O ₄	380	441	0.880	5.45	5.12	0.012
25.0% Si, 1.0 %FG, 74.0 % Pb ₃ O ₄	392	390	1.005	4.26	6.88	0.021
35.0% Si, 1.0 %FG, 64.0 % Pb ₃ O ₄	323	300	1.080	6.77	6.35	0.034
45.0% Si, 1.0 %FG, 54.0 % Pb ₃ O ₄	349	343	1.017	5.22	10.08	0.036
50.0% Si, 1.0 %FG, 49.0 % Pb ₃ O ₄	340	NR	NA	NA	NA	NA
55.0% Si, 1.0 %FG, 44.0 % Pb ₃ O ₄	340	NR	NA	NA	NA	NA

Note. MCW = Mean charge weight (mg); MBT = Mean burning time (ms); MCC = Mean charge consumed (mg/s); MSDCW = Mean standard deviation in charge weight; MSDBT = Mean standard deviation in delay burning time; MSDCC = Mean standard deviation in charge consumed. NR = Not recorded. NA = Not applicable

Result shows that at 5.0 wt. % Silicon contents, the delay composition did not completely burn, partial burning was observed and the flame did not detect by the detector due to its low intensity, so the Si/Pb₃O₄/FG pyrotechnic delay composition at this ratio of fuel and oxidizer failed to produce reliable combustion propagation. In order to increase the sensitivity of this delay mixture at 5.0 wt. %, the loading pressure was reduced from 40000 to 20000 psi but the composition did not fire even at this decreased loading pressure, and similar result was observed. It means that the heat produced by the standard percussion was not enough to cause complete combustion of the delay mixture.

When the Silicon contents were increased from 5.0 wt% to 10 wt %, the delay mixture started reliable burning and complete burning propagation was observed, the mass consumption of 0.536 mg/ms was recorded, whereas the burning time recorded at 10 wt % was 663 ms. As the mass consumption increases, the burning time decreases because these are inversely proportion to each other. On further increasing the Silicon content to 15 wt%, the mass consumption increased to 0.829 mg/ms and burning time decreased to 456 ms, it means that the mass consumption increased by 55 % and burning time decreased by 31 %. On further increasing the Silicon content to 20 %, the mass consumption further increased to 0.880 mg/ms, which shows a further 6.0 % increase in mass consumption, whereas the burning time decreased from 456 ms to 441 ms, which shows 3.3 % decrease in burning time .

When the fuel content was increased to 25 %, the mass consumption also increased from 0.880 mg/ms to 1.005 mg/ms and burning time decreased from 441 to 390 ms, which show a further 14% increase in mass consumption and about 11.6 % decrease in burning time. The trend of increasing the mass consumption and decreasing the burning time of this delay mixture continued and the maximum mass consumption of 1.08 mg/ms and minimum burning time of 300 ms for Si/Pb₃O₄/FG pyrotechnic delay composition was recorded at 35 % Silicon content. A similar result had also been earlier reported by S.S. A.

Kazraji [14]. But in that reported work Corboxymethyle Cellulose (CMC) was used as binder, where as in this research work Fish Glue has been used as a binder. On further increase in fuel content to 45 %, the mass consumption started decreases and burning time increases until the composition fails to produce combustion propagation. The mass consumption of 1.017 mg/ms and burning time of 343 ms was recorded at 45 % Silicon content. At 50 % Silicon partial burning was observed and on further increasing the silicon content to 55 %, the composition did not initiate/ ignite by the percussion, so Si/Pb₃O₄/FG pyrotechnic delay composition at this ratio failed to be ignited by the standard percussion when hit with a spring loaded striking pin. At these fuel oxidizers ratio the sensitivity reduced and the energy produced by the percussion was not enough to initiate the delay composition and even partial burning was also not observed, the pressing load was then reduced from 40000 to 20000 psi in order to increase the sensitivity of the composition to standard percussion primer but similar result was observed and the composition failed to be ignited. Hence results show that this delay mixture produced reliable burning propagation from 10 wt% to 45 wt % and can be reliably used in a short pyrotechnic delay device.

Effect of Silicon content on Si/PbO/ FG delay mixtures

The effect of silicon content on the mass consumption and burning time of Si/PbO/FG delay mixtures was experimentally investigated. The ingredients of different delay mixtures are shown in Table-2. Test results of mass consumption and burning time were record and are shown in Table-2 and Fig 3. The Silicon content was varied from 5.0 wt. % to 40 wt. % in these delay mixtures. A total of eight delay mixtures with different Silicon contents were prepared. Five tests were conducted for every mixture in stainless steel tube of 4 mm internal diameter and the measured results were averaged. The loading pressure for pressing the delay composition in the delay body kept 40000 psi. The mean standard deviations of charge weight, burning time and mass consumption were also calculated.

Table-2: Test results of Si/PbO/ FG delay mixtures at different Silicon contents.

Pyrotechnic mixture	MCW	MBT	MCC	MSDCW	MSDBT	MSDCC
5.0% Si, 1.0 %FG, 94.0 % PbO	365	NR	NA	NA	NA	NA
10.0% Si, 1.0 %FG, 89.0 % PbO	370	1209	0.306	5.12	10.9	0.006
15.0% Si, 1.0 %FG, 84.0 % PbO	375	819	0.458	3.49	5.45	0.003
20.0% Si, 1.0 %FG, 79.0 % PbO	363	913	0.398	3.50	20.2	0.096
25.0% Si, 1.0 %FG, 74.0 % PbO	363	751	0.484	2.80	19.2	0.104
30.0% Si, 1.0 %FG, 69.0 % PbO	348	574	0.609	3.50	25.4	0.923
35.0% Si, 1.0 %FG, 64.0 % PbO	355	NR	NA	NA	NA	NA
40.0% Si, 1.0 %FG, 59.0 % PbO	360	NR	NA	NA	NA	NA

Note. MCW = Mean charge weight (mg); MBT = Mean burning time (ms); MCC = Mean charge consumed (mg/s); MSDCW = Mean standard deviation in charge weight; MSDBT = Mean standard deviation in delay burning time; MSDCC = Mean standard deviation in charge consumed. NR = Not recorded. NA = Not applicable

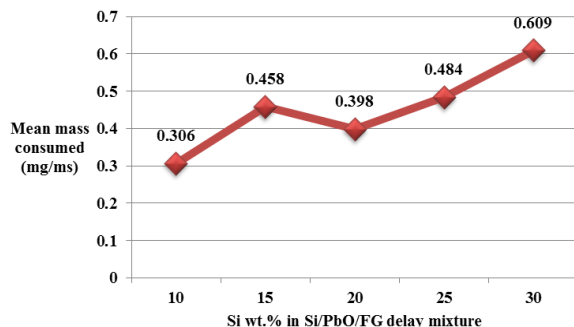


Fig. 3 Effect of Silicon content on mass consumption of Si/PbO/FG delay mixture.

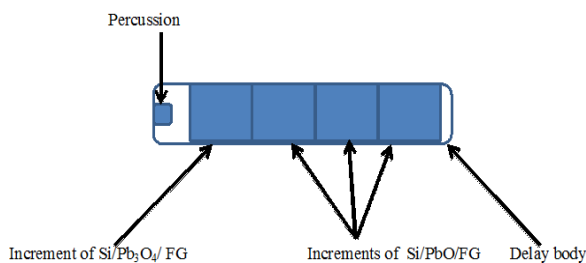


Fig. 4 Design of modified Delay device.

Results in Table-2 show that at 5.0 wt % Silicon, the composition did not initiate/ ignited by the standard percussion primer, so Si/PbO/FG pyrotechnic delay composition at 5.0 % Silicon contents failed to be ignited by the standard percussion when hit with a spring loaded striking pin. At this fuel oxidizer ratio the energy produced by the percussion was not enough to initiate the delay composition. In order to increase the sensitivity of this delay mixture at 5.0 wt. %, the loading pressure was then reduced from 40000 to 20000 psi but still the composition did not fire by the percussion primer and similar result was observed. When the Silicon contents were increased to 10%, the delay mixture started reliable burning and complete propagation was observed, the mean mass consumption of 0.306 mg/ms and burning time of 1209 ms was recorded. On further increasing the Silicon content to 15%, the mass consumption increased to 0.458 mg/ms, while the burning time decreased to 819 ms, which

shows an increase of 50 % in mass consumption and decrease of 30 % in burning time of this delay mixture. As the mass consumption increase the burning time decreases accordingly, because the mass consumption and burning time are inversely proportional to each other.

On further increasing the silicon contents beyond 15%, the delay composition did fire but did not produce consistence results. At 20, 25 and 30 % Silicon, the mean mass consumption were, 0.398, 0.484 and 0.607 milligram per millisecond and mean burning time was 913, 751 and 574 ms respectively. The mean standard deviations in burning time of this delay mixtures were 20.2, 19.2 and 25.4 respectively, which is very high and not acceptable in any pyrotechnic delay mixtures. Moreover at 30 % Silicon, one sample out of 5 partially fired and the flam could not be detected and thus the delay time was not recorded. When the fuel content was increased to 35 % Silicon, the composition did not completely burn, partial burning was observed and the flam did not detect by the detector due to its low intensity, so the Si/PbO/FG pyrotechnic delay composition at 35% silicon failed to produce reliable combustion when pressed at 40000 psi. The pressing pressure was reduced to 20000 psi to increase the sensitivity of the delay mixture to percussion, but similar result was observed and the propagation failed. It means that the heat produced by the delay composition was not enough to sustain propagation at this ratio, in other words the heat loss was more than the heat produce and thus burning stopped. At 40 and above 40 % Silicon content the composition did not initiate/ ignited by the standard percussion.

Effect of Fast red lead mixture on mass consumption of Si/PbO/ FG delay mixtures

In order to improve the performance of Si/PbO/ FG delay mixtures and to produce reliable combustion propagation, an increment of 100 mg of fast red lead (Si/Pb₃O₄/FG=20/79/1.0) was incorporated between primer and Si/PbO/FG delay mixture .Test results in Table-3 and Fig. 5 show that when an increment of first fire was added, the mass consumption of Si/PbO/FG delay mixture considerably increased.

The mass consumption of Si/PbO/FG = 10/89/1.0 delay mixture increased by 39 % when an increment of 100 mg fast red lead was added. Similarly, the mass consumption of Si/PbO/ FG delay mixtures with 15 %, 20 % and 25 % Silicon contents increased by 34 %, 75% and 46 % respectively. Result also shows that the mass consumption was not much affected at 30 % Silicon contents of Si/PbO/FG mixture by incorporated an increment of fast red lead mixture.

Table-3: Test results of different Si/PbO/ FG delay mixtures by incorporating an increment of fast red lead (Si/Pb₃O₄/FG=20/79/1.0) mixture

Pyrotechnic mixture	MCW	MBT	MCC
10.0% Si, 1.0 %FG, 89.0 % PbO	385	906	0.426
15.0% Si, 1.0 %FG, 84.0 % PbO	376	614	0.612
20.0% Si, 1.0 %FG, 79.0 % PbO	358	515	0.695
25.0% Si, 1.0 %FG, 74.0 % PbO	377	534	0.706
30.0% Si, 1.0 %FG, 69.0 % PbO	384	625	0.614
35.0% Si, 1.0 %FG, 64.0 % PbO	385	625	0.616
40.0% Si, 1.0 %FG, 59.0 % PbO	380	NR	NA

Note. MCW = Mean charge weight (mg); MBT = Mean burning time (ms); MCC = Mean charge consumed (mg/s); NR = Not recorded. NA = Not applicable

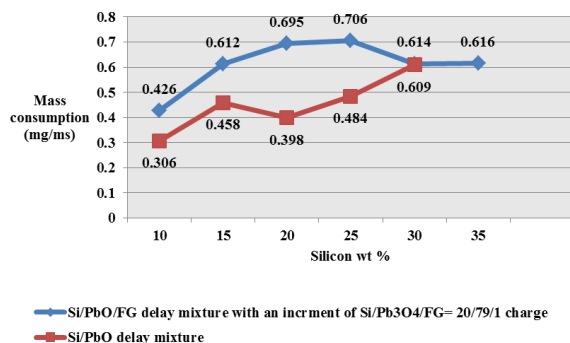


Fig 5 Effect of Fast red lead (Si/Pb₃O₄/FG=20/79/1.0) mixture on mass consumption of Si/PbO/ FG delay mixtures.

Conclusions

It is concluded from these results that Si/Pb₃O₄FG delay composition produced consistence and reproducible results between 10 % to 35 % silicon contents. Beyond these ranges the composition failed to produce reliable burning performance. This delay mixture can be reliably used in short and medium time delay producing cartridges and detonators. Si/PbO/FG delay composition can be reliably used with Silicon contents between 10 % to 15 % without using first fire pyrotechnic mixture. The mass consumption and thus performance of the Si/PbO/FG delay composition improves by incorporating an increment of fast red lead (Si/Pb₃O₄/FG=20/79/1.0) as first fire charge between primer and Si/PbO/FG delay composition in the delay tube. It is also concluded that Si/Pb₃O₄/FG is a fast burning pyrotechnic delay compositions, whereas Si/PbO/FG is a slow burning delay mixture.

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