Defence Science Journal, Vol. 65, No. 1, January 2015, pp. 31-38, DOI : 10.14429/dsj.65.7986

Propellant Grade Hydrazine in Mono/Bi-propellant Thrusters: Preparation and Performance Evaluation

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ABSTRACT

Propellant grade hydrazine was prepared with 64 per cent yield and 95.5 per cent purity. Purity of the propellant grade hydrazine was determined using wet chemical, gas chromatographic (GC) and eudiometric methods. It was observed that the compositions containing blends of hydrazine-methyl alcohol-ammonium nitrate and hydrazine-methyl alcohol-ammonium perchlorate were not found to be frozen even after cooling to -65 °C for 30 minutes. Mono and bi-propellant thrusters were designed and developed to demonstrate the performance of prepared propellant grade hydrazine as a promising rocket fuel**.** Five static tests with 22 N thruster and one static test with 1 N thruster were performed successfully in mono-propellant mode. The hurdles of chamber pressure oscillations were overcome by compact packing of the catalyst. The desired decomposition and chamber pressure were achieved. One static test was performed successfully with 60 N bi-propellant thruster. The desired chamber pressure and thrust were achieved. The combustion was smooth and C* achieved was higher than that of UH-25, N_2O_4 combination. The performance of prepared propellant grade hydrazine shows it as a promising rocket fuels.

Keywords: Propellant grade hydrazine, static test, mono-propellant, bi-propellant

1. INTRODUCTION

Hydrazine is used as fuel for thrusters, in spacecraft propulsion and attitude control systems. Hydrazine thrusters have been widely used in missile and satellite attitude control, velocity correction of spacecraft, and gas generation to provide auxiliary power in aviation because of simplicity and reliability (monopropellant); lowest cost propulsion system (other than cold gas); space storable for long periods (> 12 years demonstrated); low thrust capability and moderate thrust levels available (≤ 400 *N*)[1-4].

In the design of the hydrazine thruster assembly, the collective effects of the injector type, the amount of catalyst packed, and the shape of the catalytic reactor, were the primary concerns[5-6]. These thrusters have limited life because of voids which ultimately occur due to catalyst loss. The temperature of fuel and catalyst would crucially affect the ignition-delay and induction period of the thruster[7-9]. Another potential thruster failure mode is poisoning of the catalyst, which reduces the effective area for decomposition. The reduced activity of the catalyst results in a drop in chamber pressure and degradation of the thruster performance due to incomplete decomposition of hydrazine. The decomposition of hydrazine on Shell 405 can be described by the reaction[6,10-11]. Controlling of the degree of dissociation of NH_3 generated from N_2H_4 decomposition to achieve better characteristic velocities of the thruster, became the basic criterion in the design of injector and catalytic reactor[12]. Besides temperature, pressure would affect the rate of hydrazine decomposition as well[13-14]. The hydrazine thruster would acquire better start-up characteristics with small size catalyst, yet a larger pressure drop in the reactor[15]. In the operation of the hydrazine propulsion system, generally pressure fluctuation was observed. In cold start, a large pressure spike might be generated by excess hydrazine injection and accumulation of hydrazine in the induction period of the decomposition reactions[16]. Pressure oscillation might also be initiated by valve actuating disturbance and reaction instability.

The performance evaluation of propellant grade hydrazine was tested using self-designed 5 *N* and 22 *N* hydrazine propulsion systems at high altitude[6,17-18]. Low cost catalysts were tested in 1 *N*, 35 *N*, and 400 *N* hydrazine thrusters[19]. Schmitz describes the qualification test sequence for a long life monopropellant thruster for a satellite reaction control system[20]. The development of hydrazine monopropellant thrusters carried out at INPE and 400 *N* monopropellant engine were reported[21-23].

However, all these static tests were done with 98.5 per cent pure propellant grade hydrazine. Also with the monopropellant hydrazine thruster, the amount of iridium loading and the percentage of NH_3 dissociation aimed are not clearly known. The objective of this work is to demonstrate propellant grade hydrazine in monopropellant and bi-propellant modes with N_2O_4 as oxidizer and to evaluate thruster performance by studying

Received 12 September 2014, revised 23 December 2014, online published 27 February 2015

crucial parameters such as catalyst packing, percentage of NH_3 dissociation in monopropellant thruster, and controlling pressure oscillations. In addition, efforts were made to also envisage the feasibility of application of prepared propellant grade hydrazine by formulating few low-freezing hydrazine-based monopropellant blends.

In this work we have prepared propellant grade hydrazine (99.5 per cent) and determined its purity. We have designed and developed both monopropellant and bi-propellant thruster to demonstrate the performance of prepared propellant grade hydrazine as a promising rocket fuel**.** This paper also discusses about the predictive and static test performances of hydrazine thrusters in both monopropellant and bi-propellant modes.

2. EXPERIMENTAL

2.1 Materials

Hydrazine hydrate, 99 per cent LR grade was procured from M/s SD Fine Chemicals Ltd., Mumbai, and Sodium hydroxide, 99.5 per cent AR grade from M/s Finar chemical, Ahmedabad.

2.2 Preparation of Propellant Grade Hydrazine[24-26]

To 1000 ml round-bottomed flask, 500 ml commercially available 99 per cent pure hydrazine hydrate (64 per cent pure as hydrazine) and various quantities of *NaOH* (200-415 g) shown in Table 1 were added. The temperature of the water bath was maintained between 65 °C to 85 °C for 1 h to 2 h, a homogenous thick viscous white fluid was obtained. After the completion of the reaction, propellant grade hydrazine was distilled at 65 °C to 70 °C for 2 h under vacuum. The yield obtained varied between 16 per cent -64 per cent, respectively.

2.3 Wet Chemical Analysis

Percentage of hydrazine content was determined by non-aqueous (acetic acid) medium acid base and iodometric (KIO_3) titrations[1,27-28]. The percentage of water content was determined by eudiometric method using the standard procedures[29-30]

2.4 Gas Chromatography[31]

All the experiments were carried out in Thermo Quest Trace GC 2000 using packed column using thermal conductivity detector (TCD). Because of the higher polarity of hydrazine, a polar column, 25 per cent PEG on Anakrom (80-100 mesh) was selected and used for all the experiments.

2.5 Determination of Freezing Point at Sub-zero

Tests were conducted for freezing point with sub-zero analysis instrument by taking samples in small glass bottles caped and then sealed with teflon tape. An alcohol thermometer of range -70 °C to +200 °C was placed to check the temperature inside the chamber. The temperature was brought down to -40 °C. The frozen state of the samples were removed and observed. Samples which were not frozen were again placed in the chamber and the temperature was brought down to -65 °C.

2.6 Predictive Performance of Propellant Grade Hydrazine in Mono/Bi-propellant Modes

Predictive performance of propellant grade hydrazine in the monopropellant and bi-propellant modes was determined using hydrazine dissociation reactions and NASA CEA code[32], respectively. Heat of formation data calculated from heat of combustion value was used for the theoretical performance evaluation. Performance parameters were obtained with varying air-to-fuel (A/F) ratio.

2.7 Static Test Performance of Propellant Grade Hydrazine

2.7.1 Static Test Performance on Monopropellant Thruster

The stationary rocket combustor was operated at ambient temperature with 1 *N* and 22 *N* monopropellant thrusters. The fuel used was propellant grade hydrazine. The pressure measured in the combustion chamber was 7 bar, and at exit nozzle it was ambient. The density of fuel was 1.01 g/cc. The mass flow rate for 1 *N* was 0.46 g/s and for 22 N thrusters, it varies from 8.62 g/s to 10.5 g/s. The tests were performed with cumulative burn time of 63 s for 1 N thruster, and 23-63 s for 22 N thruster.

2.7.2 Static Test Performance on Bi-propellant Thruster

The stationary rocket combustor was operated at ambient temperature with N_2O_4 as an oxidizer. The fuel used was Propellant grade hydrazine, at a weight ratio of oxidizer-to-fuel 1.44. The pressure measured in the combustion chamber was 20 bar, and at exit nozzle it was ambient. The density of fuel was 1.01 g/cc. The oxidizer flow rate was 17.7 g/s and fuel flow rate was 12.3 g/s. The test was performed with cumulative burn time of 23 s.

3. RESULTS AND DISCUSSIONS

3.1 Preparation of Propellant Grade Hydrazine

Anhydrous hydrazine, used as rocket propellant is obtained by dehydrating hydrazine hydrate. Hydrazine forms an azeotrope with water and ordinary distillation provides hydrazine in the form of hydrate (64 wt per cent of hydrazine). The use of hydrazine in rocket propulsion requires hydrazine to be in the anhydrous form, and hence, the removal of water from hydrate state to produce anhydrous hydrazine is essential to make it suitable for such purposes. The conventional separation techniques for the removal of water experience difficulty as hydrazine forms an azeotrope with water at 71.5 wt per cent

Table 1. Reaction parameters for preparing monopropellant grade hydrazine

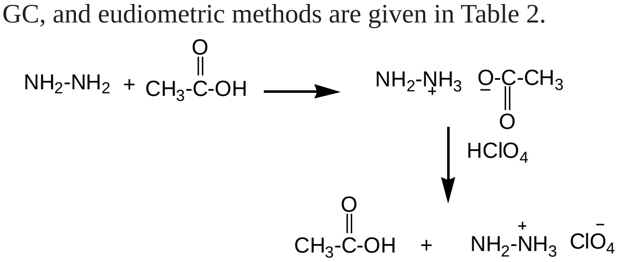
Sample code	Hydrazine hydrate (ml)	Wt of *NaOH* (wt)	Reflux temp (°C)	Distillation temp (°C)	Yields (%)
S1	500	415	85	65 - 70	16%
S2	500	400	70-75	60-65	30%
S3	500	200	75-80	65-70	64%

of hydrazine. Further, hydrazine and water are highly polar by nature, and there is a strong hydrogen bonding between these. Hence, the combinations of processes are required to seek dehydration[24-26].

Three experiments were carried with varying ratios of *NaOH* at different reaction conditions for preparation of propellant grade hydrazine using 99 per cent pure hydrazine hydrate (64 per cent pure as hydrazine). The quantity of *NaOH* was varied from 200 g to 415 g and time duration was kept between 1 h - 2 h. The reflux temperature was varied between 65 °C to 85 °C. The products were distilled by maintaining the temperature between 60 °C-70 °C. The yields obtained for the three experiments were 16 per cent, 30 per cent, and 64 per cent, respectively. The reaction parameters maintained for these three experiments are given in Table 1.

3.2 Determination of Purity of Hydrazine

The purity of hydrazine was determined using wet chemical method which is based on non-aqueous acid–base titration in acetic acid media and gas chromatographic (GC) method. The total basicity due to hydrazine is calculated using the reaction scheme shown in Eqn (1) and wt per cent of hydrazine will be obtained. From the GC experiments, water content is varied from 10-14 per cent depending on the distillate (S-1, S-2 and S-3) and the method of distillation. However S-4 obtained by distillation of S-3 yielded 99.46 per cent pure hydrazine and 0.54per cent water. No other organic impurities were found in sample. The chromatograms of samples S-3 and S-4 are given in Fig. 1. Water content in the hydrazine determined by the eudiometric method is based on the reaction of water and calcium hydride. Analysis results obtained from wet chemical, GC, and eudiometric methods are given in Table 2.



(1)

3.3 Low Freezing Hydrazine-based Monopropellant Blends

A tactical monopropellant should have a wide operating temperature ranging from - 60 °C to + 60 °C. Addition of water to hydrazine lowers the freezing point to forms an azeotropic mixture having boiling point of 120.3 °C and freezing point of - 40 °C. A monopropellant fuel mixture with hydrazine, water and hydrazine nitrate ($N_2H_4\,HNO_3$) had disadvantage of having large percentage of water.

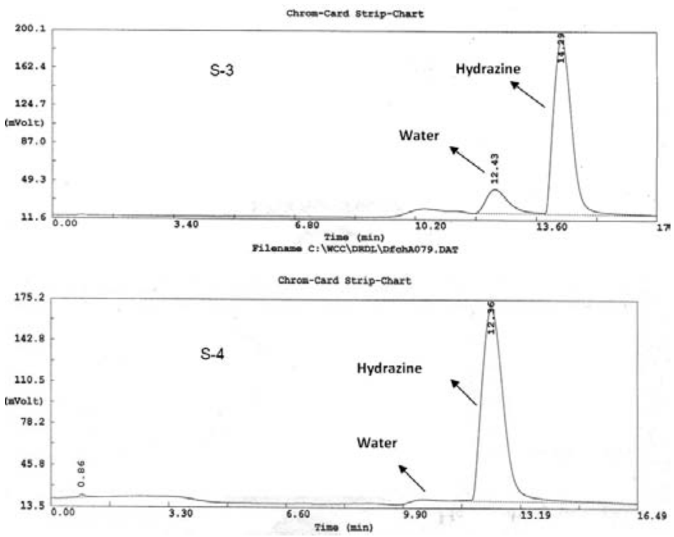


Figure 1. Gas chromatogram of samples S-3 and S-4.

The fuel blend combination chosen for present study are anhydrous hydrazine, methyl alcohol, ammonium nitrate or ammonium perchlorate. Methyl alcohol is miscible with hydrazine and reduces the freezing point of the blend. Methyl alcohol has a carbon content of 37.48 per cent, a hydrogen content of 12.58 per cent and an oxygen content of 49.37 per cent . The substitution of methyl alcohol with water increases the energy value available and makes the blend combination superior. Both ammonium nitrate and ammonium perchlorate have oxidizer as well as fuel part in these; there are the salts obtained from ammonia. This supports the combustion process and produce low molecular weight products during decomposition of hydrazine. Five blends are prepared with varying composition of additives mentioned above by adding propellant grade hydrazine to decrease the freezing point. Tests carried out for analysis of freezing point at sub-zero were shown in the Table 3. It was observed that the composition containing blend 5, i.e., hydrazine and methyl alcohol blend, has frozen. Blend samples 1, 2, and 4 were not frozen at -65 °C even after 30 min whereas blend sample 3 was frozen after 30 min.

3.4 Performance Evaluation of Propellant Grade Hydrazine in Monopropellant Mode

3.4.1 Predictive Performance in Monopropellant Mode

For maximum specific impulse (I_{sp}), it was considered to limit the catalyst length to 26 mm for 40 per cent to 50 per cent ammonia dissociation. All calculations are done for steady-state

Table 2. Analysis results of distillate samples

Sample code	% Yield	Hydrazine, % wt		Water, %wt	
		Wet chemical Method	GC Method	Eudiometric Method	GC Method
S-1	15	88.96	89.70	10.92	10.30
S-2	30	87.52	88.59	12.30	11.41
S-3	64	85.21	85.07	14.51	14.93
S-4	56	98.82	99.50	0.95	0.54

Table 3. Blend formulations for low freezing applications

Ingredient	Blend				
	1	2	3	4	5
Anhydrous hydrazine, wt. %	65	58	65	58	85
Methyl alcohol, wt. %	15	17	15	17	15
Ammonium nitrate, wt. %	20	25	--	--	--
Ammonium perchlorate, wt. %	--	--	20	25	--
Test temperature: -65ºC, for 30minutes	Not Frozen	Not Frozen	Frozen	Not Frozen	Frozen

firing condition of the thruster. Hydrazine purity was considered as 95 per cent, 99 per cent, and 100 per cent, respectively. Various performance parameters such as characteristic velocity (C*), specific impulse (I_{sp}) were determined assuming frozen flow conditions at 7 bar chamber pressure. The parameters were evaluated in chamber, throat and nozzle exit. Supersonic area ratio (A_e/A_t) was assumed to be 2.75. The characteristic velocity with ammonia dissociation ratio for 95 per cent , 99 per cent and 100 per cent of pure hydrazine was observed as 1284 m/s, 1275 m/s and 1260 m/s, respectively was shown in Fig. 2. The thruster-designed parameters are shown in Table 4.

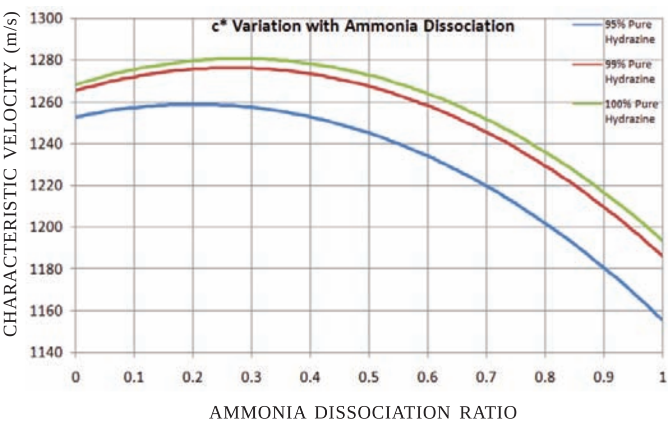


Figure 2. Characteristic velocity variation with ammonia dissociation.

Table 4. Monopropellant thruster-designed performance parameters for high altitude application

Parameter	Thruster
Chamber pressure (bar)	7
Area ratio	50
Exit pressure (bar)	0.023
Thrust coefficient	1.83
C* (m/s)	1240
Specific Impulse (eff 94%) (s)	220

3.4.2 Static Test Evaluation in Monopropellant Mode

The performance of the prepared propellant grade hydrazine (99.5per cent) as rocket propellant in monopropellant mode was investigated by operating stationary rocket motors. Five static tests were conducted with 22 N monopropellant thruster and one test with the 1 N monopropellant thruster in pulse mode at ambient temperature which were designed for sea-level conditions for static testing, and the performance parameters were compared with the designed values.

Table 5 summarized the thruster parameters employed in the tests for monopropellant mode. The monopropellant thrusters Both 1 N and 22 N are made of SS-321 alloy. The shell has a thickness of 2 mm and a factor of safety above 5 for the operating pressure. The injector is a shower head in which the injection tube has a length of 30 mm and is brazed to the injector head. Three injection tubes of 0.58 mm ID are used for 22 N thruster whereas a single tube of 0.19 mm ID is used for 1 N case. The injector and the catalyst chamber are joined via a flange joint using an asbestos gasket for sealing. The pressures are measured upstream of the fuel inlet valve in the fuel tank and at the catalyst chamber exit (entry to the CD nozzle) using Sensotech make strain gauge type transducers.

Table 5. Thruster parameters in monopropellant mode

Parameter	1 N	22 N
Chamber pressure	7 bar	7 bar
Mass flow rate	0.47 g/s	10.2 g/s
Injector pressure drop	2 bar	2 bar
Injector dimensions	1 tube of 0.19 mm dia	3 tubes of 0.5 mm dia
Particle size of catalyst	600-800 microns	600 micron granules and 3mm pellets
Catalyst bed length (effective)	26 mm	10 mm + 16 mm
Catalyst bed diameter	12 mm	28 mm
Throat diameter	1.0 mm	4.4 mm
Area ratio (sea level test)	2.75	3.0

The motor was operated at a combustion chamber pressure of 7 bar and expanded to ambient pressure of 0.94 bar. Figure 3 represents 22 N thruster with injector. Table 6 represents the test results of granules of iridium-coated catalyst used with alumina. Table 7 provides the details about the amount of catalyst packed for all the six tests. Pulse train was given manually and different pulse widths used for these tests are given in Table 8. Chamber pressure was measured at nozzle entry. The chamber skin temperature reached a maximum of 520 °C with an injector head temperatures less than 160 °C. Chamber pressure oscillations were observed in first test, which was eliminated by proper packing of the catalyst. Table 9 compares the achieved parameters with the designed parameters for all the tests.

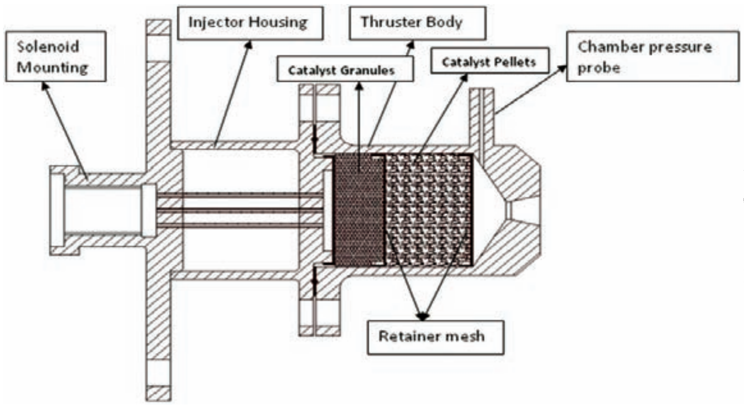


Figure 3. Assembly drawing for 22N thrusters.

Table 6. Test results of iridium-coated with alumina granules

Nomenclature	Results
Particle size, (µ)	702
Iridium content by weight, (%)	46
Crush strength, (N)	62.46
Surface area, (m^3/g)	90

Table 7. Catalyst packed in all the tests

Test	Pellets (g) (3 mm)	Granules (g) (F 600 – 800 µ)
Test – 1	19.76	7.41
Test – 2	19.66	9.36
Test – 3	19.50	10.04
Test – 4	18.88	11.61
Test – 5	18.88	11.61
1N	-	6.14

*Tests 1-5: Thruster capacity-22N; Test 6: Thruster capacity-1N

Figure 4 represents variation of absolute chamber pressure with time for Test 2. A steady chamber pressure was observed over the 5 s pulse of 7.16 bar. The tank pressure also remained near-constant and the response time of around 90 ms was observed. Figure 5 represents variation of oxidizer manifold pressure with time for Test 6. Steady-state firing was achieved and the tank pressure was steady throughout the test. Response time was around 100 ms. Figure 6 shows variation of nozzle, chamber and injector temperatures with time for Test 6. The maximum skin temperature was observed as 470 °C.

3.5 Performance Evaluation of Propellant Grade Hydrazine in Bi-propellant Mode

3.5.1 Predictive Performance Evaluation in Bi-propellant Mode

Hydrazine purity was considered as 99.5 per cent. Various performance parameters were determined assuming frozen flow conditions at 20 bar chamber pressure. The parameters were evaluated in chamber, throat and nozzle exit. Supersonic area ratio (A_e/A_t) was taken as 12.5. N_2O_4 was used an oxidizer for the bi-propellant thrusters. The oxidizer and fuel flow rates are 17.5 g/s and 12.5 g/s, respectively. The thruster-designed parameters are provided in Table 8.

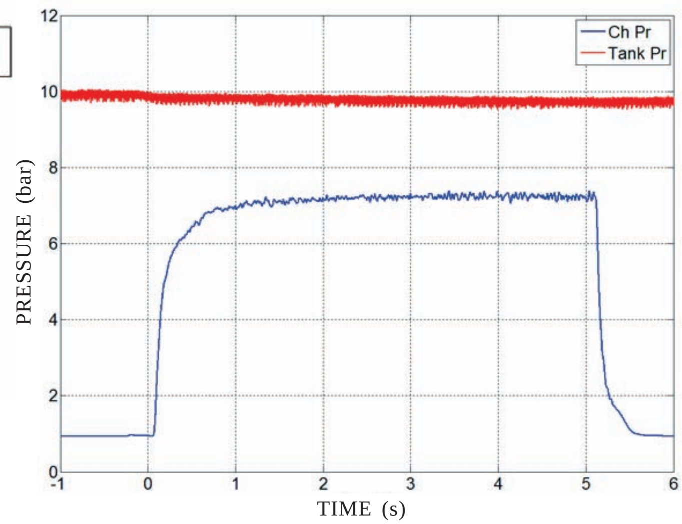


Figure 4. Variation of absolute chamber pressure with time for Test 2 (22N).

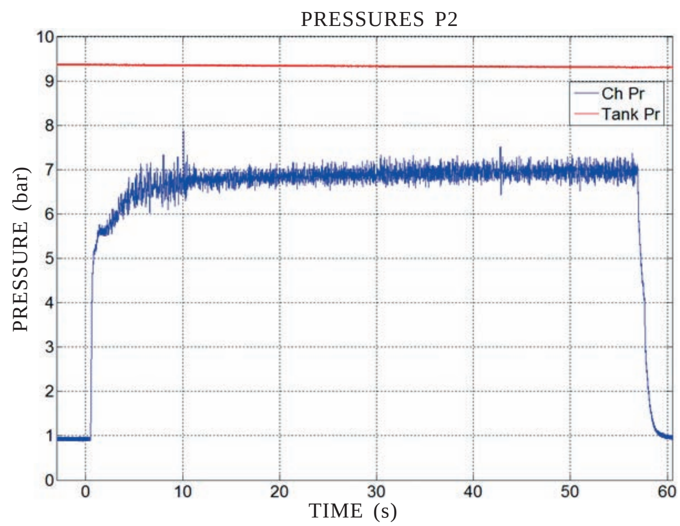


Figure 5. Variation of oxidizer manifold pressure with time for Test 6 (1N).

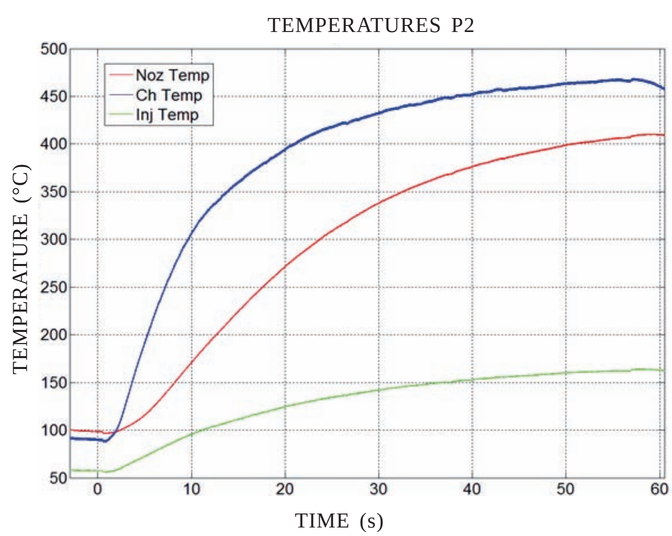


Figure 6. Variation of nozzle, chamber and injector temperatures with time for Test 6 (1N).

Table 8. Designed and achieved parameters for monopropellant test

Parameter	Designed	Test-1	Test-2	Test-3	Test-4	Test-5	Test-6(1N)
Ch. Pr. (bar)	7.0	7.45	7.16	5.93	7.45	7.21	6.88
Mass flow rate (gm/s)	10.5	9.34	9.68	8.62	10.12	9.40	0.463(0.46)
C* (m/s)	1205	1204*		1067^	1109^	1171^	1166^
No. of pulses	---	3	2	4	4	2	2
Pulse widths (s)	---	5,4,14	5,25	2,3,3,55	4,10,11,23	2,61	7,56
Cum. burn time (s)	---	23	30	63	48	63	63

* Propellant batch 1 : 95% Purity , ^ Propellant batch 2 : 99% Purity

Table 9. Design parameters of bi-propellant thruster

Parameter	Design Value
Propellant densities at 20 °C	Oxidizer- 1440 kg/m^3 Fuel - 1010 kg/m^3
Chamber pressure	20 bar
O/F (mass)	1.44
Oxidiser injector diameter	0.64 mm X 2
Fuel injector diameter	0.83 mm
Exit area ratio	12.5
Exit diameter	20 mm
Chamber temperature	3138 K
Exit pressure	0.2 bar
Thrust coefficient (S.L)	1.15
Characteristic velocity (actual)	1707m/s
Specific impulse (S.L)	200 s
Thrust (Design)	9 Kgf / 88.2N
Thrust (SL)	60 N

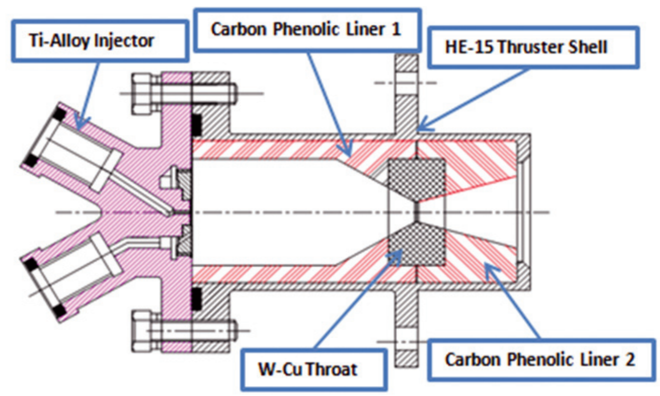


Figure 7. Bipropellant thruster cross-section.

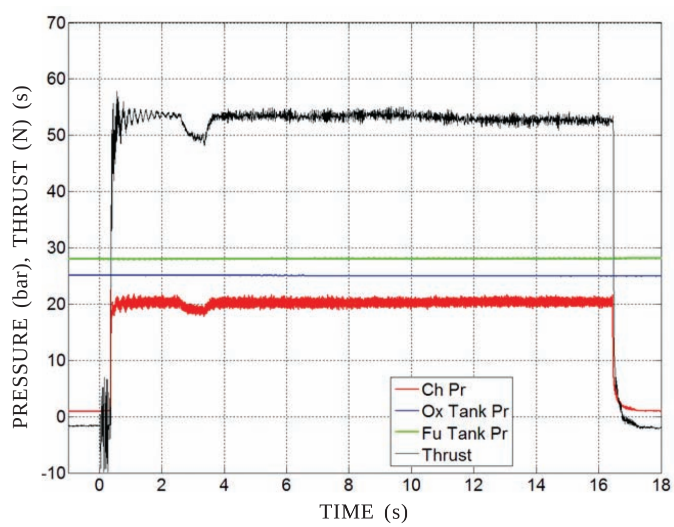


Figure 8. Variation of absolute chamber pressure, oxidizer manifold pressure, fuel manifold pressure, and thrust with time.

3.5.2 *Static Test Evaluation in Bi-propellant Mode*

The performance of the prepared propellant grade hydrazine in bi-propellant mode was investigated by conducting static test using carbon phenolic lined 60N bi-propellant thruster. The ratio of the cross-sectional area of the nozzle exit to-throat cross-sectional area (area ratio) was 12.5. Figure 7 shows schematic diagram of thrusters cross-section used for the test. The bi-propellant thruster is made of HE-15 shell which is internally lined with carbon-phenolic liner for thermal management. The injector is made of *Ti-6Al-4V* alloy with a triplet injector in O-F-O configuration. The pressures are measured at the upstream locations of both fuel and oxidiser inlet valves and their tanks. The chamber pressure port is located on the injector head. The pressure transducers used are similar to the ones used for mono-propellant thruster testing.

The motor was operated at a combustion chamber pressure of 20 bar and expanded to ambient pressure of 0.94 bar. One static test was performed at ambient temperature. Figure 8 represents variation of absolute chamber pressure, oxidizer manifold pressure, fuel manifold pressure and thrust with time. A single pulse was given for 16 s and a steady chamber pressure of approximately 20 bar and thrust of 56 N was achieved. Combustion instability is not observed. Table 10 represents the performance parameters summary of the static test.

4. CONCLUSIONS

(i) Propellant grade hydrazine was prepared in 64 per cent yield with 99.5 per cent purity and determined using wet chemical, GC and eudiometric methods.

(ii) It was observed that the compositions containing blends of hydrazine-methyl alcohol-ammonium nitrate and

Table 10. Designed and achieved parameters for bi-propellant test

	Design	Test
Chamber pressure (bar A)	20	20.3
O/F (mass)	1.44	1.49
Oxidiser flow rate (g/s)	17.7	17.5
Fuel flow rate (g/s)	12.3	11.76
Thrust coefficient (S.L)	1.19	1.14
Characteristic velocity (m/s)	1740.4	1782
Specific impulse (S.L) (s)	211	207
Thrust (S.L) (N)	60 ± 6	56

hydrazine-methyl alcohol-ammonium perchlorate were not found to be frozen even after cooling to -65°C for 30 minutes.

(iii) Five static tests with 22N thruster and one static test with 1N thruster were performed successfully in monopropellant mode. The percentage of dissociation aimed for NH_3 is taken as 40 per cent to 50 per cent in the monopropellant thruster. The energetic performance of the thruster matched very closely to the predictive performance with iridium catalyst. Long duration pulse up to 60 s was successfully tested. The hurdles of chamber pressure oscillations were overcome by compact packing of the catalyst. The desired decomposition and chamber pressure were achieved.

(iv) One static test was performed successfully with 60 N bi-propellant thruster. The desired chamber pressure and thrust were achieved. The combustion was smooth and C* achieved was higher than that of UH-25, N_2O_4 combination.

Both monopropellant and bi-propellant thrusters were designed, fabricated, and validated to demonstrate its capability to meet the demanding mission requirements in reaction control thrusters. The concept of mono/bi-propellant-based thrusters using hydrazine fuel was established. The performance of prepared propellant grade hydrazine shows these as promising rocket fuels.

ACKNOWLEDGEMENT

The authors wish to thank Director, DRDL and Director, Propulsion for supporting this study.

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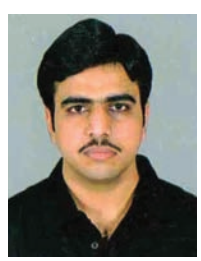

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