Performance Assessment of Fuels TMEDA-DMAZ and Tonka250 with Liquid Oxidizer AK27

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ABSTRACT: *Tonka250 is a liquid fuel containing equal percentages of triethylamine and xylidine which has attracted the attention of space industries due to its favorable performance characteristics. Tonka250 has a low Ignition Delay (ID) time in the presence of AK27 as a liquid oxidizer (about 24 ms) and can be used as a starter fuel in engines working with a non-hypergolic combination of fuels and oxidizers. In recent years, due to the carcinogenic effect of Tonka250, novel fuels have been introduced to replace it with high-performance properties and non-carcinogenic effects. A combination of tetramethyl ethylene diamine (TMEDA) and dimethyl aminoethyl azide (DMAZ) can be a candidate for this purpose. According to the results obtained from the current research, the equal weight percentage of TMEDA and DMAZ has an ID time of 14 ms in reaction with AK27. The ID time was measured through the cup test method with a fuel droplet in the liquid oxidizer. The density and density specific impulse of fuel TMEDA-DMAZ are close to those for Tonka250 while the values of specific impulse for fuel TMEDA-DMAZ are about 6 seconds more favorable than Tonka250 at the chamber and exit pressures 6.8 MPa and 0.1 MPa, respectively. Therefore, it seems that fuel TMEDA-DMAZ is a good candidate for replacement for Tonka250.*

KEYWORDS: *TMEDA-DMAZ; Tonka250; AK27; Ignition delay time; Specific impulse.*

INTRODUCTION

Liquid bipropellants have been used in space programs since 1950. Space rockets may contain two or three stages. In the first stage of these rockets, petroleum cut like kerosene is combusted in a combustion chamber in the presence of a liquid oxidizer such as nitric acid or nitrogen tetroxide. Although there are various fuel candidates for use in the first stage, kerosene is consumed extensively due to its low price. However, the combustion of kerosene with the liquid oxidizer needs an igniter because its ignition is not instantaneous. Several types of igniters have been proposed for this purpose. Among them, the safest way is using a hypergolic bipropellant. In the hypergolic bipropellants, the fuel and the oxidizer contact each other and produce flame instantaneously. The produced flame is the source of heat for the combustion of kerosene and the oxidizer. Various hypergolic bipropellants have been suggested. Among them, Tonka250 is low price and used extensively [1-6]. The criterion for self-ignition or instantaneous combustion of the bipropellants is ignition delay time or ID time. The ID time is the time

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between contact of liquid fuel and liquid oxidizer to appear the first flame in the combustion chamber of a rocket. High ID time leads to the accumulation of unburned propellant in the combustion chamber. If the propellant combusts, sudden pressure causes an explosion in the chamber and destroys it. Very short ID time causes melting of the injectors. Therefore, the amount of ID time in space programs should be less than 50 ms for hypergolic propellants and preferably less than 16 ms [7-10].

Liquid fuel Tonka250, a mixture of triethylamine and xylidine with an equal weight ratio, has usually been consumed as starter fuel in space rockets and also as rocket fuel in ballistic rockets in several Eastern Bloc countries [11,12]. The ignition delay time or ID time of the fuel with liquid oxidizer AK27 is less than 30 ms [13]. Short ID time of Tonka250 causes to use of this fuel as a starter in non-hypergolic propellants (like kerosen+AK27 in the first stage of space rockets) [14]. On the other side, AK27 is a liquid oxidizer containing nitric acid (73 wt%) and nitrogen tetroxide (27wt%) [15]. Although the mentioned fuel in reaction with AK27 has high-performance characteristics, due to its carcinogenic effect [14], scientists and engineers in space industries pursue alternative green or noncarcinogenic fuels for replacement.

Among the proposed green fuels, tertiary amine azides, and tertiary diamines seem to be good candidates for this purpose [16-18]. Based on the published reports, among the tertiary amine azides, dimethyl aminoethyl azide (DMAZ) is a good option. It has excellent thermo-physical and performance properties [19-21]. One of the main challenges for the direct application of DMAZ in liquid fuel rocket engines is its slightly high ID time in reaction with nitric acid-based oxidizers [22]. On the other hand, the researchers confirmed that the application of DMAZ together with chemical compounds such as tertiary di-amine, tertiary tri-amine, or tetra-amine family may lead to a favorable synergy effect in ID time [23-25]. A famous member of the tertiary di-amines family is tetra-methyl ethylene diamine (TMEDA).

Initial studies in recent years showed that the combination of DMAZ and TMEDA has acceptable physicochemical properties along with low toxicity issues [23-25]. Therefore, finding the appropriate formulation for DMAZ-TMEDA binary fuel can help to introduce a novel fuel with high possible performance and low toxicity as a green replacement for traditional fuels. Since there is no published data about the binary mixtures TMEDA-DMAZ in reaction with oxidizer AK27, obtaining a proper composition for the mixture and its assessment in comparison with Tonka250 is the aim of this research.

Since the existence of start fuel and sustain or flight fuel in a space rocket complicates the rocket design, such a combination can remove using of Tonka250 as a carcinogenic starter fuel. In other words, this combined fuel may be used as a starter and sustainer or flight fuel. Finally, this combined fuel may simplify the design, manufacturing, and control of a space rocket.

EXPERIMENTAL SECTION

Materials

DMAZ (purity of 99.66 wt%) and TMEDA (purity of 99.43 wt%) were purchased from 3M Co. (USA) and Merck Co (Germany), respectively.

Triethylamine (purity of 99.9 wt%) and xylidine (purity of 99.8 wt%) were supplied from Merck Co (Germany). Both chemicals were mixed in equal weight ratio (50:50).

Nitric acid (≥99.5 wt% purity, from Merck Co., Germany) and N_2O_4 (purity of 99.5 wt%, from Kaida Technology Co., UK) were purchased. AK27 was prepared from the dissolution of about 27wt% of N_2O_4 in nitric acid.

Evaluation methods

As mentioned, Ignition Delay (ID) time and specific impulse (I_{sp}) are two important parameters for the evaluation of the performance properties of a bipropellant. Both properties are related directly or indirectly to combustion phenomena. In the following, the methods used for evaluating the ID time and specific impulse will be given.

ID time measurement

A proper method for measuring the ID time is a drop test or open cup test [22]. It is an exact and relatively easy method. About 100×10^{-9} m^3 liquid oxidizer is poured into a glass vial (inner diameter is 0.0127 m). The glass vial is then placed in a bottom cylindrical chamber with an inner diameter of 0.05 m and a length of 0.13 m. A syringe dispenses the fuel, and it is placed in the glass vial and points to the center of the oxidizer pool. The syringe is kept in the top cylindrical chamber, and the drop is released by the motion from the piston of a pneumatic actuator at a height of 0.05 m above the pool center. The chamber is first purged by N_2. In each test, one droplet (about 7×10^{-9} m^3) fell onto the pool.

As it was stated, the ID time is defined as the time delay between the contact of two liquids and the occurrence of luminosity. The ignition delay time was measured by using a high-speed camera (1,000 frames per second, model CASIO EXLIM FX-X1 [CASIO Co., Tokyo, Japan]). Therefore, the temporal resolution of ignition delay measurement is 1.0 ms. The ID time test for each propellant was performed five times and an average value was reported.

Specific impulse (I_{sp}) assessment

Specific impulse (I_{sp}) is an important parameter in rocket design. It is defined as the thrust delivered per unit flow weight of propellants consumed [26]. The initial assessment of specific impulses was performed *via* combustion software. Rocket Propulsion Analysis (RPA) software was applied for the evaluation of I_{sp} of binary fuel DMAZ-TMEDA with liquid oxidizer AK27. RPA software is a multi-platform software for prediction of rocket engine performance. This software uses a chemical species library and is upgraded by NASA Glenn thermodynamic database and Gurvich thermodynamic database which contains data for different fuels and oxidizers. The software works on the basis of Gibbs energy minimization. For equilibrium combustion calculations, the chamber and exit pressures were assumed 6.8 MPa and 0.1 MPa, respectively. Moreover, the gas flow was assumed entropic and one-dimensional.

RESULTS AND DISCUSSION

ID time analysis

The ID time is an important parameter indicating the hypergolicity of a bipropellant. Unfortunately, theoretical estimation of the ID time is very difficult because ignition has a very complex nature [27-33]. Therefore, it is measured experimentally. The ID time of the propellants in space programs is about 0.5 to 100 ms. To prevent the hard start of the engine (accumulation of fuel in the chamber before combustion initiation and therefore the sudden increase of pressure during combustion start), the ID time should be less than 50 ms for hypergolic propellants and preferably less than 16 ms [7].

The different compositions of DMAZ in TMEDA were prepared experimentally and tested with liquid oxidizer AK27 for measuring the ID time through the open cup test. The results are given in Table 1. The ID time of each propellant was measured five times at 25 ^oC and atmospheric pressure. The average results are given in Table 1.

As it is presented in Table 1, pure TMEDA has a lower ID time concerning pure DMAZ. Also, adding a low amount of TMEDA to DMAZ leads to an erratic ignition. More addition of TMEDA in DMAZ decreases the ID time. Finally, the composition of 90%TMEDA+10%DMAZ has the minimum ID time. After that, the ID time increases slightly.

Although the reactions that occur in the ignition delay phenomenon are complex, however, when a liquid fuel droplet falls into a pool of liquid oxidizer, there may be three main mechanisms of reaction between them [22]:

a) Neutralization in which the reaction between fuel and oxidizer occurs. In other words, an acid-base neutralization reaction occurs. This reaction generally is very exothermic. The heat released leads to evaporating the unreacted species and products.

b) Nitration where fuel is nitrated via a nitration agent.

c) Oxidation in which the evaporated species in the gas phase are oxidized. The heat released in the neutralization reaction leads to more oxygen radical generation in the gas phase, and thereby oxidation reaction occurs faster. More oxygen radical generation causes a faster oxidation reaction.

Generally, the main mechanisms in the ignition process are neutralization and oxidation, and minor nitration.

N_2O_4 and HNO_3 in liquid oxidizer AK27 are potential sources of oxygen radical generation. It is considerable that N_2O_4 is easily converted to NO_2 by heating [34]. Also, HNO_3 has an acidic group that participates in the neutralization reaction with $-N(CH_3)_2$ group in TMEDA and DMAZ. As described before, heat generation in the neutralization reaction in the liquid phase leads to the release of more oxygen radicals in the gas phase. Thus, HNO_3 is mainly a neutralizing agent and N_2O_4 is an oxidizing agent in ignition reactions.

As it was mentioned, TMEDA-HNO_3 has a lower ID time than DMAZ-HNO_3. One explanation may be that TMEDA has two $-N(CH_3)_2$ groups whereas DMAZ has one. Thus, TMEDA is more basic than DMAZ and therefore is more reactive in neutralization reaction.

Table 1: The results of ID time tests for different compositions of TMEDA-DMAZ in reaction with liquid oxidizer AK27.

Composition	ID time (ms)
Pure DMAZ	185.9
10%TMEDA+90%DMAZ	Non hypergol
20%TMEDA+80%DMAZ	121.00
30%TMEDA+70%DMAZ	120.00
40%TMEDA+60%DMAZ	69.33
50%TMEDA+50%DMAZ	14.13
60%TMEDA+40%DMAZ	9.86
65%TMEDA+35%DMAZ	9.86
70%TMEDA+30%DMAZ	8.80
80%TMEDA+20%DMAZ	8.00
90%TMEDA+10%DMAZ	7.60
Pure TMEDA	7.88

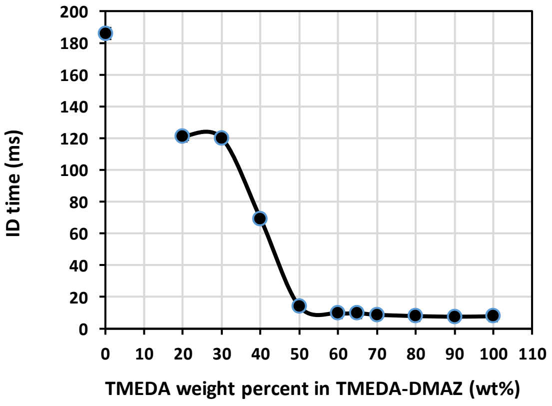

Fig.1: Effect of TMEDA weight percent in TMEDA-DMAZ on ID time in reaction with oxidizer AK27

In other words, the neutralization rate in TMEDA-HNO_3 is more than DMAZ-HNO_3 and consequently, the combination of TMEDA-HNO_3 generates oxygen radicals more rapidly. *Liu et al.* also reported such an observation [35]. They concluded that the heat generation in TMEDA-HNO_3 is more than DMAZ-HNO_3. The heat generated in the neutralization reaction increases the system temperature and evaporation and/or decomposition of the species present in the reaction medium and accelerates the other reaction steps. They stated that the energy barrier for hydrogen abstraction of DMAZ by nitrogen dioxide resulting from HNO_3 decomposition is a reason for the longer ignition delay time of DMAZ-HNO_3 than TMEDA-HNO_3 [18]. Also, *Zhang et al.* [36] explained that there are two amine groups with equal energy in TMEDA whereas in DMAZ, an azide group with less energy than the amine group has been substituted. Moreover, the amine group in DMAZ has lower energy than the amine group in TMEDA. Therefore, DMAZ releases lower energy than TMEDA in reaction with HNO_3. This lower energy is a reason for the next slower reaction in the gas phase.

As it was mentioned, it is expected that TMEDA in the presence of HNO_3 in oxidizer AK27 can supply the required heat for conducting the reactions in the gas phase. The amount of TMEDA in DMAZ should not be 20 wt% which is shown in Fig.1.

The gas-phase reactions between DMAZ and NO_2 are another important step in ignition that contains the initial abstraction of hydrogen from DMAZ. The energy barrier for the abstraction of hydrogen via NO_2 (resulting from HNO_3) is another reason for the longer ID time of DMAZ-HNO_3 than TMEDA-HNO_3. Moreover, DMAZ+3NO_2 causes the reaction path leading to N_2 formation which is more exothermic than TMEDA+3NO_2 [36]. Thus, the reaction of TMEDA with NO_2 (or N_2O_4) releases lower heat so that TMEDA-N_2O_4 system is non-hypergolic whereas the ID time for DMAZ-N_2O_4 is 68 ms [37]. Therefore, adding TMEDA to DMAZ in the presence of N_2O_4 (in oxidizer) leads to adverse effects at ambient temperature and reduces the heat released from initial reactions. Consequently, this event leads to non hypergolic or erratic behavior of DMAZ with oxidizer AK27 at low amounts of TMEDA addition.

If the amount of TMEDA is high enough to react with nitric acid and generates enough heat to continue the reaction of TMEDA-DMAZ with NO_2 in the gaseous phase, then the ID time will be reduced. This phenomenon indicates that DMAZ will be a good hypergolic fuel in reaction with HNO_3 if DMAZ-NO_2 reaction is initiated via a suitable amount of heat due to the reaction of TMEDA-DMAZ with HNO_3. In other words, the main problem of DMAZ in reaction with oxidizer AK27 is the low heat of the initial reaction in the liquid phase with liquid HNO_3. This problem can be solved by adding enough TMEDA to DMAZ. As it can be seen in Fig.1, 50 wt% TMEDA in DMAZ can provide enough initial heat for the reactions and decrease the ID time to values lower than 20 ms.

Although it was tried the ambient temperature to be constant, depending on the temperature fluctuations during the ID time measurement (about ±0.5 °C), there are uncertainties in the ID time measurements. This is also right for the preparation of the binary mixture. It is due to measurement accuracy of materials' mass or volume. Thus, the uncertainty for Table 1 is given in Table 2.

The average uncertainty is ±1 ms. Therefore, it seems that this average uncertainty is good for ID time measurement.

I_{sp} analysis

The ID time for liquid fuel Tonka250 and oxidizer AK27 was measured as 24 ms through the open cup test method. On the other hand, to have low ID time for TMEDA-DMAZ with AK27, the weight percent of TMEDA in the binary mixture should be more than 50 wt%.

Fig. 2 shows the result of the I_{sp} versus O/F ratio at sea level for different compositions of TMEDA-DMAZ and Tonka250 with liquid oxidizer AK27. Kerosene, as a conventional fuel in the first stage of a rocket is only given for comparison. The pressure in the combustion chamber was considered as 6.8 MPa and the exit pressure was 0.1 MPa or atmospheric pressure. This is a standard condition for rocket engine tests [26].

As mentioned previously, for I_{sp} analysis, the weight percent of TMEDA in the binary mixture was more than 50 wt% in all I_{sp} analyses.

As it is shown in Fig.2, all compositions of the binary fuel have more I_{sp} than Tonka250. The maximum I_{sp} for TMEDA-DMAZ with AK27 was derived in equal weight percentages of TMEDA and DMAZ or 50:50. The maximum I_{sp} was obtained at O/F ratio of 3.2. With increasing the TMEDA in the binary mixture, the optimum O/F ratios were shifted to the higher values, and the values of I_{sp} were decreased slightly. The maximum I_{sp} was obtained at an O/F ratio of 4.2 for Tonka250 with oxidizer AK27.

The effect of TMEDA content in the binary mixture on I_{sp} at different O/F ratios with AK27 has been demonstrated in Fig.3. An increase in optimum O/F ratio tends to pure TMEDA and a decrease in O/F ratio tends to pure DMAZ. As it is shown, pure DMAZ at O/F ratio of 2.6 has the maximum I_{sp} but as it was mentioned earlier, the ID time for DMAZ-AK27 is more than 50 ms which is not suitable.

Table 2: The uncertainty for the ID time measurements for different compositions of TMEDA-DMAZ in reaction with liquid oxidizer AK27.

Composition	ID time (ms)
Pure DMAZ	185.9±1.7
10%TMEDA+90%DMAZ	Non hypergol
20%TMEDA+80%DMAZ	121.00±1.7
30%TMEDA+70%DMAZ	120.00±1.7
40%TMEDA+60%DMAZ	69.33±1.5
50%TMEDA+50%DMAZ	14.13±1.1
60%TMEDA+40%DMAZ	9.86±0.9
65%TMEDA+35%DMAZ	9.86±0.9
70%TMEDA+30%DMAZ	8.80±0.7
80%TMEDA+20%DMAZ	8.00±0.3
90%TMEDA+10%DMAZ	7.60±0.3
Pure TMEDA	7.88±0.3

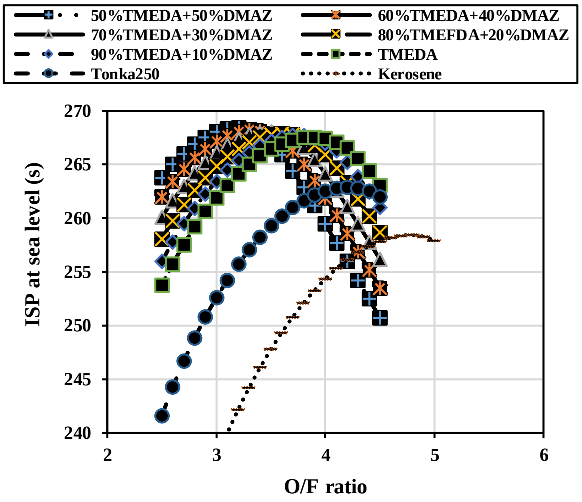

Fig. 2: Isp versus O/F ratio for different compositions of TMEDA-DMAZ and Tonka250 with AK27.

Density-specific impulse

A liquid bipropellant consists of liquid fuel and a liquid oxidizer. Also, as stated previously, a certain amount of oxidizer to fuel mass ratio maximizes the I_{sp} parameter. The specific gravity of propellant or $\delta_{\text{propellant}}$ is defined as [26]:

$$\delta_{\text{propellant}} = \frac{\delta_{\text{oxidizer}} \times \frac{O}{F}}{1+\frac{O}{F}} + \frac{\delta_{\text{fuel}}}{1+\frac{O}{F}} \tag{1}$$

Table 2: The density-specific impulse for TMEDA50%+DMAZ50% and Tonka250 with oxidizer AK27 at optimum O/F ratio.

Fuel	δ_{fuel}	$\delta_{oxidizer}$	Optimum O/F ratio	Optimum I_{sp} (s)	$\delta_{propellant}$	DI_{sp}(s)
TMEDA50%+DMAZ50%	0.848	1.606	3.2	268.42	1.42	381.18
Tonka250	0.845	1.606	4.2	262.86	1.45	381.14
Kerosene	0.806	1.606	4.8	258.42	1.46	379.36

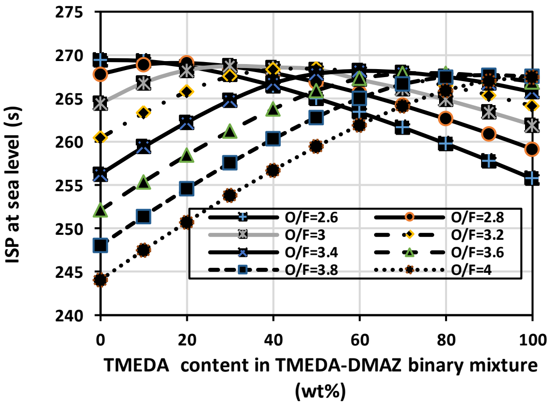

Fig.3: Dependency of Isp on TMEDA content in TMEDA-DMAZ binary mixture at various O/F ratios with oxidizer AK27.

Where $\delta_{propellant}$, $\delta_{oxidizer}$, δ_{fuel}, and *O/F* are the specific gravity of propellant, the specific gravity of oxidizer, the specific gravity of fuel, and the mass ratio of oxidizer to fuel, respectively.

Density-specific impulse is a very important parameter to assess the propellants for designing a rocket. This parameter is calculated by multiplying I_{sp} and the specific gravity of propellant or $DI_{sp}=I_{sp}\times \delta_{propellant}$.

For equal weight percentages of TMEDA in DMAZ at the best O/F ratio (maximum impulse) and also Tonka250 with oxidizer AK27, the results are given in Table 2. Kerosene is given as a comparison.

As it is presented in Table 2, the specific gravity and density-specific impulse of TMEDA50%+DMAZ50% and Tonka250 are nearly the same.

Instead of Tonka250, TMEDA50%+DMAZ50% is noncarcinogen. The performance of both fuels is the same. The ID time of TMEDA50%+DMAZ50% with AK27 is 14.13 ms whereas the ID time for Tonka250+AK27 is 24 ms. Conclusively, the binary mixture of MEDA50%+DMAZ50% can be introduced as a replacement for Tonka250 in reaction with oxidizer AK27.

CONCLUSIONS

Although Tonka250 (a mixture of **triethylamine** and xylidine with an equal weight ratio) has been used as starter fuel in the first stage of space rockets, it is carcinogenic. For replacing Tonka250, two noncarcinogenic liquid fuels tetramethyl ethylene diamine (TMEDA) and dimethyl aminoethyl azide (DMAZ) are good candidates. DMAZ has a slightly longer ID time and TMEDA has low density. Both fuels are noncarcinogen. The results obtained from experiments showed that a 50:50 wt% of TMEDA-DMAZ with AK27 has ID time of about 14 ms which is lower than Tonka250-AK27 (24 ms). The results obtained from RPA software illustrated that this proportion of the binary mixture had a specific impulse of about 6 seconds more than Tonka250 in reaction with oxidizer AK27. On the other hand, a mixture of 50:50 wt% of TMEDA-DMAZ and Tonka250 have nearly the same specific gravity and density-specific impulse. Such a combination can remove using of Tonka250 as a carcinogenic starter fuel. In other words, this combined fuel may be used as starter and sustainer or flight fuel in space rockets. It seems this combined fuel can simplify the design and manufacturing of a space rocket.

Nomenclatures

AK27	A liquid oxidizer containing nitric acid (73 wt%) and nitrogen tetroxide (27wt%)
DI_{sp}	Density-specific impulse, s
DMAZ	Dimethyl aminoethyl azide
HNO_3	Nitric acid
ID time	Ignition delay time, ms
I_{sp}	Specific impulse, s
NO_2	Nitrogen dioxide
N_2O_4	Nitrogen tetroxide
O/F	Mass ratio of oxidizer to fuel
RPA	Rocket Propulsion Analysis software
TMEDA	Tetramethyl ethylene diamine
Tonka250	Mixture of triethylamine and xylidine with the equal weight ratio

Greek

δ_{fuel}	Specific gravity of fuel [-]
$\delta_{oxidizer}$	Specific gravity of oxidizer [-]
$\delta_{propellant}$	Specific gravity of propellant [-]

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