Research paper

Novel method for measuring primary and secondary smoke density of reduced HCl exhaust composite solid rocket propellant (CSRP) based on nitramine energetic material

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Abstract

Tactical and technical demands of modern types of composite solid rocket propellant (CSRP) focused recently on decreasing both the primary smoke concentration, by minimizing the amount of solid particle as metal oxides and carbon soot, and secondary smoke concentration, by minimizing the amount of gaseous hydrogen chloride in the exhaust plume. In this work, reports the theoretical and practical methods for determining the primary smoke of composite solid propellant based on energetic material as (AP-HMX-RDX-$NaNO_3$...) and polyurethane (PU) binder based on Hydroxyl terminated polybutadiene (HTPB) which used in many kinds of composite propellant such as Conventional, Scavenger, and non-chlorine and neutralized composite propellant. Formulations were prepared and the densities of primary smoke were experimentally determined. A smoke device was invented for practical measuring of the primary smoke concentration by determining the smoke density and compared with the theoretical results from ICT thermodynamic program which used to calculate the ballistic parameters and smoke concentration of composite propellants. Secondary smoke was experimentally determined then compared with the theoretical data from ICT thermodynamic program which used to calculate the ballistic parameters and smoke concentration of composite propellants.

Keywords: conventional, scavenger, non-chlorine and neutralized propellant, smoke density

1. Introduction

The most commonly used types of composite propellants consists mainly of polyurethane binder based on hydroxyl terminated polybutadiene (HTPB), ammonium perchlorate (AP) as an oxidizer, aluminum powder (Al) as metallic fuel and some processing and ballistic additives[1)–3)]. The main disadvantage of such types of composite propellant is the evolution of huge amount of both primary and secondary smoke during the process of propellant combustion which may reveal the launch location of the missile, allow the tracking of missile trajectory during flight, cause the loss of control of optically guided missiles beside the emission of some dangerous gases as hydrochloric gas which have very bad environmental effect on launching crew[4), 5)]. Many techniques were applied to reduce the amount of generated smoke which include; reducing the amount of Al metal fuel, replacing Al metal by magnesium Mg fuel, reducing the amount AP oxidizer or replacing it by another non-chlorinated one as ammonium nitrate (AN) or one of the nitramine family as cyclo-tetramethylene tetranitramine (HMX). The amount of generated smoke may be determined by visual comparison between photo pictures taken to rocket trajectories, thermochemical calculations of the content of combustion gaseous products or by using some experimental techniques[6), 7)]. One of the modern types of composite propellants which markedly decrease the amount of generated smoke is called the scavenger propellant which uses sodium nitrate ($NaNO_3$) oxidizer instead of about one-half of the AP oxidizer. As the propellant burns in the combustion chamber the sodium ions scavenge most of the chlorine ions to form sodium chloride instead of forming HCl gas[8]. Another propellant type which also greatly reduces the smoke evolution is the neutralized propellant at which the Mg metal is used instead of Al as the metal fuel while all other ingredients are the same. As the propellant burns, Mg is oxidized to magnesium oxide MgO which upon contact with water it forms magnesium hydroxide $Mg(OH)_2$ which is a strong base reacts with HCl to form magnesium chloride[9]. Another method to avoid the presence of chlorine in the propellant formulation is to use nitramines such as HMX instead of ammonium perchlorate. The incorporation of 10–20% HMX by weight into an HTPB/AP propellant composition not only reduced the HCl smoke by 20–30% by weight but also improved the specific impulse[10, 11]. The chlorine-free propellant is another type of reduced smoke composite propellant at which the AP has been replaced by ammonium nitrate (AN) which is more difficult in processing than AP due to the hygroscopic nature of AN[12]. Energetic prepolymers and plasticizers have been considered for some time in connection with improving the performance of AN propellant, such as glycidyl azide polymer (GAP)[13]. Smoke classified into two types: a) primary smoke is easily detected because it exhibits the triple capacity at the same time absorbing, emitting and scattering ultraviolet-visible or infrared radiation. Most of the gaseous products such as CO, CO_2, H_2, H_2O, Al_2O_3 and HCl present in the primary smoke may be sometimes carbon soot exist due to burning the inhibitor of end-burning grain: and b) secondary smoke in which the combustion of propellant containing ammonium perchlorate produces HCl gas, under specific atmospheric condition of temperature and humidity. The combination with air results in the formation of white cloud. The increase of absorption and scattering of the visible and infrared light occur simultaneously, due to the growth both in number and size of drops. Methods used to measure the density of primary and secondary smoke such as a visual comparison between photo pictures taken to rocket trajectories, thermochemical calculations of gaseous products content and using devices for measuring smoke in the exhaust plume (primary smoke and secondary smoke).

In this paper, a smoke device (smoke filter device) was invented to measure the smoke concentration of propellant formulations based on a concept called in-line capture, a smoke sample is simply sucked through a filter which is weighed before and after the test and the mass of smoke found. Prepared propellant formulations were characterized by measuring of smoke density in the exhaust plume (primary smoke). Measured output value of primary smoke density is (mg/m^3) and then the results compared with theoretical results from ICT thermodynamic program to evaluate the accuracy of the primary smoke device. Also, the theoretical and practical methods using for determination of secondary smoke of composite solid propellant based on energetic material as (HMX-RDX...) and polyurethane (PU) binder based on HTPB which used in the propellant. The main reason for making secondary smoke is the production of HCl gas in the exhaust plume. The compositions of this work are chosen with high ballistic performance and low HCl concentration compared to the conventional aluminized propellant. Determination of secondary smoke by using theoretical calculations which estimate the gaseous products especially HCl gas of the prepared samples. Then followed by analytical detection of HCl gas concentration by using Gasmet dx4030 portable gas analyzer device then compared the results to verify between the predicted results and the practical one.

2. Thermo chemical calculations

Thermochemical calculations have been used in this work with considerable success as a fast-effective tool to investigate the performance parameters and to calculate the propellant formulations of reduced both primary and secondary smoke for many types of composite propellant formulations that are a candidate for the experimental investigation. The main objective of the presented work is to evaluate the accuracy and efficiency of the primary smoke device. ICT Thermodynamic code which is based on the chemical equilibrium and steady-state burning model was employed for the calculations of the smoke density of composite propellant formulations of different types of reduced smoke composite propellant families (scavenger, non-chlorine and neutralized) and also conventional one. The input data for the program are the percentage, density, enthalpy of formation and summary formula of each component in one kg of the propellant formulation. The operating pressure was selected to be 70 bar. The output results from ICT thermodynamic code are specific impulse, density, exhaust velocity, combustion temperature of one kg of the examined propellant formulation and the number of moles of solid and gaseous products of one kg of the required formulation which are used to calculate the concentrations of primary and secondary smokes in the exhaust plume in ppm. In this work, the parallel need of castable and high-performance propellant formulations with reduced smoke in exhaust plume in rockets. According to known the effect of RDX, HMX, Mg, $NaNO_3$ and NH_4NO_3 in many composite propellant formulations based on PU using either HTPB to keep propellant formulation castable and they are available and secure good ballistic performance parameters and reduced smoke in the exhaust plume.

3. Primary smoke particle size

In solid propellants, aluminum is widely used to improve the performance; however the condensed combustion products especially the large agglomerates generated from aluminum combustion significantly affect the combustion and internal flow inside the solid rocket motor. The chemical compositions of the collected products were studied by using scanning electron microscopy coupled with energy dispersive (SEM-EDS) method. Various structures have been observed in the condensed combustion products[14]. Apart from the typical

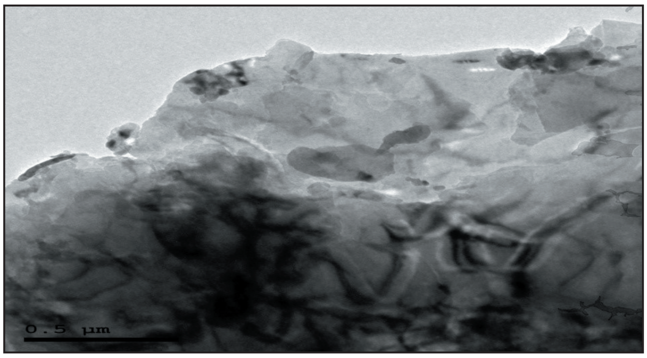

Figure 1 SEM image of smoke oxide cluster.

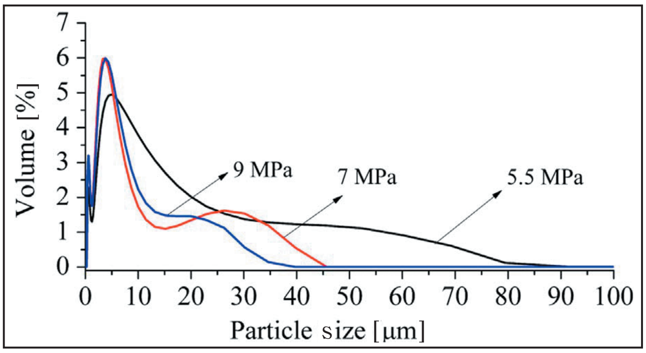

Figure 2 Size distribution of condensed combustion product at different pressure.

agglomerates or smoke oxide particles observed before, new structures including the smoke oxide clusters, irregular agglomerates and carbon-inclusions are discovered and investigated. Smoke oxide particles have the highest amount in the products[15]. The highly dispersed oxide particle is spherical with a very smooth surface and is on the order of 1–2 μm, but due to the high temperature and long residence time, these small particles will aggregate into smoke oxide clusters which are much larger than the initial particles shown in Figure 1[16]. Three types of spherical agglomerates have been found. As the ambient gas temperature is much higher than the boiling point of Al_2O_3, the condensation layer inside which the aluminum drop is burning would evaporate quickly, which result in the fact that few "hollow agglomerates" has been found compared to "cap agglomerates" and "solid agglomerates". Irregular agglomerates usually larger than spherical agglomerates, The formation of irregular agglomerates likely happens by three stages: deformation of spherical aluminum drops; combination of particles with various shapes; finally production of irregular agglomerates[17].

Pressure has a significant effect on aluminum agglomeration, that increasing pressure reduces the agglomeration this because high pressure can reduce the time of aluminum particles on the propellant surface and short residence time does not allow complete agglomeration (Figure 2).

As shown in Figure 2 the smoke particle size is 50, 30, 20 μm under pressure 5.5, 7, 9 MPa respectively. From the previous figures get that the average mean particle size of smoke particulates from 30 μm to 500 nm.

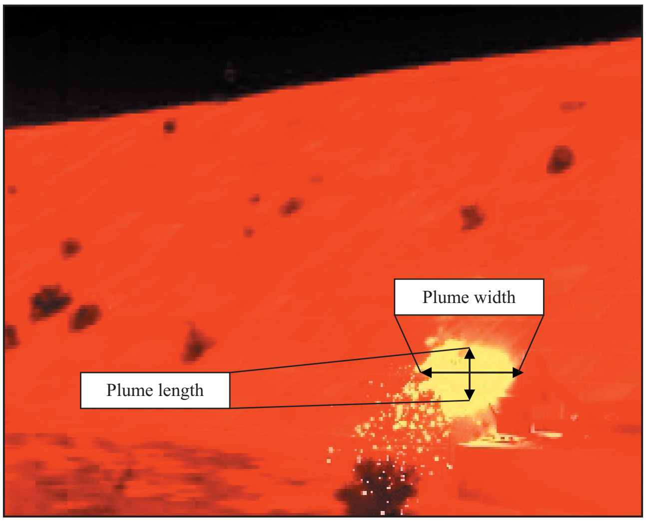

Figure 3 Photo of IR camera of the jet plume after nozzle of rocket motor the primary smoke device.

4. Flame length of rocket plume

One of the plume characteristics of minimum smoke propellant is the infrared (IR) radiation signature, which may be useful for detection of the rocket. The minimum smoke propellant afterburning flame length and mid-range IR intensity were measured while conducting static firing tests of the smoke device rocket motors loaded with minimum smoke propellants of the different contents of materials[18]. The exhaust plume can be divided into two section of the illuminating zone and smoke zone, the most important zone is the illuminating one and determined the length of this zone by using an infrared camera as shown in Figure 3. Determination of the illuminating length and the diameter also the temperature of this plume and the pressure outside the nozzle is important for the design of steal collection chamber dimensions will show later.

The length of the plume of conventional propellant is about 30 cm and the diameter about 7–8 cm as the plume come from small rocket motor and the temperature is about 1200 °C and ambient pressure as the design of nozzle is adapted nozzle. The design of motor described in Section 5.3.

5. Experimental

5.1 Preparations of composite rocket propellant formulations

All the chemicals used in this work; ammonium perchlorate (AP), Mg, Al, $NaNO_3$, ammonium nitrate (AN), hydroxyl terminated polybutadiene (HTPB), Isophoron diisocyanate (IPDI) or Hexamethylene diisocyanate (HMDI) as curing agent, Dioctylazelate (DOZ) as plasticizer, methyl aziridinyl phosphine oxide (MAPO) as bonding agent are of high purity. Casting technique was employed for the preparation of the 7 composite propellant formulations (one of them was the reference conventional formulation and 6 reduced smoke composite propellant formulations from scavenger, non-chlorine and neutralized families. The binder used in this work was HTPB prepolymer of 0.85 mg equivalent OH/g HTPB with DOZ as plasticizer and MAPO as a bonding agent. The

Table 1 Compositions of the prepared reduced smoke propellant formulations based on PU binder.

Formulation	AP	AN	$NaNO_3$	Al	Mg	HMX	Binder
C0 (Conventional)	69	–	–	17	–	–	14
C1 (Neutralized)	73	–	–	–	13	–	14
C2 (Non chlorine)	–	53	–	22	–	11	14
C3 (AN modified)	18	40	–	18	–	10	14
C4 (Neutralized modified)	60	–	–	–	17	9	14
C5 (Scavenger)	34.5	–	20	20	–	11.5	14
C6 (AN Propellant)	26	40	–	20	–	–	14

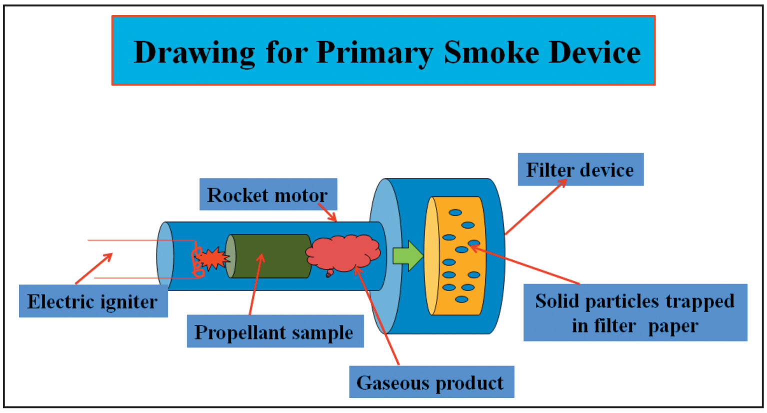

Figure 4 Drawing of primary smoke device.

prepolymer, bonding agent and the plasticizer were mixed together at 50–60 °C then solid ingredients were added in portions and thoroughly mixing was continued for 30 minutes. At the end of the mixing process, the curing agent HMDI of 11.9 mg equivalent NCO/g HMDI was added by a certain amount which keeps the NCO/OH ratio as 0.7 and mixing was carried out for a relatively short time. The special degassing unit was employed to get rid of any air bubbles before slurry casting in special molds for different measurements. Finally, the prepared formulations were cured at about 60 °C for two weeks[15]. The main constituents of the prepared reduced smoke composite propellants formulations are listed in the Table 1 in weight percentage.

5.2 Measurement of ballistic performance parameters

The performance parameters of the prepared propellant formulations as burning rate, specific impulse, and characteristic exhaust velocity were measured using standard two-inch testing rocket motor provided with a nozzle of fixed throat area diameter which means certain operating pressure and certain burning rate for each formulation.

5.3 Measurement of primary smoke concentration in the exhaust plume

The combustion of propellant samples of known weight was carried out in a special combustion chamber provided with a high-density polyethylene trap or steel trap filled with a special filter which is weighed before and after combustion to determine the mass of trapped solid particles (primary smoke) as shown in Figure 4. This method can calculate the density of smoke as follow:

Getting the value of the following:

- The volume of gaseous products (liter).
- The number of gaseous products moles.
- For all solid such as (Al_2O_3 carbon soot, NaCl....etc.) in gaseous products must calculate the weight = (number of mole*molecular weight) = output value in (mg).
- For $MgCl_2$ using "Equation (1)" $Mg(OH)_2 + 2\ HCl \rightarrow MgCl_2 + 2\ H_2O$.
- Smoke density = (weight of smoke solid content in gaseous products / volume of gaseous products) = output value in (mg L^{-1}) = (ppm).

Then calculate both theoretical and experimental one and compare the results for all compositions.

Specifications	Value
Density [kg m^{-3}]	96
Working temperature [°C]	1200
Thickness [mm]	25
Pore diameter [nm]	400

Figure 5 The ceramic fiber sheet and specification table.

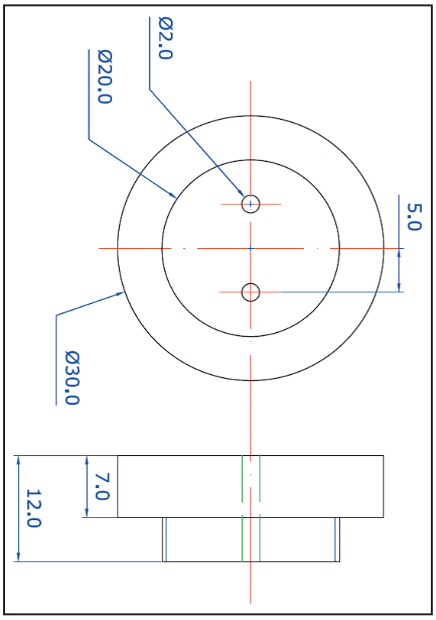

Figure 6 (a) Motor head.

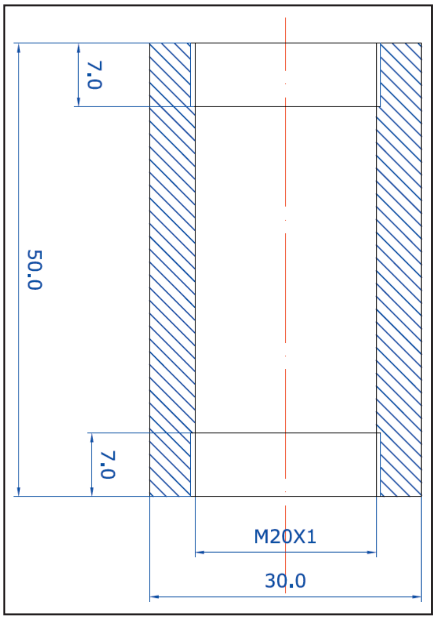

Figure 6 (b) Combustion chamber.

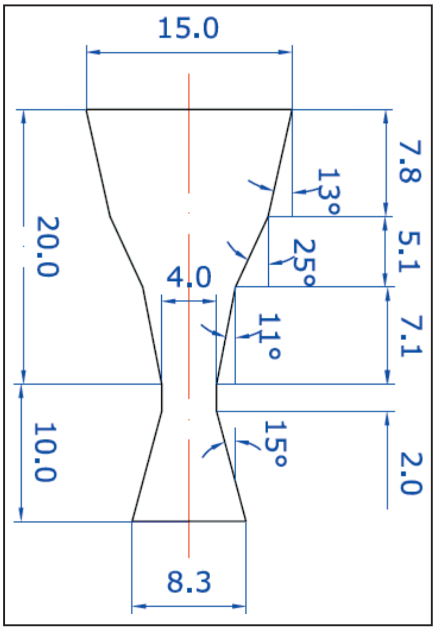

Figure 6 (c) Nozzle assembly.

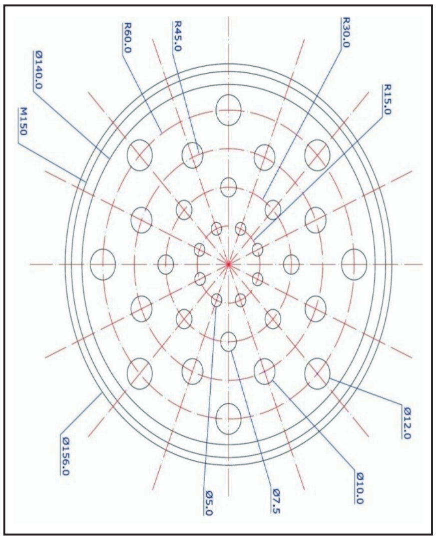

Figure 6 (d) Face of collection chamber.

5.4 Measurement of secondary smoke concentration in the exhaust plume

This method can calculate the density of smoke as follow:

- The volume of gaseous products (liter).
- The number of moles of HCl gas in gaseous products.
- In case of neutralized propellant do not forget consume HCl using in reaction with $Mg(OH)_2$ from calculation using also $Mg(OH)_2 + 2\,HCl \rightarrow MgCl_2 + 2\,H_2O$.

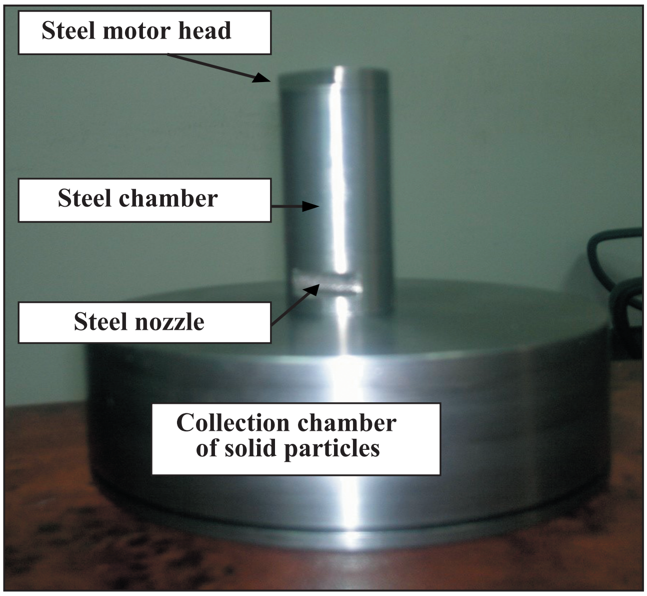

Figure 7 (a) Assembly of the whole device.

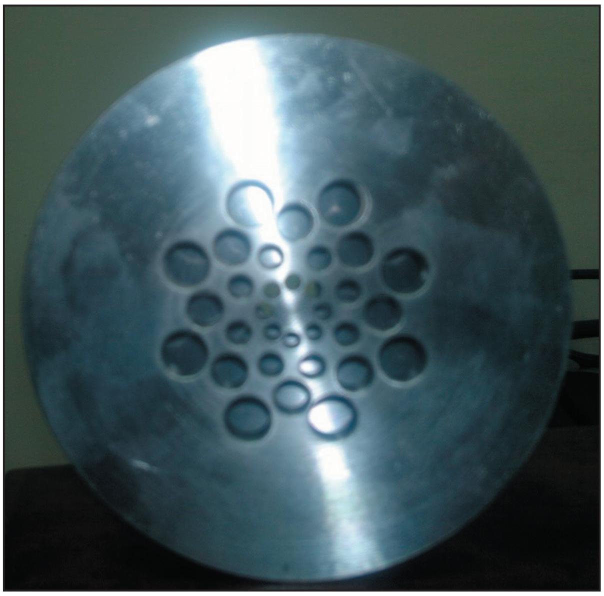

Figure 7 (b) Face of collection chamber.

- Smoke density = (weight of HCl gas in gaseous products / volume of gaseous products) = output value in (mg L^{-1}) = (ppm).

Then calculate both theoretical and experimental one and compare the results for all compositions.

5.5 Primary smoke device (smoke filter device)

Smoke device designed with the same expansion ratio of the real rocket motor nozzle and with same blocking factor and the same area of burning even if exist insulator or inhibitor must be made in the small rocket in order to get the full simulation and get highly accurate results from this device should used a good analytical balance and must make one fire with only the igniter charge to avoid the error in result also prefer the material of the gas chamber is highly resistance to the high temperature and the pressure. .

Components: a- ceramic fiber.

b- Small rocket motor: - Steel cylindrical chamber.
- Steel head.
- Steel nozzle.

c - Chamber contains from steel to collect the gaseous product.

Specification of ceramic fiber: descriptions of ceramic fiber blanket are made through a process of pricking, heat-shaping, cutting and rolling. They feature a high quality of tensile strength, texture uniformity and plane surface, high temperature, chemical corrosion resistance, thermal shock stability, and low thermal conductivity. It has low density, good resiliency and excellent sound absorbing and filtering property.

Density (kg m^{-3}) is 96, working temperature is 1250–1300 °C, the thickness is 25 mm and the pore diameter is 400 nm which can trap all the smoke particulates as the range of it about 30 μm to 500 nm, Figure 5 shows the ceramic fiber used in experiments.

The rest of the motor and collection chamber was shown in the following figures from the Figure 6 to Figure 9 which present each part of the smoke device and assembly of the whole device. Also the dimensions of steel rocket motor and collection chamber are shown in the following figures.

5.6 Device used to measure secondary smoke concentration in the exhaust plume

Gasmet dx4030 portable gas analyzer was used to determine the concentration of HCl gas in the gaseous products of the combustion exhaust plume. For all tested reduced smoke composite propellant samples, accurately weighed 0.5 g of each tested sample was combusted in a special testing chamber, Figure 10 provided with built-in suction pump to get rid of the combustion gases after each sample and the gas analyzer which is used to determine the percentage of each produced gas including HCl.

Gasmet dx4030 multi-component gas analyzer can detect up to 25 gases simultaneously providing validated results in less than 30 second. This is made possible with the means of FTIR (Fourier Transform Infrared Spectroscopy), which provides nearly instantaneous measurements with low detection limits and true multi compound analysis capability. Furthermore, an internal calibration is run simultaneously with the sample measurement. There is no need for any span calibrations. Also, with the Gasmet dx4030 no sample preparation is needed; the sample gas is drawn into the analyzer with a built-in pump through a particle filter as in Figure 11.

6. Results

6.1 Results of ballistic performance parameters

It is clear from the results in Table 2 that by comparing the values in the theoretical results with the practical one are close to each other as shown in combustion temperature and characteristic exhaust velocity and the measured results of specific impulse have about 5% deviation from calculated one, the measured results have the same behavior as theoretical one as predicted with a little difference which realized that the benefit of the theoretical thermochemical calculations which predict the

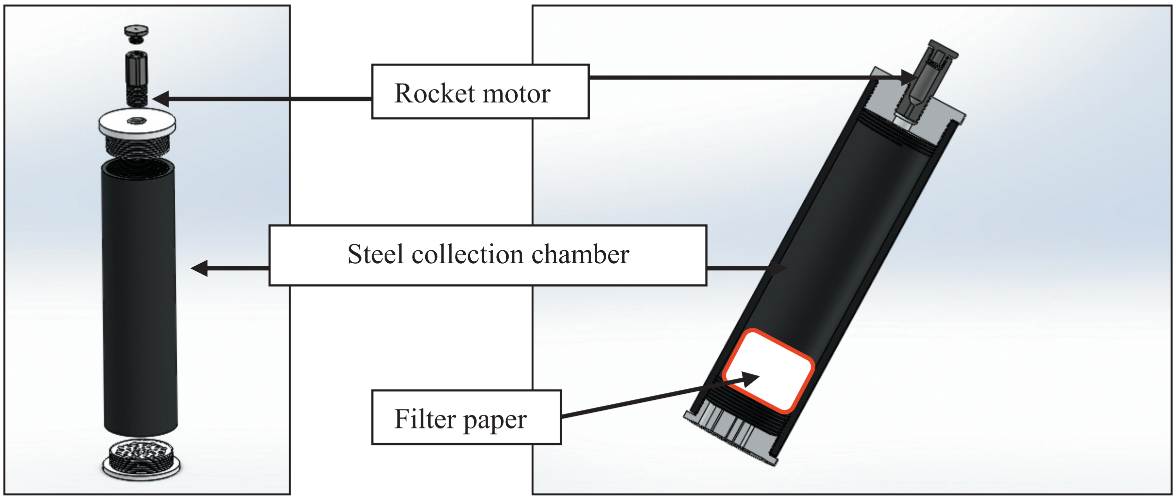

Figure 8 Assembly of smoke device.

Figure 9 Cross section of final assembly.

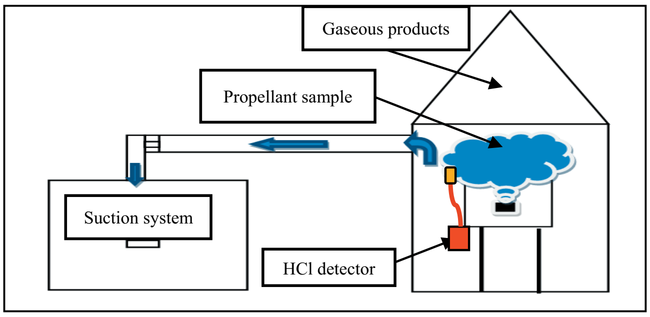

Figure 10 Secondary smoke testing chamber.

Figure 11 Gasmet dx4030 portable gas analyzer.

Table 2 Compositions of the prepared reduced smoke propellant formulations based on PU binder.

Ballistic parameters	Theoretical				Measured			
Formulation	ρ	T_c	C^*	I_{sp}	ρ	T_c	C^*	I_{sp}
C0 (Conventional)	1.76	3468	1529	250	1.76	3393	1520	235
C1 (Neutralized)	1.65	3105	1489	244	1.66	3190	1505	231
C2 (Non chlorine)	1.67	2897	1499	249	1.56	2805	1490	217
C3 (AN modified)	1.68	2948	1506	247	1.64	2511	1490	215
C4 (Neutralized modified)	1.65	3134	1480	246	1.65	3170	1511	233
C5 (Scavenger)	1.81	3318	1448	240	1.81	3360	1493	223
C6 (AN propellant)	1.69	3081	1505	249	1.60	3110	1512	220

best results for formulations C1 and C4 corresponding to the reference one C0 and the other formulations. For the measured specific impulse, the best-obtained results are represented by C1 and C4 (neutralized propellant family) realized good values compared with the conventional propellant C0 which can be replaced instead of C0 with little adjustment in density in order to give the advantage for mission performance. Also the same for the characteristic exhaust velocity, neutralized propellant family have good results when compared with other formulations and for formulations based on low values of specific impulse and characteristic exhaust velocity were

Figure12 Filter paper before test.

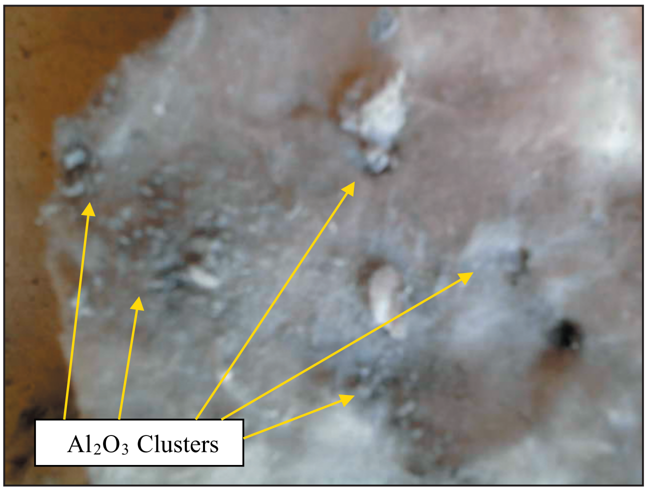

Figure13 Filter paper after test.

Table 3 Concentration of primary smoke of propellant formulations.

Formulation	Concentration of primary smoke [ppm]	
	Theoretical	Measured
C0	219.7	223.5
C1	195.9	198.6
C2	290.9	303.4
C3	237.6	258.7
C4	226.2	232.8
C5	378.1	387.1
C6	265.1	297.4

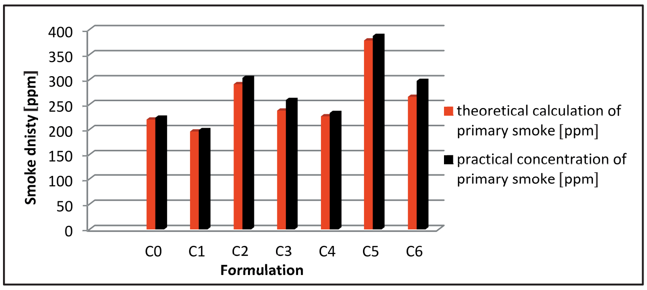

Figure14 Theoretical and practical results of primary smoke concentration.

obtained due to insufficient surface treatment for the available ammonium nitrate which needs specific preparation and burning rate accelerator to adjust the desirable ballistic performance. For combustion temperature, all formulations have acceptable and lower values compared with conventional propellant which realized the safety of motor case during the mission and enhances the thermal insulation characteristics.

6.2 Results of primary smoke concentration measurements

According to the combustion of seven propellants types of accurately weighed tested propellant sample, combustion gases were exhausted through mounted filter paper which is trapped and weighed before and after the test. Figures 12 and 13 show the trapped solid particles of primary smoke determined and recorded by simple and accurate for primary smoke concentration. The amount of primary smoke measured experimentally and those obtained from thermochemical calculation are listed together in the Table 3 and Figure 14.

It is clear from the results that the values of primary smoke concentration obtained from both theoretical and practical measurement for all tested propellant formulations are close to each other this emphasizes the high accuracy of both applied experimental setup technique and the results of ICT thermodynamic code employed in this work. Neutralized propellant C1 has the lowest concentration of primary smoke compared to conventional one C0 and when compared to the other propellant formulations due to the low concentration of magnesium metal (13%) in C1 formulation. The higher amount of primary smoke in all propellant formulation is scavenger one C5 due to the high content of aluminum metal (20%) and the formation of sodium chloride (NaCl) and aluminum oxide (Al_2O_3).

6.3 Results of secondary smoke concentration measurements

The amount of secondary smoke measured experimentally using portable gas analyzer (Gasmet dx4030) and those obtained from thermochemical calculation are listed together for comparison in the Table 4 and Figure 15. The values of secondary smoke concentration which obtained from either theoretical calculations or practical measurements are approximately the same. Those results show the great advantage of the

Table 4 Concentration of secondary smoke of propellant formulations.

Formulation	Concentration of secondary smoke [ppm]	
	Theoretical	Measured
C0	143.7	146.8
C1	0	1.7
C2	0	0.17
C3	38.5	48.6
C4	0	0.31
C5	21	24.8
C6	55.8	58.0

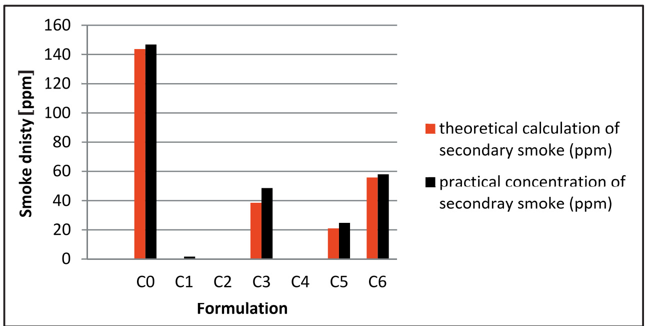

Figure 15 Concentration of secondary smoke of propellant formulations.

neutralized propellant family (C1 and C4) and non-chlorine propellant (C2) over all other formulations including the conventional one (C0) not only in reducing the amount of secondary smoke but also we can emphasize that those types of propellants are secondary smoke-free. The scavenger propellant (C5) and formulations (C3 and C6) have a markedly small amount of secondary smoke when compared with that produced from conventional one (C0).

7. Conclusion

The measuring smoke density values which were carried out by primary smoke device was fast-effective tool for evaluating the accuracy of primary smoke devices comparing with theoretical results getting from ICT thermodynamic code which not only keep the acceptable performance of the smoke device but also markedly can use for many types of rocket motor and also gas generators for evaluating the concentration of primary smoke which contains soot and choosing the lowest amount of smoke that effect on actuation systems. Neutralized propellant family (neutralized and neutralized modified with HMX has the best results in all other families as the density is nearly the same compared to conventional aluminized propellant so we can replace the propellant in the same volume and has very good results of performance parameters than the conventional propellant and also the lowest amount of primary smoke in all families. Neutralized propellant is the best results for the ballistic performance including modified neutralized propellant also for secondary smoke is reduced and eliminate HCl gas which realized that the reaction of magnesium hydroxide with HCl occur and for the modified scavenger propellant have also good results and also sure that the reaction occurs but the ammonium nitrate and even the modified one with HMX have good results for eliminating the secondary smoke. Additions of nitramine energetic material as HMX not only modified the ballistic performance but also reduced the source of producing HCl gas in which HCl is the main reason for secondary smoke. According to the obtained theoretical and practical results from this work, the neutralized rocket propellant family (C1 and C4) may replace the conventional one because they are considered as secondary smoke free and in the same time they give high specific impulse values relative to their densities.

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