CHARACTERIZATION OF PYROTECHNIC COMPOSITION USED IN TRACER AMMUNITIONS

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In this paper various pyrotechnic compositions used in VIS and IR tracer ammunition were studied to improve the required optical effect. These mixtures are designed to mark the direction of the bullet. The thermal behaviour of the compositions was determined by employing DTA tests. Additionally, spectral analysis and visual studies were used to assess the light output and radiant energy of the tracer combustion and establish the efficiency of the compositions. Since each well-known tracer blend that was tested needed optimisation, a new and improved configuration of VIS and IR composition was reported. Consequently, the optimal VIS and IR pyrotechnic mixture used in tracer ammunition was chosen based on their burn time, average luminous intensity and burning temperature.

Keywords: VIS/IR tracer ammunition, pyrotechnic composition, burn time, luminous intensity, burning temperature

1. Introduction

Pyrotechnic compositions made of inorganic or organic chemical fuels and oxidizers that generates mechanical, audible, thermal, or visual thermal effects (such as motion, sound, colour, light, and smoke) are widely used in both civil and military applications [1] [2], due to their lower cost components, compactness, reliability, and technological simplicity [3] [4].

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Tracer projectiles are frequently used in combat and warfare training to provide a visual path of a projectile during the night time. It is possible to observe the trajectory of the projectile once the pyrotechnic mixture in the tracer has ignited, since it emits a particular spectral radiation. There are numerous common tracer compositions that can produce detectable amounts of visible light, as well as considerable amounts of infrared light (IR). While luminescence can be produced by all materials, its intensity and spectral range may vary. Conventional tracers have the downside of emitting a significant amount of light in the visible spectrum (VIS), which could allow the opponent to identify the shooter's location.

The IR tracer bullets have a projectile path that is imperceptible in VIS domain but is observable using near infrared energy detection equipment. When ignited, the IR tracer emits a controlled amount of near-infrared (NIR) light, allowing the flight path of the projectile/bullet to be tracked without causing "bloom" (blinding the receiving sensor) to the observation equipment (night vision goggle with image intensifier). It is desired to create a tracer composition that will not cause the malfunctioning of the IR optoelectronic equipment when is fired.

Usually, a chamber in the back of the cartridge is used to place the tracer charge. Another technique it involves a pre-installed charge capsule, which is subsequently compressed or shrunk into the cavity (fig.1.). The tracer device also includes an igniter, a delay, and a primer pyrotechnic composition. A current problem that may arise is related to the attachment between the tracer charge capsule and the projectile. The forces that are acting on the projectile during the discharge phase can increase the risk of the tracer charge to separate from the projectile, which can be a complete or only a partial separation.

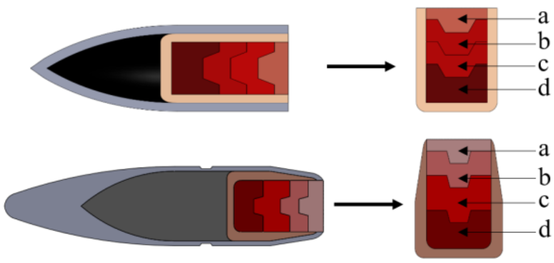

Fig. 1. Geometric example of pyrotechnic tracer
a - igniter composition; b – delay composition; c – primer composition; d – tracer composition

A pyrotechnic tracer composition (PTC) is composed of strontium nitrate ($Sr(NO_3)_2$) as an oxidizer and magnesium (Mg and Mg-Al alloy) as a dual-fuel, that provides high light output [6] [7] with red emission in the visible light spectrum. VIS trajectory of a red tracer projectile can be perceived in the night better than the green, blue, and yellow colour [8]. The electromagnetic signature of the tracer can be easily adjusted [9]; the light output can be tuned to emit in either the visible or

NIR spectrum. The fuel-oxidizer pairing and/or the stoichiometric relationship between them can be modified to achieve the optimal adjusting.

The manufacture of red-tracers in the visible spectrum and IR tracers in the infrared spectrum are presented in this work. These steps involve the synthesis, characterization, and examination of the material formulations. The important parameters that are pursued to be adjusted are the following: burning time, average luminous intensity, burning temperature, and spectrum emission. Several types of compositions are presented in order to optimize the effect and also to demonstrate the method of controlling the light intensity emission by changing the values of key parameters. In addition to Arabic gum (AG), poly vinyl chloride (PVC), and dextrin, a new component, polyurethane (PU), is used as a binder to provide a better charge density without a pressing process and without the fragmentation of the surface of the tracer.

2. Materials and methods

2.1 Materials

Pyrotechnic initiator – RR4 (UM Băbeni SA); *oxidizers*: barium nitrate ($Ba(NO_3)_2$, Sigma Aldrich), strontium nitrate ($Sr(NO_3)_2$, Sigma Aldrich), barium peroxide (BaO_2, Sigma Aldrich), potassium perchlorate ($KClO_4$, Sigma Aldrich), potas-sium nitrate (KNO_3, 99%, Honeywell Fluka™, Seelze, Germany), barium-chromate ($BaCrO_4$, Honeywell Fluka™, Seelze, Germany); *metallic fuels*: magnesium (Mg, powder, Sigma Aldrich), aluminium-magnesium alloy powder, with an average particle size <5 μm (PAM, Sigma Aldrich, St. Louis, MO, USA), antimony sulfide (Sb_2S_3, Honeywell Fluka™, Seelze, Germany); *binder*s: Sethatane D1160 (SET, hydroxyl content – 5.4%, Allnex, Brussels, Belgium), diphenylmethane–4,4'–diisocyanate (MDI, -NCO content – 31.5% (weight %)), phenol-formaldehyde resin (Iditol $C_{13}H_{12}O_2$, UM Sadu Gorj); *additional elements*: polyvinyl chloride (PVC, powder, Oltchim S.A.), sodium bicarbonate ($NaHCO_3$, supermaket), magnesium carbonate ($MgCO_3$, Sigma Aldrich), strontium carbonate ($SrCO_3$, Sigma Aldrich), was used as received.

2.2 VIS and IR compositions

The smoke generating composites were prepared using different formulations named PC1 to PC7 for VIS tracer composition that are specified in Table 1, and PC1-IR to PC4-IR for IR tracer composition that are stipulated in Table 2. In this work the technological process of the final pyrotechnic mixture was made at laboratory-scale, so a quantity of 50 g per sample is given for each composition. The first step consisted in dry mixing of the solid components (oxidizer, metallic fuel, catalyst) until they were completely homogenized, followed by wet mixing the organic components (blends of polyurethane - PU: MDI+SET; Iditol: 50% phenol-formaldehyde resin + 50% ethanol solution). The obtained wet-mixtures were dried in a laboratory oven at 60 ºC for 8 h. The samples were granulometric sorted and casted in metal tracer tubes in 2 layers as a final phase of the process (igniter composition RR4 – superior layer and tracer composition – based layer).

Table 1

VIS tracer composite formulation

Sample	Compound proportions (wt. %)								
	$Ba(NO_3)_2$	$Sr(NO_3)_2$	Mg	PAM	$SrCO_3$	$MgCO_3$	PU	PVC	$C_{13}H_{12}O_2$
PC1	-	58	18	-	-	5	-	20	4
PC2	-	72	21	-	-	-	-	-	7
PC3	-	62	22	-	2	-	-	8	6
PC4	-	62	22	-	2	-	4	10	-
PC5	-	49	15	15	-	-	-	13	8
PC6	-	58	24	-	-	-	4	16	-
PC7	65	-	18	-	-	-	-	17	-

Table 2

IR tracer composite formulation

Sample	Compound proportions (wt. %)								
	$Sr(NO_3)_2$	$BaCrO_4$	KNO_3	$KClO_4$	BaO_2	Sb_2S_3	$MgCO_3$	PU	$C_{13}H_{12}O_2$
PC1-IR	-	79		10	-	11	-	2	-
PC2-IR	-	-	70	-	-	-	15	-	20
PC3-IR	45	-	-	-	35	-	10	10	-
PC4-IR	40	-	-	-	45	-	5	-	10

2.3 Methods

Several analyses were carried out to assess the safety and performance characteristics of the synthesized VIS/IR pyrotechnic tracer compositions by static and dynamic ignition tests which represents the first stage in the development/optimization of these mixtures.

The thermal properties of the VIS/IR tracer compositions developed in this study were investigated using differential thermal analysis (DTA) [10]. The thermal behaviour of the tracer compositions was evaluated using a DTA OZM 551 Ex (OZM Research, Hrochv Tnec, Czech Republic) and Meavy dedicated software. The investigation was performed on 25-30 mg samples that were heated with a rate of 5°C /min from 25°C to 500°C.

The safety and stability characteristics were assessed by employing the following investigations: friction sensitivity test, impact resistance test and vacuum test. Friction sensitivity of the samples was investigated by using BAM friction equipment according to STANAG 4487 [11]. As a result, each sample underwent 25–30 tests at different weights to analyse the friction force according to Bruceton method with a 50% probability. Impact sensibility was determined on KAST Hammer device, where the initiation energy was established for each sample, according to STANAG 4489 [12]. Vacuum stability tests (VST) were performed in accordance with STANAG 4556 [13], in which 5 g of each sample was heated to 100°C and kept at this temperature for 40 hours, under vacuum. The recorded pressure variation was used for calculating the released gas volume, according to relation (1).

$$V_{deg} = \left[V_c + V_t - \frac{m}{d}\right] \cdot \left[\frac{P_2 \cdot 273}{t_2 + 273} - \frac{P_1 \cdot 273}{t_1 + 273}\right] \cdot \frac{1}{1.013} \quad (1)$$

where V_{deg} - volume of gases released by the sample (cm^3); V_c - volume occupied by the transducer and the adapter (cm^3); V_t - volume of the heating tube (cm^3); m - sample amount, (g); d - density of the tested sample, (g/cm^3); P_1 - initial pressure, (bar); P_2 - final pressure, (bar); t_1 - initial ambient temperature, (°C); t_2 - final ambient temperature, (°C).

For thermodynamic characterization an AVL 1805 - adiabatic calorimeter was used to estimate the heat of combustion and the specific volume for 2±0.1g of sample. The equipment includes a 25 cm^3 calorimetric bomb and a Julius-Peters gas-meter with a volume of 3180 cm^3. The tracer composition was initiated in the closed vacuum bomb using a radiant wire with 1.5 cal/cm. The heat of combustion and specific volume were calculated using relation (2) and (3), based on the temperature variations.

$$Q_v = \frac{K \cdot \Delta t - q}{\omega} \quad (2)$$

$$V_{sp} = \frac{W \cdot \Delta P \cdot 273.15}{\omega \cdot 760 \cdot (273.15 + t)} \quad (3)$$

where K is caloric equivalent of the apparatus, (K=1364.393 cal/°C); Δt - temperature variation measured, (°C); q – combustion heat produced by the ignition wire, (cal/cm); W - vessel volume + calorimeter bomb volume, (l); ΔP – pressure variation (mercury column height) (mmHg); ω - sample amount, (g); t - ambient temperature, (°C).

Both static and dynamic ignition tests were performed to determine the combustion parameters. For static ignition test (fixed tracer tub) a thermal high-speed camera FLIR X6580sc with InSb detector, was used to investigate the combustion mechanism in atmospheric conditions during the day. The thermal measurements (*combustion temperature – T_c and combustion time – t_c*) were captured at 300 frames per second and interpreted with the FLIR ResearchIR Max® software as a temperature-time plot. In accordance with the results that were obtained it was possible to calculate the *combustion rate* (4) and *combustion flow* (5) by measuring the *total combustion time*.

$$v_c = \frac{\Delta H}{\Delta t_c} \quad (4)$$

$$\dot{m} = \frac{\Delta m_{pc}}{\Delta t_c} = \rho \cdot S \cdot v_c \quad (5)$$

where v_c is the combustion rate (m/s), $\dot{m}$ is combustion flow (g/s), H – cylindrical tablets height (mm), m_{pc} –charge mass (g) and t_c – combustion time (the moment between the combustion charge starts to when it ends).

In the same setup, a visual optical camera KIKON D5600 was used to evaluate the VIS light effect. At 1 m from the tracer source, the illuminance (luminous flux per unit area) was measured using a lux-meter device (PeakTech 5035 Environment Meter), and it is used to determine the luminous intensity with the relation (6).

$$I = \frac{E \cdot L^2}{\cos \gamma} \tag{6}$$

where I - luminous intensity (cd); E is illuminance (lux); L is distance between source and sensor (m); γ is the angle between the source direction and the sensor position ($\cos\gamma = 1$ for $\gamma = 0^o$).

The radiation spectrum of the samples was measured using an Ocean Optics HR4000 High-Resolution Fiber Optic Spectrometer during the combustion process that had analysed the emission spectrum. The radiation emitted by the compositions was measured using an optical fibre which operates between 200 nm and 1200 nm.

Dynamic tests were completed for determining the qualitative and comparative visual characteristics in the IR and VIS spectrums, using an optical visual camera NIKON D5300 and a NIGHT VISION military camera OVN-1 with image intensifier. The tracer is connected to a system in which, after is ignited, the setup moves up to 10 m at a constant speed of 2 m/s, in to a dark room and at atmospheric conditions.

For the combustion experiments (static and dynamic ignition test), tracer tubes measuring 7.42 mm in diameter and 16.5 mm in length with an interior volume of 0.74 cm^3 were used. A hydraulic press with a compressive force of 15 bars was used to press the sample into the tracer tubes. The density of the sample was measured based on the equation of $\rho = m/v$ (ρ-charge density (g/cm^3); m – sample mass (g); v – tracer tube volume (cm^3)). Three samples of each composition were used for the combustion test, and the results were averaged.

3. Results and discussions

DTA analysis showed that the tracer composition has a typical sensitivity behaviour [14] [15] to thermal stimulus between 300 ºC and 500 ºC. The thermograms evaluation results illustrated in Fig.2 and Fig.3 shows that for PC2, PC4, PC7, PC2-IR, and PC4-IR no decomposition process was recorded in the temperature range 25 ºC to 550ºC. This can indicate a superior thermal stability, in addition to a difficulty for the composition to ignite. According to the exothermic area from the Fig.2 and Fig.3, the VIS tracer generates higher combustion temperatures compared to the IR tracer. DTA shows that PC1, PC2, PC5 and PC6 are stable up to 500ºC, while PC1-IR and PC3-IR up to 363 ºC and respectively 249 ºC. PC6 provides a good thermal interpretation for pyrotechnic composition based on graphic parameters represented in Fig. 2 that displays a decomposition temperature at 498 ºC. The exothermic process is shorter for PC6 than PC1, PC3, and PC5. Furthermore, Fig.2 shows that the thermal process of VIS samples has two exothermic peaks, first between 220ºC-285ºC, and second between 470ºC-520ºC, which represent the ignition phases of the compositions.

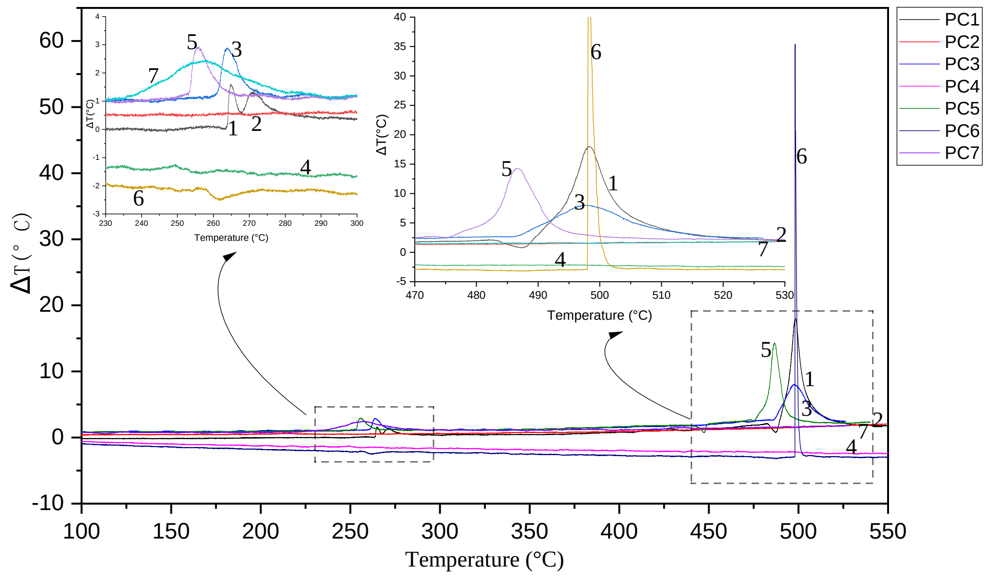

Fig. 2. DTA thermograms of VIS tracer composition

The endothermic reactions represented in Fig. 3 are indicating the following transitions: PC1-IR melting point of $BaCrO_4$ at 300 ºC; PC2-IR melting point of $MgCO_3$ can be seen at 125 ºC, but the melting point of $MgCO_3$ is hardly noticeable for PC3-IR and PC4-IR, where the weight proportions of $MgCO_3$ is less than or equal to 10%; for PC2-IR the melting point of KNO_3 is visible at 334 ºC.

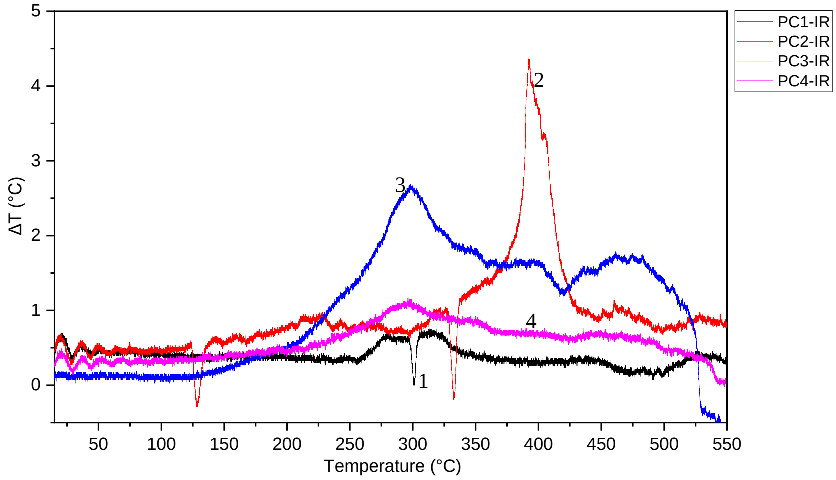

Fig. 3. DTA thermograms of IR tracer composition

The pyrotechnic materials are typically subjected to three types of investigations to determine the safety and security measures needed in handling the composition, such as: impact sensitivity test, friction sensitivity test and vacuum stability test (results are displayed in Table 3 and Table 4). Friction tests revealed that the materials have a standard friction sensitivity that is greater than 20 daN. The impact sensitivity investigations evaluated the minimum energy required to initiate the combustion of the tracer samples. The vacuum test showed that the material is unstable and produces a large quantity of gaseous products, as the pressure significantly increases during this test. In this case all samples generated a negligible quantity of gaseous products. An acceptable stability is defined by STANAG 4556 [13] as a composition that produces a gas volume that is below 1 cm^3/g.

Table 3

Safety and stability characteristics for VIS tracer

Investigation type	Sample	PC1	PC2	PC3	PC4	PC5	PC6	PC7
Friction sensitivity	Friction force (daN)	22	22	26	28	24	28	20
Impact sensitivity	Initiation energy (J)	5	5	5	8	3	8	5
Vacuum stability	Vacuum gas volume (cm^3/g)	0.21	0.12	0.22	0.14	0.31	0.16	0.28

Table 4

Safety and stability characteristics for IR tracer

Investigation type	Sample	PC1-IR	PC2-IR	PC3-IR	PC4-IR
Friction sensitivity	Friction force (N)	28	22	24	24
Impact sensitivity	Initiation energy (J)	8	8	6	5
Vacuum stability	Vacuum gas volume (cm^3/g)	0.19	0.22	0.15	0.17

The analysis of thermodynamic data provided in tables 5 and 6 reveals that the IR tracer samples showed a slightly reduced combustion heat and specific volume compared to the VIS tracer, which is a mandatory recommendation for this kind of compositions.

Table 5

Thermodynamic characteristics for VIS tracer

Sample	**PC1**	**PC2**	**PC3**	**PC4**	**PC5**	**PC6**	**PC7**
Combustion heat (kcal/kg)	1248.89	1386.34	1319.17	1412.69	1756.13	1333.5	1313.23
Volum specific (l/kg)	225.95	225.30	173.73	229.78	210.84	251.61	189.27

Table 6

Thermodynamic characteristics for IR tracer

Sample	**PC1-IR**	**PC2-IR**	**PC3-IR**	**PC4-IR**
Combustion heat (kcal/kg)	616.87	760.21	595.01	621.60
Volum specific (l/kg)	57.09	176.23	193.31	201.32

A key characteristic of red VIS tracer composition is that it produces greater combustion temperatures in contrast to the IR tracer, in relation to thermodynamic data, demonstrating the ability to emit good visual radiation between 620 – 750 nm.

The combustion parameters obtained during the static experiments are presented in Table 7 and Table 8. When there was no metallic fuel present, the IR tracer recorded temperatures of 900 ºC with light intensities of less than 200 cd, while the VIS tracer recorded combustion temperatures higher than 1200 ºC, resulting in elevated luminous intensity values. According to relation (6), luminous intensity (I) has the value of illuminance (E) from the Table 7 for a distance of 1 m and an angle of 0^{o}.

Table 7

Combustion characteristics of VIS tracer

Sample	m [g]	m_r [g]	ρ [g/cm^3]	T_c [°C]	t_c [s]	V_c [mm/s]	$\dot{m}$ (g/s)	E (lux)
PC 1	1	0.2	1.35	1483	4.26	3.873	0.23	769
PC 2	1.2	0.6	1.62	1231	6.73	2.451	0.17	537
PC 3	1.2	0.2	1.62	1268	4.72	3.490	0.25	592
PC 4	1	0.4	1.35	1227	6.24	2.640	0.16	482
PC 5	1	0.2	1.35	1449	4.27	3.864	0.23	876
PC 6	1	0.5	1.35	1386	7.09	2.324	0.14	498
PC 7	1.2	0.4	1.62	1392	1.88	8.753	0.63	578

Table 8

Combustion characteristics of IR tracer

Sample	m [g]	m_r [g]	ρ [g/cm^3]	T_c [°C]	t_c [s]	v_c [mm/s]	$\dot{m}$ (g/s)	E (lux)
PC 1-IR	1.4	1	1.89	871	10.63	1.552	0.13	202
PC 2-IR	1.2	0.6	1.62	783	11.87	1.386	0.10	189
PC 3-IR	1.2	0.8	1.62	725	7.98	2.065	0.15	169
PC 4-IR	1.2	0.8	1.62	763	8.39	1.965	0.14	181

The pyrotechnic composition for VIS tracer aims to provide a high residual mass (m_r) in the tracer tub in order to ensure the aerodynamic stability of the bullet; can produce a longer combustion time with an optimal combustion temperature and with less gases thus avoiding the destruction of the tub; and to offer an optimal intensity radiation by obtaining a good visual flame. In Fig.4, the flame presented in the burning zone for each VIS tracer has a higher light intensity in red colour compared to the green colour. The significant visual difference between red and green colour can be noticed in Fig.4 at PC6 vs PC7. This explains why red tracer cartridges are used more frequently than green ones.

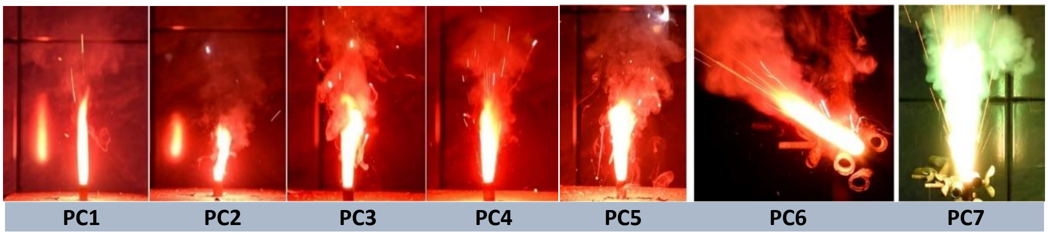

Fig. 4. VIS tracer sample combustion effect for PC1 to PC7

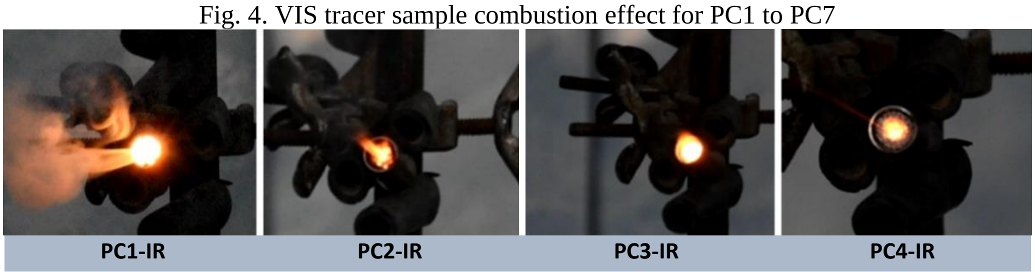

Fig. 5. IR tracer sample combustion effect for PC1-IR to PC4-IR

Another purpose of the IR tracer is to achieve low combustion temperatures, resulting in a hot spot with low luminous intensity in the front of the combustion zone, as shown in Fig.5. The residual mass and the combustion time is similar as in the case of the VIS tracer. In Fig.6 displays the time-temperature plot of the combustion process of PC3, PC6 and PC3-IR samples, obtained with thermal camera. According to the graph, the IR tracer burning process had a constant temperature that provides a hot spot with good NIR emission stability, as shown in the emission analysis in Fig. 8. The shape of the combustion flame for VIS tracer represented in Fig. 4 is closely related to the temperature-time graphic, Fig.7, that illustrate temperature variation during the process, which influences the emission.

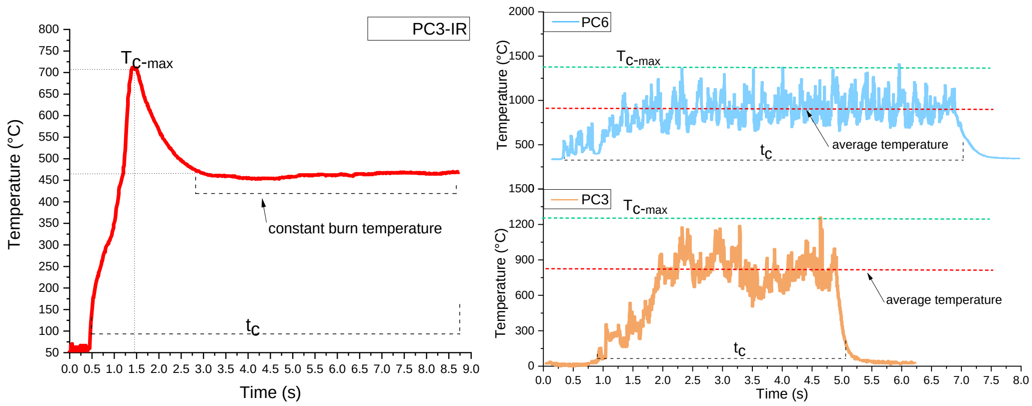

Fig. 6. Example of data acquisition of temperature-time

Based on Fig. 7, the spectrum data, PC1, PC2, PC4, PC5 and PC6 samples emitted wavelengths between 550 – 700 nm in VIS range with high intensity of red colour, and low radiation in NIR spectrum (780 nm). However, PC3 has a multispectral emission in the range 450 – 900 nm with high intensity. PC7, the only one with green colour emission (500 – 550 nm), has a good signal in the IR zone. Similar to PC7, PC3, has a high value of illuminance (high visible radiation in burning zone, Fig.4). This radiation will blind the night vision sensor, which is why these samples failed meet the IR tracer condition and to fully satisfy the VIS tracer condition.

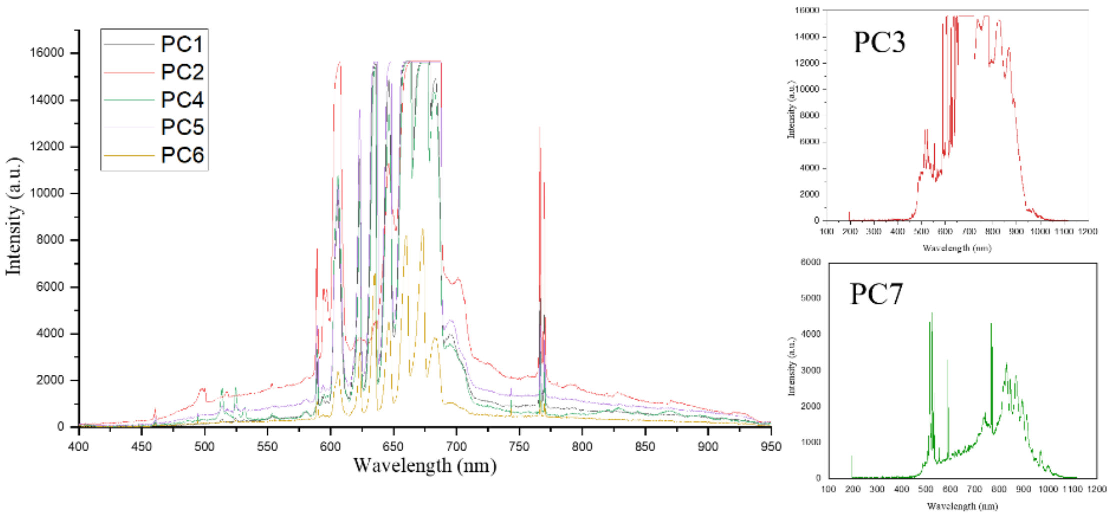

Fig. 7. Spectrum Emissivity of VIS tracer composition

For the IR samples, with good signal in NIR zone, the emission is evaluated in Fig.8 where it can be observed the difference between two types of tracers, VIS and IR tracer.

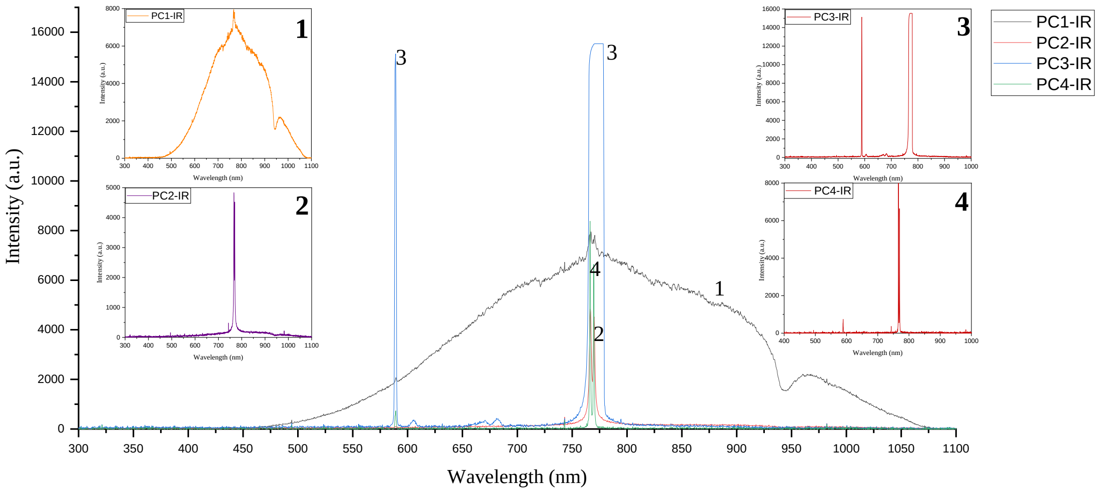

Fig. 8. Spectrum Emissivity of IR tracer composition

The PC1-IR oxidizer ($BaCrO_4$) emitted radiations from 550 nm to 900 nm, with low intensity but did not fulfil the IR condition. After the composition adjustment, starting with a low heat combustion, continuing with a low combustion temperature, and burning rate under 3 m/s, a hot spot with low illuminance and emission in the infrared zone has been obtained.

Based on the data, all samples can be classified as VIS and IR tracer compositions, but only PC6 and PC3-IR exhibits optimal properties. The same two were classified for dynamic testing. They were analysed by VIS and NIR camera to determine the visual effect and the results are represented in Fig.9 and Fig.10.

Fig. 9. PC-6 VIS tracer in the dark room recorded with VIS camera

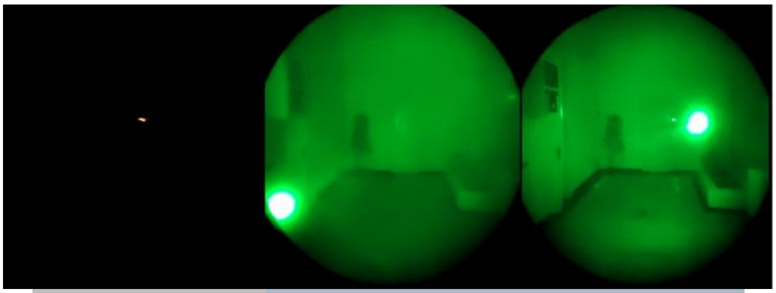

Fig. 10. CP3-IR tracer in the dark room recorder with VIS and NIR camera

4. Conclusions

In this paper were tested six VIS tracer compositions that had a different proportion of strontium nitrate as an oxidizer and magnesium/magnesium-aluminium as fuel, to achieve the best pyrotechnic effect, and one with barium nitrate to compare the spectrum radiation and visual effect of the flame. Only four compositions were tested for the IR tracer, two of which contained strontium nitrate with a cooling agent and the other with potassium nitrate and barium-chromate, in order to control the phenomenon with different oxidants for better results. The proposed mixtures, in both cases, aim to obtain a large mass of residues left in the tube after combustion. Various binders and additives were also chosen to enhance the effect, homogenize the mixture, and achieve high mechanical strength. Based on these principles, all presented tests were necessary to evaluate the two types of compositions with diametrically opposed performances. Based on the final data only PC6 and PC3-IR exhibit optimal properties. In order to adapt the electromagnetic signature of the VIS and IR tracers, the fuel-oxidizer combination had to be adjusted. Also, considering a good VIS tracer with clear red light in the combustion zone, a high value of heat combustion, a higher combustion temperature, a proper luminous intensity, and a radiation between 0.6-0.7 µm was needed. On the other hand, for the IR tracer the metal fuel it is not necessary; instead, the combustion needs to have lower temperature and to control the reaction that is needed to obtain a simple hot spot on the combustion surface with a radiation over 0.75 µm, and low combustion temperature. Because the night vision system functions with an image intensifier in the NIR spectrum, a high illuminance will blind the sensor, which is why a low heat in the combustion zone is essential.

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