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Optimisation and performance evaluation of an environmentally friendly rocket composite propellant

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ABSTRACT

This paper investigated the effects of laboratory synthesised rocket composite propellants' formulations on propellant performance parameters using response surface methodology (RSM). For the sake of a safe environment, the propellant is made of chemical constituents that have less severe effects on air quality. Experimental measurements of propellant performance parameters were carried out using the ballistic evaluation method. The thrust generated during combustion, the burn time and the propellant combustion temperature were measured using a data acquisition facility equipped with a button load cell and a thermocouple. Specific impulse, density, temperature, characteristic velocity, molecular weight and heat ratio were computed from the measured parameters. The resulting optimum rocket composite propellant formulations were 65.1%, 18.1%, 9.1%, 3.6%, 2.2% and 1.9% for the oxidiser, binder, fuel, opacifier, burning rate enhancer and catalysts, respectively. The corresponding performance indices including specific impulse, propellant density, propellant temperature, propellant characteristic velocity, molecular weight and heat ratio were 122.03 s, 1697.08 kg m⁻³, 2025.07 K, 987.65 m s⁻¹, 31.50 kg mol⁻¹ and 1.02, respectively. Findings revealed the accuracy and the adequacy of RSM in analysing and optimising the effects of composite rocket propellant formulation on its performance parameters and also a step towards producing an environmentally friendly rocket composite propellant.

KEYWORDS

Design; environment; factors; propellant; optimisation

1. Introduction

As the space rocket propulsion technology advances, there is a continuous demand for environmental-friendly rocket composite propellants with low pollution and high performance [1]. The present focus is to develop rocket composite propellants which have high-performance indices and less severe effects on the environment [2]. Developing rocket composite propellant and improving its performance characteristic involve a combination of a lot of different chemical components. The design of a rocket composite propellant whose focus is on meeting certain mission requirements can be specified as a search for a set of chemical interaction between the chemical constituents within the overall formulation. A wide variation in such chemical constituents and the large number of possible combinations of these chemical constituents turns the composite propellant formulation

into a search with a very large number of possible answers. These interactions play a very serious role in controlling the propellant's performance parameters [3] and emissions of air pollutants associated with rocket propellant combustion [4]. However, the interactions of the propellant ingredients when combined into a practical solid propellant are too complex. Controlling such interactions so as to protect the air around is still lacking and unavailable, thus, making rocket composite propellant technology difficult, expensive and pose a serious threat to air quality.

Many researches on composite solid propellants have been performed over the past few decades and much progress has been made [5–7]. However, most of the propellants were made with constituents (ammonium perchlorate, magnesium, boron) that are hazardous to lives and properties. Ammonium perchlorate-based propellant liberates hydrochloric acid which is environmentally undesirable [8]. Magnesium present in the propellant is attributed to the emissions of dangerous magnesium oxide [9]. Ammonium nitrate produces purely gaseous products. This reduces the amount of particulate emissions. However, this constituent has a low surface temperature and a low burn rate. The decomposition chemistry of ammonium nitrate is largely responsible for the low energy released [10]. Thus, this makes the ignition of the propellant a big challenge. Ammonium dinitramide decomposes very rapidly and is potentially a good propellant [11–13]. However, this constituent from the reproductive, fertility, pre-implantation and post-implantation studies is embryotoxic, and a mouse embryo toxicity study also showed that ADN affects the embryo [14]. Therefore, design or improving the propellant performances which meet future emissions regulations with a minimal pollution cost penalty, an optimised mixture ratio, good ballistic characteristics and readily available constituents is of great importance and remains highly empirical.

Optimisation is essentially required during the design process, in order to ensure that the best possible design among all achievable designs is being acquired. The composite propellant design is a multivariable design thus optimisation is applicable. Response surface methodology (RSM) could be used to optimise the effects of propellant formulation on its performance parameters [15]. It is based on the centre composite design which is a quadratic model, it has uniform precision, larger runs and highly efficient. It is applied when a response or a set of responses of interest are influenced by several variables [16]. It reduces the design time and has the ability to efficiently and consistently realise design behaviour [17]. The factors are chemical constituents of the composite propellant while the responses are the propellant performance parameters such as the specific impulse, temperature, density, characteristic velocity, the molecular weight and heat ratio. Therefore, the effects of chemical interaction among the reactive ingredients of the propellant on its performance parameters are examined in this study using RSM, as this significantly helps in arriving at an optimised formulation which in turns controls the air emissions from its combustion.

2. Material and methods

In applying the optimisation approach to formulate the rocket composite propellant, regression equations were used to relate the composite propellant formulation ingredients with the propellant performance parameters [18]. This method involves the selection of the propellant constituents, RSM model development, propellant preparation, performance parameters measurements, model optimisation and model validation.

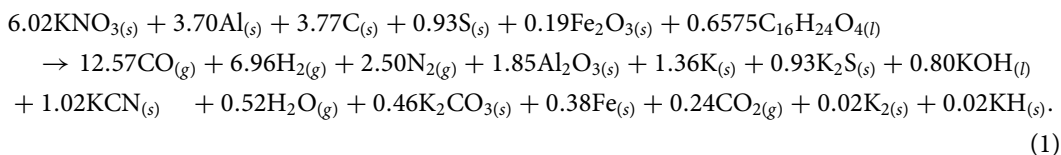
Variables which have major effects on the propellant formulation were selected. This was carried out to determine which of the several experimental variables and their interactions present more significant effects [19]. The selected input variables (factors) were the composite propellant ingredients such as oxidiser (potassium nitrate), binder (epoxy 201), fuel (aluminium), opacifier (carbon), burning rate enhancer (sulphur) and catalyst (iron II oxide). The output variables were the propellant performance parameters (responses) such as propellant specific impulse, density, combustion temperature, characteristic velocity, molecular weight and heat ratio (Table 1).

RSM central composite design (CCD) (in the Design Expert^R version 6.0.8) was used to design the experiments with the focus on optimising responses of the designed composite propellant

Table 1. Major propellant variables and the design centre points.

Inputs	Ingredients	Symbol	Centre points (kg)
	Amount of oxidizer	A	61
	Amount of binders	B	18.5
	Amount of fuel	C	10
	Amount of opacifier	D	4.5
	Amount of burn rate enhancer	E	3
	Amount of catalysts	F	3
Outputs	Propellant performance parameters		
	Specific impulse	I_{sp}	
	Density (kg m^{-3})	ρ_p	
	Temperature (K)	T	
	Characteristic velocity (m s^{-1})	C^*	
	Molecular weight (kg mol^{-1})	M	
	Heat ratio	k	

formulations [16]. CCD enables the estimation of the regression parameters to fit a polynomial regression model to a given response [16,20]. RSM has proven to be a useful mathematical approach that has been used successfully to investigate the combinatorial effects of several process variables on the dependent variable(s) [21]. In addition, the CCD has been described as a powerful design tool that is being used to describe factor interaction in multifactor complex processes [21]. CCD allows for sequential experimentation and fits quadratic responses well when used with the RSM [22]. The choice of the varied parameters was made based on the derived stoichiometric equations in place of the established formulation. The propellant compositions at the centre points were derived from the propellant stoichiometric equation of combustion as shown in Equation (1). Then, 20% increment (variation) about the formulation centre point was chosen as the acceptable experimental limits. Codification of the level of the variables was carried out by transforming each studied real propellant component ratio into coordinates inside a scaled with dimensionless values. This must be proportional at its localisation in the experimental space. Codification is of concern because it enables the investigation of variables of different orders of magnitude without a greater influence on the evaluation of the lesser [18]. The resulting experiments from the RSM regression analysis are shown in Table 2.



The propellant was produced using the compression method in order to obtain a more compact composite propellant [20]. The selected ingredients of the composite propellant were potassium nitrate, epoxy 201, aluminium, carbon, sulphur and Iron II oxide, and their proportion were in the ranges as follows 40–65%, 17–25%, 8–25%, 1–8%, 1–5% and 1–5%, respectively. These components were chosen for their consistent performance, availability, safety of handling and castability as reported by Rahmat et al. [23], Yang [24]. For a predetermined weight of the propellant, the appropriate quantity of each ingredient was weighed as obtained from the CCD experimental design. In the method, the oxidiser crystal (KNO_3) was weighed, dried in an oven preheated to 65–95 °C for 2 h or more, after which it was grinded to a fine powder using an electric grinder (typically for 40–50 s per 50 g batch). The dried oxidiser powder crystal was then blended with the other additive constituents (carbon, sulphur and Iron II oxide) for about 15 min. This is to ensure proper coating of the individual oxidiser particles, as this intensifies the performance [24]. Separately, the fuel and the binder were blended together until all the metal fuel is coated by the binder [8]. It is conventional in pyrotechnic designs to avoid combining oxidiser with metal fuel at the initial stage. Finally, the premixed metal

Table 2. CCD arrangement for propellant formulations.

Runs	Coded level					Actual composition (mass fraction)					
	X_1	X_2	X_3	X_4	X_5	A	B	C	D	E	F
1	0	0	-1	0	0	61.0	18.5	6.6	5.9	4.0	4.0
2	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
3	-1	1	1	-1	-1	57.7	22.2	10.8	3.3	2.7	3.3
4	-1	-1	1	-1	1	57.7	17.5	13.3	4.1	4.1	3.3
5	0	0	0	1	0	61.0	18.5	10.0	5.8	2.4	2.3
6	-1	-1	1	1	-1	57.7	17.5	13.3	5.6	2.6	3.3
7	-1	-1	-1	-1	1	57.7	17.5	10.7	5.0	5.0	4.1
8	1	1	-1	1	-1	63.8	19.0	7.4	4.8	2.2	2.8
9	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
10	-1	-1	-1	-1	-1	57.7	17.5	10.7	5.0	4.1	5.0
11	1	1	-1	-1	-1	63.8	19.0	7.4	3.5	2.8	3.5
12	0	0	1	0	0	61.0	18.5	12.1	3.6	2.4	2.4
13	1	-1	1	1	1	63.8	14.9	11.4	4.8	2.8	2.3
14	1	1	1	-1	-1	63.8	19.0	9.2	2.8	2.3	2.9
15	-1	1	1	1	1	57.7	22.2	10.8	4.6	2.6	2.1
16	1	1	-1	1	1	63.8	19.0	7.4	4.8	2.7	2.3
17	1	-1	1	-1	-1	63.8	14.9	11.4	3.5	2.8	3.6
18	-1	1	-1	1	1	57.7	22.2	8.6	5.6	3.2	2.7
19	0	-1	0	0	0	61.0	11.7	13.3	6.0	4.0	4.0
20	0	0	0	0	-1	61.0	18.5	10.0	4.5	2.1	3.9
21	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
22	1	1	1	-1	1	63.8	19.0	9.2	2.8	2.8	2.4
23	0	0	0	0	1	61.0	18.5	10.0	4.5	3.6	2.4
24	1	-1	-1	-1	-1	63.8	14.9	9.1	4.3	3.5	4.4
25	-1	1	1	-1	1	57.7	22.2	10.8	3.3	3.3	2.7
26	-1	1	1	1	-1	57.7	22.2	10.8	4.6	2.1	2.6
27	-1	-1	1	1	1	57.7	17.5	13.3	5.6	3.2	2.7
28	1	1	-1	-1	1	63.8	19.0	7.4	3.5	3.5	2.8
29	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
30	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
31	1	-1	1	-1	1	63.8	14.9	11.4	3.5	3.5	2.9
32	1	0	0	0	0	67.1	15.6	8.4	3.8	2.5	2.6
33	-1	-1	-1	1	1	57.7	17.5	10.7	6.9	4.0	3.2
34	-1	1	-1	-1	1	57.7	22.2	8.6	4.1	4.0	3.4
35	-1	-1	1	-1	-1	57.7	17.5	13.3	4.1	3.3	4.1
36	1	1	1	1	1	63.8	19.0	9.2	3.9	2.2	1.9
37	-1	1	-1	-1	-1	57.7	22.2	8.6	4.1	3.3	4.1
38	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
39	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0
40	1	-1	-1	-1	1	63.8	14.9	9.1	4.3	4.3	3.6
41	1	-1	-1	1	1	63.8	14.9	9.1	5.9	3.4	2.9
42	1	0	0	0	0	52.1	22.7	12.3	5.5	3.7	3.7
43	1	-1	1	1	-1	63.8	14.9	11.4	4.8	2.3	2.8
44	-1	-1	-1	1	-1	57.7	17.5	10.7	6.9	3.2	4.0
45	1	-1	-1	1	-1	63.8	14.9	9.1	5.9	2.8	3.5
46	0	0	0	-1	0	61.0	18.5	10.0	2.3	4.1	4.1
47	0	1	0	0	0	61.0	22.6	8.0	3.6	2.4	2.4
48	1	1	1	1	-1	63.8	19.0	9.2	3.9	1.8	2.3
49	-1	1	-1	1	-1	57.7	22.2	8.6	5.6	2.6	3.3
50	0	0	0	0	0	61.0	18.5	10.0	4.5	3.0	3.0

fuel–binder mixture and oxidiser-additives mixture were mixed together using a mixer (Figure 1(a)) as adopted in the previous study [25]. The mixing was allowed to proceed for 2 or more hours [26–28] for uniform and consistent mixing between the mixtures. A means of vacuum degassing for air bubbles removal was incorporated into the mixer (Figure 1(b)). This was done by including a suction pump for the removal of the air trapped inside the propellant grains. This was to allow a higher burning rate with higher and uniform density [29]. After the initial mixing, the curing agent was then added to the propellant and mixed again [30]. The propellant mixture was then cast into a special casting mould (Figure 1(c)) using the cast-in-place-core method to form the grain geometry and was then

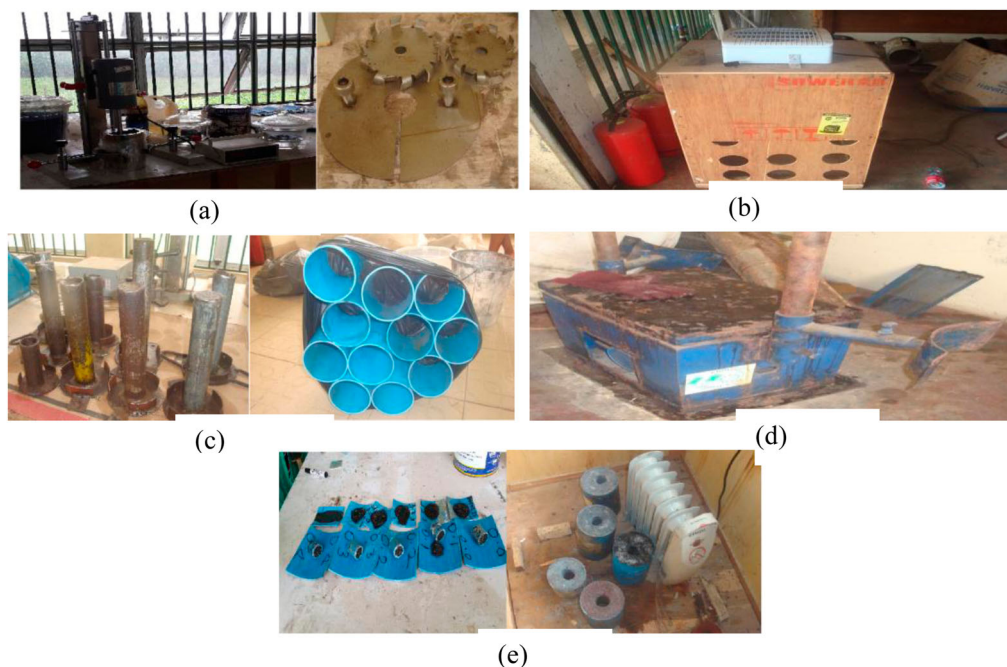


Figure 1. Typical propellant preparation. Mixer (b), vacuumiser (c), propellant casting moulds (d), and propellant casting vibrator (e) composite propellant samples and curing of propellants.

vibrated on a vibrator (Figure 1(d)) for 10 s to ensure consistency and relatively flaw-free propellant grain. The casting mould was then removed after few hours, and the propellant was allowed to cure (solidify and dry) usually for 72 h or more as shown in Figure 1(e) [31].

Experimental measurements of propellant performance parameters were carried out using the standard ballistic evaluation method [32] at the premises of the Department of Chemical Engineering, Faculty of Technology, Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria. This method involved the design of the thrust chamber and the required pressure and the thermal energy for ignition of the propellant grain was produced by the igniters (Figure 2(a)).

Generated thrust during combustion, the burn time and the propellant combustion temperature were measured using a data acquisition facility (Figure 2(b)), equipped with a button load cell (CZL204E, 31370 characterised by 0–2000 N force with 0.037% accuracy) (Figure 2(c)), Acquisition Module (DI-194 characterised by 4-channel input, 10 bit resolution, measure 10 V (plus or minus) AC or DC and two digital inputs for remote start/stop and remote event marker control up to 240 samples per second) (Figure 2(d)) and an embedded thermocouple.

The specific impulse was derived using the thrust per unit weight flow rate of the propellant (Equation (2)) as reported [27]:

$$I_{sp} = F / \dot{m}_p^* \times g, \quad (2)$$

where I_{sp} is the specific impulse, F is the measured average thrust (N) and $\dot{m}_p^*$ is mass flow rate of combustion gases (kg s^{-1}). The propellant density, combustion temperature, characteristic velocity, the molecular weight and heat ratio were computed from the measured parameters as described in Equations (3)–(7), respectively.

$$\rho_p = \dot{m}_p^* / A_e V, \quad (3)$$

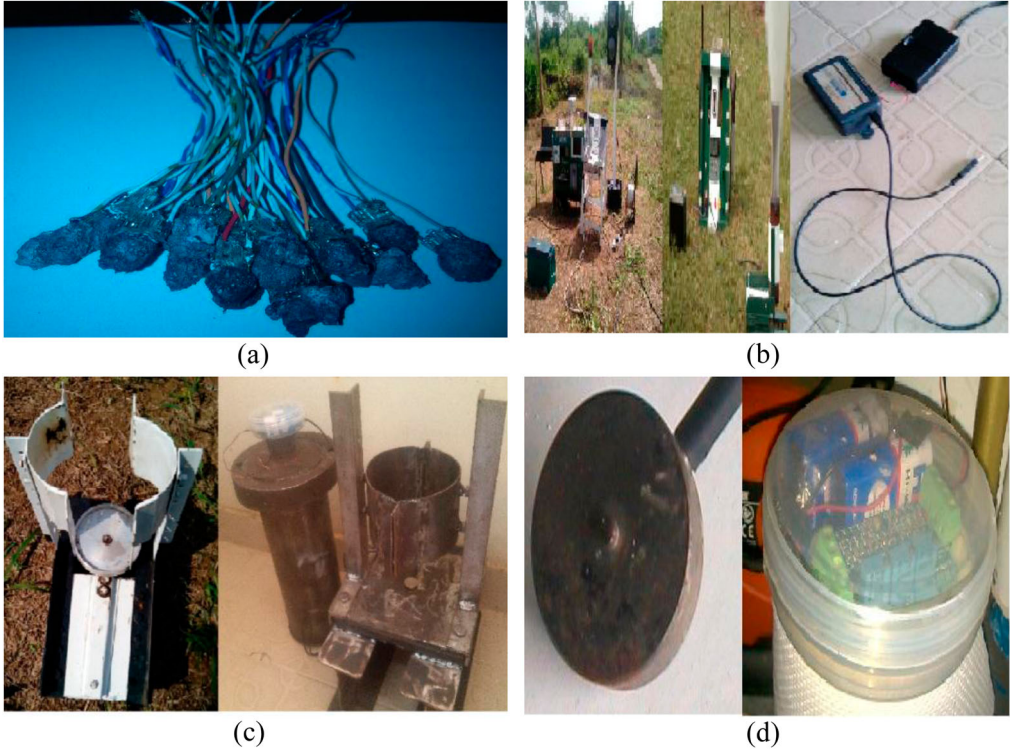


Figure 2. Ballistic experimental set-up. (a) Igniters, (b) data acquisition system, (c) the load cell setup and (d) the button load cell and data acquisition module.

where ρ_p is the propellant density of the each of the components (kg m^{-3}), $\dot{m}_p^*$ is the propellant mass flow (kg s^{-1}), A_e is the nozzle exit area (m^2) and V_e is the exit velocity (m s^{-1}).

(a) Igniters (b) Data Acquisition System (c) The Load Cell Setup (d) The Button Load Cell and Data Acquisition Module

$$T_c = T_e(P_c)^{(\gamma-1)/\gamma}(\gamma+1) / 2(P_e)^{(\gamma-1)/\gamma}, \quad (4)$$

where T_c is the adiabatic flame temperature (K), T_e is the nozzle exit temperature (K), P_e is the nozzle exit pressure (assumed to be atmospheric pressure) (Pa) and γ is the heat ratio.

$$C^* = (\sqrt{\gamma R_g T_c})(V_e)^2 / R_g T_c (C_f)^2, \quad (5)$$

where C^* is the propellant characteristic velocity (m s^{-1}), R_g is the gas constant (J (kg K)^{-1}), γ is the heat ratio, V_e is the exit velocity, C_f nozzle coefficient and A_T is the nozzle throat area in (m^2).

$$M = 2R' \gamma \left(T_c - T / (\gamma - 1)(I_{sp}g)^2 \right), \quad (6)$$

$$C^* = \sqrt{R' T_c / \gamma \left(\frac{2}{\gamma + 1} \right)^{\frac{\gamma + 1}{\gamma - 1}}}. \quad (7)$$

R' is the universal constant, while the propellant heat ratio was iterated from all the equations. The heat ratio was obtained from the value that equates the measure characteristic velocity. The details of the measured propellant performance parameters are shown in Table 3.

After acquiring the values of the propellant performance parameters from each of the experimental point from the CCD analysis, second-order polynomial equations were fitted to describe the behaviour of the responses (propellant performance parameters) with the factors (propellant ingredients) based on the levels of values that were studied (i.e. generating the lowest residual) [16]. Matrix notations were formulated, with the Y_h representing the responses and X_i representing the

Table 3. Measure composite propellant performance parameters from the CCD experiments.

Runs	Specific impulse (s)	Density (kg m^{-3})	Temperature (K)	Characteristic velocity (m s^{-1})	Molecular weight (kg mol^{-1})	Heat ratio
1	134.4	1859.2	1682	926.714	34.349	1.1453
2	144.1	1866.6	1840	990.112	33.733	1.1737
3	144.6	1822.9	1802	1009.22	31.893	1.1736
4	151.2	1897.1	2086	1033.91	33.714	1.18
5	141.7	1855.9	1823	973.165	33.687	1.1679
6	147.7	1892.6	1293	1020.23	33.834	1.161
7	143.3	1892.1	1863	971.398	33.743	1.1783
8	139.9	1847.8	1786	949.117	34.492	1.1581
9	144.1	1866.6	1840	990.112	33.733	1.1737
10	142.5	1901.4	1838	925.891	33.883	1.1765
11	142.0	1858.4	1824	963.259	34.771	1.1605
12	149.6	1871.5	1891	1024.49	32.985	1.1907
13	149.0	1918.4	1347	1012.24	35.018	1.1882
14	147.0	1861.5	1883	994.928	34.189	1.1713
15	142.8	1808.0	1794	1002.37	31.839	1.1691
16	140.0	1842.9	1783	966.003	34.339	1.1604
17	149.9	1936.3	2076	1015.96	36.111	1.1804
18	137.4	1802.8	1647	952.683	32.023	1.164
19	151.0	1990.2	2319	1032.69	37.106	1.1703
20	143.3	1875.7	1844	1004.44	33.958	1.1694
21	144.1	1866.6	1840	990.112	33.733	1.1737
22	147.4	1856.5	1888	999.013	34.121	1.1729
23	144.6	1860.6	1838	976.854	33.584	1.1767
24	144.0	1932.6	1987	980.572	36.947	1.1654
25	145.1	1817.2	1804	1018.34	31.856	1.1759
26	142.3	1812.7	1780	995.66	31.904	1.1684
27	148.7	1886.5	2083	1019.37	34.072	1.1695
28	142.5	1851.5	1829	966.948	34.603	1.1627
29	144.1	1866.6	1840	990.112	33.733	1.1737
30	144.1	1866.6	1840	990.112	33.733	1.1737
31	150.5	1928.8	2140	1010.5	36.295	1.1767
32	145.2	1903.2	1998	984.656	37.091	1.1624
33	139.7	1877.3	1858	969.02	33.805	1.1668
34	139.6	1813.4	1697	968.441	32.197	1.1674
35	149.9	1905.4	2020	1040.92	33.502	1.1838
36	145.3	1848.4	1845	1040.04	33.873	1.1712
37	139.0	1820.1	1703	959.724	32.369	1.164
38	144.1	1866.6	1840	990.112	33.733	1.1737
39	144.1	1866.6	1840	990.112	33.733	1.1737
40	144.6	1924.1	1993	984.565	36.844	1.1683
41	142.6	1911.8	1893	968.197	35.788	1.1701
42	145.1	1817.1	1967	994.501	30.852	1.1528
43	148.5	1923.7	1959	1006.63	35.08	1.1864
44	138.9	1885.4	1830	947.105	33.869	1.1659
45	142.1	1918.1	1887	971.641	35.886	1.1681
46	148.0	1884.2	1928	1006.05	34.348	1.1769
47	140.8	1799.2	1730	964.448	32.529	1.1617
48	144.9	1852.4	1846	1063.87	33.975	1.1693
49	136.8	1808.4	1654	949.879	32.171	1.1611
50	144.1	1866.6	1840	990.112	33.733	1.1737

coded ratios with an m independent number of propellant chemical ingredients X ($i = 1, 2, \dots, m$). The model was set for n number of mathematical functions, f ($h = 1, 2, \dots, n$) associated with each response variable, Y_h (Equation 8) as reported [15]:

$$Y_h = f_h(X_1, X_2, \dots, X_m). \quad (8)$$

Then, a second-order polynomial equation (Equation (9)) was applied to approximate this function with $n = 6$ and $m = 5$.

$$Y_h = b_{h_0} + \sum_{i=1}^m b_{h_i} X_i + \sum_{i=1}^m b_{h_{ii}} X_i^2 + \sum_{i \neq j=1}^m b_{h_{ij}} X_i X_j, \quad (9)$$

where b_{h_0} is the value of the fitted response at the centre point of the design, i.e. (0, 0), and b_{h_i} , $b_{h_{ii}}$ and $b_{h_{ij}}$ are regression main, quadratic and interaction effects, respectively.

However, the mathematical models that were generated could not explain the experimental domain satisfactorily. However, analysis of variance (ANOVA) was carried out. This is a more reliable way to evaluate the quality of the fitted model [16]. In this analysis, the dispersion of the data variance was evaluated and several significance level parameters were computed including the sum of square, significance of regression, F distribution parameters and estimation of the residual errors for all observations. The RSM optimised composite rocket propellant formulations were then validated experimentally using the standard ballistic evaluation method. The validation experiment was to establish the accuracy of the RSM model.

3. Results and discussion

Results from the multiple regression and correlation analysis are presented in this section. The models explain all the main, linear, and quadratic effects of the propellant constituents on the performance parameters of the designed composite rocket propellant formulations. The regression coefficients as well as the correlation coefficient obtained for each model are presented in Table 4.

The predicted R^2 and adjusted R^2 for propellant specific impulses are 0.9756 and 0.892, respectively, while the precision R^2_{Pred} is 0.7925 and adequacy precision is given as 9.824. The statistical significance of the linear interaction between the epoxy resin and the aluminium is also presented. The significance of the quadratic effect of the potassium nitrate, the epoxy binder and the carbon to propellant density are presented in Table 4 with the predicted R^2 and adjusted R^2 for propellant density as 0.9993 and 0.9988, respectively. The precision R^2_{Pred} is 0.9967 and adequacy precision is 12.98. Temperature is another important parameter in boosting the performance of a good composite rocket propellant. Therefore, assessing the individual and collective contributions of the ingredients is very relevant. Also, in Table 4 the predicted R^2 and adjusted R^2 for propellant temperature are 0.9412 and 0.9232, respectively. The precision R^2_{Pred} is 0.8906 and adequacy precision is 5.837. R^2 and adjusted R^2 for propellant characteristic velocity are shown as 0.9801 and 0.8567, respectively. The precision R^2_{Pred} is 0.7664 and adequacy precision is 11.29. The predicted R^2 and adjusted R^2 for propellant molecular weight are 0.9777 and 0.8379, respectively. The precision R^2_{Pred} is 0.7497 and adequacy precision is 41.12. The predicted R^2 and adjusted R^2 for propellant heat ratio as presented in the same table are 0.9878 and 0.8275, respectively. The precision R^2_{Pred} is 0.7492 and adequacy precision is 9.293. The stability of this model can also be explained from the residual analysis (Figure 3). Data points on the residual plots presented are approximately linear. A non-linear pattern (such as an S-shaped curve) indicates non-normality in the error term. This observation is an indication that there is no sign of any problem in the data used. The highlighted runs do not differ more from the predicted values; hence, the variables are within the control limits.

The developed statistical model equations representing the chemical interaction between the composite propellant constituents and the effects (i.e. main, linear and quadratic) on the propellant

Table 4. Propellant constituents and performance parameters' interaction test.

Responses	Model factors	Coefficients	t-Values	p-Values	R ²	
Specific impulse (Y ₁)	Constant	124.1444	4.513695	0.0001*	0.9756	
	X ₃	2.679334	41.22677	0.0001		
	X ₄	-1.65038	15.64201	0.0005		
	X ₂ X ₃	-1.10938	5.22164	0.0298		
	Significant at P-value L = 0.05 at 95% confident interval = 0.892, R ² _{Pred} = 0.7925, Adeq. Precision = 9.824					
Propellant Density (Y ₂)	Constant	1866.524918	2011.685559	0.0001	0.9993	
	X ₁	17.88536452	6845.993295	0.0001		
	X ₂	-38.34760808	31471.49964	0.0001		
	X ₃	2.529326138	136.9148458	0.0001		
	X ₄	-7.58202919	709.6019632	0.0001		
	X ₅	-204852683	219.8148455	0.0001		
	X ₁ ²	-1.20776329	40.05078751	0.0001		
	X ₂ ²	4.899871532	659.19939	0.0001		
	X ₄ ²	0.542325993	8.075478221	0.0081		
	X ₁ X ₂	1.809375	51.76338365	0.0001		
	X ₂ X ₄	0.809375	10.35774126	0.0032		
	X ₄ X ₅	0.403125	2.569478278	0.0001		
	Significant at P-value L = 0.05 at 95% confident interval R ² _{adj} = 0.9988, R ² _{Pred} = 0.9967, Adeq. Precision = 12.98					
	Propellant temperature (Y ₃)	Constant	1832.176423	1.741412		0.0845
X ₂		-69.00554317	9.755823	0.0040		
X ₄		-55.3573817	6.278372	0.0181		
Significant at P-value L = 0.05 at 95% confident interval R ² _{adj} = 0.9232, R ² _{Pred} = 0.8906, Adeq. Precision = 5.837						
Propellant characteristic velocity (Y ₄)	Constant	990.2391	5.843098	0.0001	0.9801	
	X ₂	-5.56359	4.68179	0.0389		
	X ₃	25.88849	101.3712	0.0001		
	Significant at P-value L = 0.05 at 95% confident interval R ² _{adj} = 0.8567, R ² _{Pred} = 0.7664, Adeq. Precision = 11.29					
Molecular weight (Y ₅)	Constant	33.73551	108.388	0.0001	0.9777	
	X ₁	1.165842	1268.154	0.0001		
	X ₂	-0.892619	743.3796	0.0001		
	X ₃	-0.22391	46.77571	0.0001		
	X ₄	-0.15335	21.94103	0.0001		
	X ₂ ²	0.193974	45.03883	0.0001		
	X ₁ X ₄	-0.18094	22.56703	0.0001		
	Significant at P-value L = 0.05 at 95% confident interval R ² _{adj} = 0.8379, R ² _{Pred} = 0.7497, Adeq. Precision = 41.12					
Heat ratio (Y ₆)	Constant	1.174006	6.955506	0.0001	0.9878	
	X ₃	0.005728	67.28621	0.0001		
	X ₄	-0.00188	7.263047	0.0116		
	X ₁ ²	-0.00257	17.39712	0.0003		
	X ₁ X ₃	0.002047	6.34893	0.0175		
	X ₁ X ₄	0.002728	11.27837	0.0022		
	Significant at P-value L = 0.05 at 95% confident interval R ² _{adj} = 0.8275, R ² _{Pred} = 0.7492, adeq. precision = 9.293					

performance parameters are presented in Equations (10)–(15). The results generated by these equations are summarised in Table 5.

Propellant specific impulse:

$$Y_1 = 124.14 + 2.69X_3 - 1.65X_4 - 1.11X_2X_3. \quad (10)$$

Propellant density:

$$Y_2 = 1866.52 + 17.89X_1 - 38.35X_2 + 2.53X_3 - 5.76X_4 - 3.20X_5 - 1.21X_1^2 + 4.90X_2^2 + 0.54X_4^2 + 1.81X_1X_2 + 0.81X_2X_4 + 0.40X_4X_5. \quad (11)$$

Propellant temperature:

$$Y_3 = 1832.18 - 69.01X_2 - 55.36X_4. \quad (12)$$

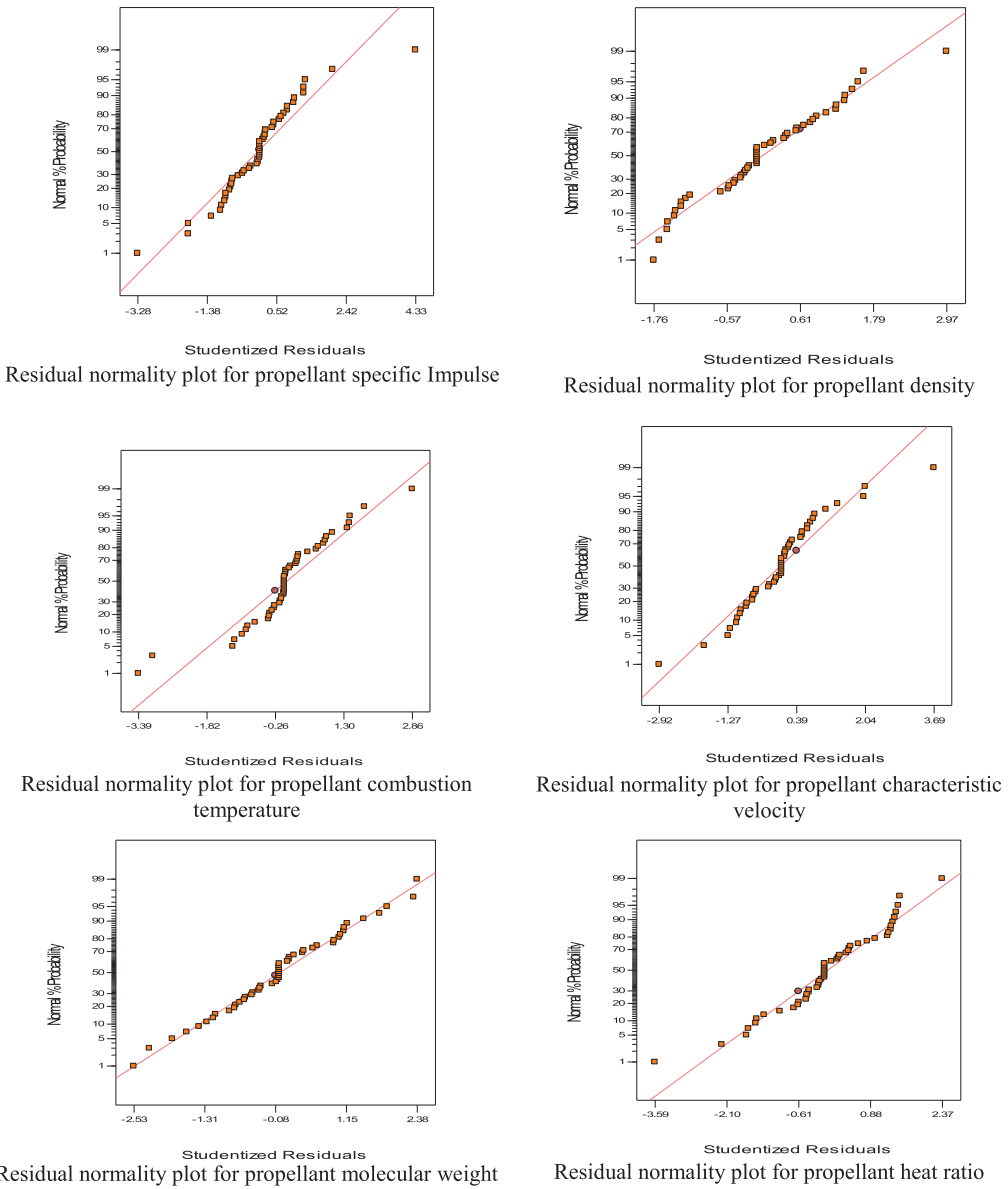


Figure 3. Residual normality plot for composite rocket propellant performance parameters.

Propellant characteristic velocity:

$$Y_4 = 990.24 - 5.56X_2 + 25.89X_3. \quad (13)$$

Molecular weight:

$$Y_5 = 33.74 + 1.17X_1 - 0.89X_2 - 0.22X_3 - 0.15X_4 + 0.19X_2^2 - 0.18X_1X_4. \quad (14)$$

Table 5. ANOVA tests.

Response	Source of variation	Sum of squares	d.f	Mean square	<i>p</i> -Value	<i>R</i> -square	<i>F</i> -Values	Adjusted <i>R</i> ²	Predicted <i>R</i> ²	adequate precision	Standard Error
Specific impulse	Regression	877.64	20	43.88	0.0001*	0.9756	57.74	0.8920	0.7925	9.824	0.87
	Residual	21.95	29	0.76							
	Total	899.59	49								
Propellant density	Regression	81429.81	20	4071.49	0.0001*	0.9993	2066.75	0.9988	0.9967	12.98	1.40
	Residual	57.04	29	1.97							
	Total	81486.85	49								
Propellant temperature	Regression	117555.88	20	5877.79	0.0001*	0.9412	23.21	0.9232	0.8906	5.837	15.91
	Residual	7344.12	29	253.25							
	Total	124900	49								
Characteristic velocity	Regression	40938.95	20	2046.95	0.0001*	0.9801	71.42	0.8567	0.7154	11.29	5.35
	Residual	831.23	29	28.66							
	Total	41770.18	49								
Molecular weight	Regression	99.71	20	4.99	0.0001*	0.9777	63.97	0.8379	0.7497	41.12	0.28
	Residual	2.27	29	0.078							
	Total	101.98	49								
Heat ratio	Regression	0.00351	20	0.00018	0.0001*	0.9878	128.57	0.8273	0.7492	9.29	0.0012
	Residual	0.00004	29	0.0000014							
	Total	0.00355	49								

Note: Significant at *P*-value *L* = 0.05 at 95% confident interval.

Heat ratio:

$$Y_6 = 1.17 + 0.005728X_3 - 0.001882X_4 - 0.002571X_1^2 + 0.002047X_1X_3 + 0.002728X_1X_4. \quad (15)$$

ANOVA for all the model responses which assess how well the model represents the design data is also presented in Table 5. The results reveal that the model F -values of 57.74, 2066.75, 23.21, 71.42, 63.97 and 128.57 at a probability greater than F less than 0.05 for the responses such as specific impulse, propellant density, temperature, characteristic velocity, molecular weight and heat ratio, respectively, are calculated. Another goal here is to understand how the response changes (composite propellant performance parameters) in a given direction by adjusting the design variables (two most significant constituents of the propellant formulations). The response surface of each of the analysed propellant performance parameters is visualised and presented graphically (Figure 4(a–f)). The graph is helpful to see the shape of a response surface. Table 6 shows 10 different optimised composite propellant formulations as suggested from the RSM optimisation analysis with their predicted and experimentally measured performance parameters.

The picture of the composite propellant produced from each of the developed propellant formulation is shown in Figure 5, respectively. It is established that the predicted optimum percentage of the constituents is as follows: the oxidiser ranges from 64.6% to 65.5%, the binder is between 17.8% and 18.2%, the fuel ranges 9.0% to 9.4%, opacifier is from 3.6% to 3.7%, burning rate enhancer from 2.1% to 2.4% and the catalysts is from 1.8% to 1.9%. The predicted optimum performance characteristics of these propellants also range as follows: specific impulse is from 128.217 to 130.492 s, the propellant density is predicted to be from 1895.71 to 1923.24 kg m⁻³, propellant temperature ranges from 1851.14 to 2094.22 K, the propellant characteristic velocity ranges from 1017.43 to 1029.22 m s⁻¹, molecular weight is from 33.1337 to 35.3143 kg mol⁻¹ and heat ratio is from 1.17415 to 1.18319. Meanwhile, the measured performance parameters are as stated: specific impulse is from 85 to 122.03 s, the propellant density is from 1697.074 to 11697.07 kg m⁻³, propellant temperature ranges from 1768.61 to 2025.07 K, the propellant characteristic velocity ranges from 695.64 to 987.65 m s⁻¹, molecular weight is from 31.50 to 38.32 kg mol⁻¹ and the heat ratio is from 1.0162 to 1.0171.

A solid rocket motor's operation and design depend on the combustion characteristics of the propellant, and the ultimate success of the motor depends significantly on knowledge of its burning rate behaviour under all operating and design limiting conditions (mainly pressure and temperature) [33]. Experimental measurement and validation of a propellant's performance parameters are important [34,35]. The design of composite rocket propellants requires that its performance characteristics be predicted and validated. From the RSM model prediction and the validation of composite propellant performance parameters, the measured parameters are observed to be close to the model predicted values [36–38]. This is an indication that the RSM model is perfectly accurate in its computations. The slight difference in the values could be attributed to the heat loss through walls of the combustion chamber, occurrence of erosive burning and propellant, and motor manufacturing and design imperfections. These imperfections could be from the purity of the chemicals, improper mixing, presence of cracks and void space, the grain and possible errors from the ignite [39].

The desire regression model needs to have significant variables and produce a high coefficient of determination (R^2) [40]. Low p -value and high coefficient of determination indicate that changes in the predictors (i.e. factor or propellant ingredients) are related to changes in the responses (i.e. propellant performance parameters) and that the model explains a lot of the response variability (i.e. uncertainty) [41]. The correlation coefficients for the responses are quite high for response surfaces and indicate that the fitted quadratic models accounted for over 94% of the variance. The statistical adjusted R^2_{adj} is a modified R^2 that has been adjusted for a number of predictors (or propellant ingredients) in the model. It increases only if the additional term improves the model, which would be expected by chance and vice versa. It provides an unbiased estimate of the R^2 and is always lower than the R^2 [42]. Therefore, the adjusted R^2_{adj} for all the propellant performance parameters responses are in close agreement with the literature. The predicted R^2 indicates how well regression

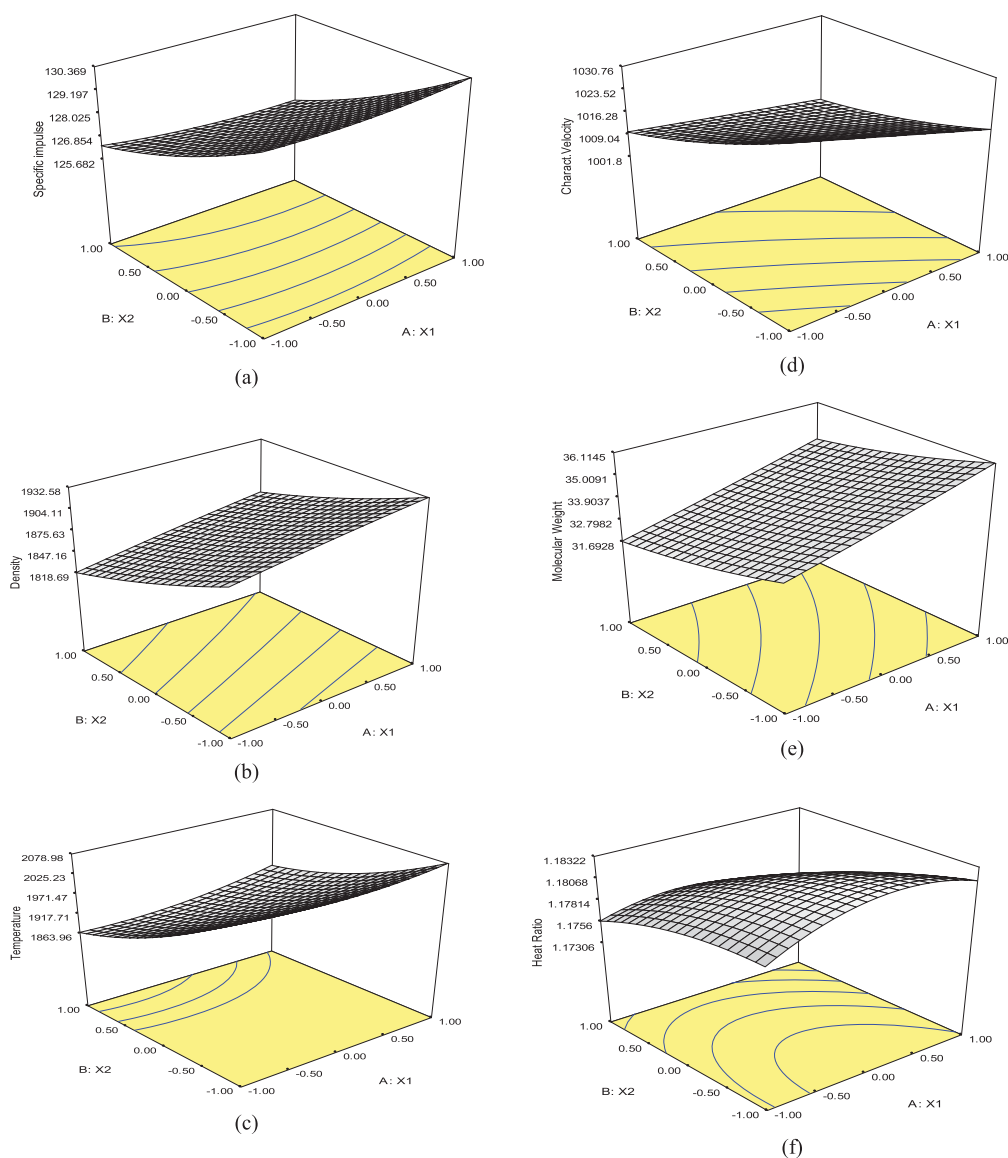


Figure 4. Response surface plots for propellant performance indices. (a) Response surface plot for the propellant specific impulse model. (b) Response surface plot of the propellant density model. (c) Response surface plot of the propellant burning temperature model. (d) Response surface plot for the propellant characteristic velocity model. (e) The response surface plot for propellant of the propellant molecular weight model. (f) The response surface plot of the propellant heat ratio model.

models predict the responses. It helps in preventing over-fitting (i.e. when the predicted R^2 is much lower than the R^2 , that is, the model has too many terms) of the model [18]. The obtained predicted R^2 for all the propellant performance parameters show that the models equations are not over-fitted. Also, the adequate precision of the responses is also greater than 4. These measure the signal to noise ratio. Therefore, all the values for all the responses indicate adequate signal and these models can be used to navigate the design space. The overall significance of the composite propellant ingredients to the performance parameters is explained by the F -values. The model F -values at a probability greater than F less than 0.05 for all the responses implies that the models' predictors (i.e. propellant reacting constituents) are significantly related to the performance parameters.

Table 6. Validation of the composite propellant performance parameters from optimised formulations.

Oxidiser (%)	Binders (%)	Fuel (%)	Opacifier (%)	Burn rate Enhancer (%)	Catalysts (%)	Specific impulse (s)		Propellant density (kg/m ³)		Temperature (K)		Characteristic velocity (m/s)		Molecular weight (kg/mole)		Heat Ratio	
						Predicted	Measured	Predicted	Measured	Predicted	Measured	Predicted	Measured	Predicted	Measured	Predicted	Measured
64.7	18.1	9.3	3.7	2.3	1.9	130.06	116.06	1900.94	1697.076	2076.58	1973.94	1030.71	939.26	33.6807	31.57	1.1794	1.0164
64.7	18.2	9.4	3.7	2.1	1.9	130.02	108.18	1902.35	1697.075	2048.21	1837.78	1029.92	875.49	33.6607	33.17	1.1792	1.0162
64.5	18.2	9.4	3.7	2.2	2	129.96	110.11	1902.47	1697.072	2053.45	1768.61	1029.58	891.12	33.7282	38.16	1.17939	1.0163
64.6	18.1	9.4	3.7	2.3	1.9	130.05	110.83	1903.56	1697.070	2094.22	1884.44	1027.7	896.96	34.1337	32.48	1.18172	1.0172
64.8	18	9.3	3.7	2.3	1.9	129.80	107.17	1895.71	1697.075	2057.72	1811.01	1029.2	867.32	33.5335	33.53	1.17941	1.0164
64.9	18	9.3	3.7	2.2	1.9	129.65	85.95	1905.73	1697.075	1973.34	1959.02	1022.97	695.64	33.6635	36.98	1.17853	1.0165
64.5	18.3	9.3	3.7	2.3	1.9	129.34	86.38	1921.28	1697.074	2013.77	1574.11	1024.38	699.12	34.6756	37.39	1.18295	1.0179
65.1	18	9.1	3.6	2.4	1.8	129.55	98.56	1923.24	1697.076	2045.71	1747.34	1022.21	797.71	35.0292	38.11	1.18319	1.0171
65.1	18.1	9.1	3.6	2.2	1.9	130.49	122.03	1919.06	1697.077	2050.52	2025.07	1017.43	987.65	35.3143	31.50	1.18317	1.0171
65.5	17.8	9	3.6	2.3	1.8	128.22	95.34	1899.5	1697.075	1851.14	1630.10	1023.59	771.65	33.5779	38.32	1.17415	1.0161



Figure 5. The typical laboratory synthesised rocket composite propellant.

The statistical significance between each composite rocket propellant ingredients (each constituent or factor) and performance parameters reveals that epoxy binder, aluminium, carbon and sulphur are all significant ($p < .0011$) for the statistical optimisation of the specific impulse of the designed composite propellant. The significance of the linear interaction between the epoxy resin and the aluminium is also established while other interaction has little or no effects. Density of a composite propellant is an important parameter which must be considered during the design. It is established that for maximising the density of the designed composite propellant, all the selected ingredients (potassium nitrate, epoxy binder, aluminium, carbon, sulphur and iron oxide) play significant contributions. The significance of the quadratic effect of the potassium nitrate, the epoxy binder and the carbon in increasing propellant was also established. The chemical interaction between the potassium nitrate and the epoxy binder, the interaction between the epoxy binder and the carbon and the interaction between the sulphur and the catalyst iron oxide posed good propellant density. Temperature is another important parameter in boosting the performance of a good composite rocket propellant. Therefore, assessing the individual and collective contributions of the ingredients is very relevant. The significance of the epoxy binder in raising the combustion temperature of the propellant is established. Epoxy binder and aluminium fuel are very significant to the composite propellant characteristic velocity. Also, the composite propellant that produces predominantly and enough combustion gases will definitely have a very low molecular weight. This is the desire of every propellant designer. Potassium nitrate, epoxy binder, aluminium and carbon are very significant in achieving this. Also, important in achieving this is the chemical linear interaction effect of both the potassium nitrate and the aluminium fuel, and the potassium nitrate and the carbon. Also significant is the quadratic effect of the epoxy binder. For a composite propellant with a good heat ratio, the main effects of the epoxy binder, the aluminium fuel, and carbon are statistically significant. The quadratic effects of the potassium nitrate and the linear interactions of the nitrate with both aluminium and the carbon are also significant.

Normal distribution demonstrated by the residual distribution curves indicates that normality assumptions for all the measured responses are satisfied. The normality assumptions are previously discussed in the literature [43]. However, the response surface of each of the analysed propellant

performance parameters is helpful in showing the shape of the optimum conditions. According to the response curve, it could be seen that the specific impulse, density, temperature, characteristic velocity, the molecular weight and heat ratio of the propellant increase by increasing the amount of the potassium nitrate while decreasing the amount of the epoxy binder. Also, indicated in the model equations are the interaction of the composite propellant constituents (main, linear and quadratic) and the effects on the propellant performance indices. The main effect of potassium nitrate on all the propellant performance parameters is positive. Meaning that an increase in the amount of the oxidiser gives better performance. Thus, less pollutant emission is released into the air, since the required oxygen for complete combustion is gotten from the oxidiser. The main effect of the epoxy binder on the performance parameters is negative. It is a hydrocarbon compound, thus produces a lot of carbon monoxide if not completely burnt. Therefore, the amount of the binder must be minimised. Aluminium fuel has a positive effect on propellant specific impulse, density, temperature, characteristic velocity and heat ratio. It produces more of the required heat for burning, thus ensures complete combustion. However, aluminium has a negative effect on the propellant's molecular weight. This is because the burning of aluminium is associated with the production of solid products or salt cake during burning (e.g. aluminium oxide, metallic aluminium and furans). This poses a great hindrance to effective burning of the composite propellant, thus a trade-off is required here. Also, the aluminium oxide product constitutes a serious threat to air quality in terms of particulate release. It is highly toxic and bits of aluminium oxide reflect the visible light from the sun back into space, which cools the Earth and can also absorb infrared radiation emanating from the planet's surface, essentially trapping heat the way a greenhouse gas does [44].

The presence of carbon black on the composite propellant performance parameters is generally negative. It also plays a negative role in the release of carbon monoxide into the air during combustion. Carbon monoxide is not only dangerous to human being but even though carbon monoxide is only a weak green house gas, its influence on the climate goes beyond its own direct effects. Its effects thus increase the concentrations of other greenhouse gases including methane, troposphere ozone and carbon monoxide [45]. Therefore, from a pollution control perspective, its amount must be minimised. Sulphur is added to the propellant to boost burning, therefore having positive effects on the propellant specific impulse, density and the molecular weight, but negative effects on temperature, characteristic velocity and heat ratio. Its negative effects on temperature can constitute to incomplete combustion of the propellant. Therefore, leading to the emission of sulphur dioxide that causes acid rain which damages buildings and kills plants. Exposure to sulphur dioxide in the ambient air is associated with reduced lung function, increased incidence of respiratory symptoms and diseases, irritation of the eyes, nose, and throat, and premature mortality. However, in spite of these disadvantages, effects of these propellant constituents on air quality are still less severe when compared with a more dangerous pollution from combustion of other solid propellants type.

4. Conclusion

The optimum compositions of the oxidiser, binder, fuel, opacifier, burning rate enhancer and catalysts in the formulated rocket composite propellant are 65.1%, 18.1%, 9.1%, 3.6%, 2.2% and 1.9%, respectively. The associated performance parameters are as stated as follows: specific impulse 122.03 s, the propellant density $1697.08 \text{ kg m}^{-3}$, propellant temperature 2025.07 K, the propellant characteristic velocity is 987.6 m s^{-1} , the molecular weight is $31.50 \text{ kg mol}^{-1}$ and heat ratio is 1.02. This study has also successfully investigated the relevance of specific impulse, density, burning temperature, characteristic velocity, molecular weight and heat ratio as major propellant performance characteristics. This propellant is made up of constituents which have less severe effects on the air quality, lives and properties of the people. It has also established that effort on air pollution control begins with a good performance and optimised rocket propellant formulation. RSM optimisation approach is a reliable and adequate method of standardising an environmentally friendly

rocket propellant formulation. It also has an adequate capability in optimising the effects of rocket propellant formulations on the propellant performance parameters.

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