



Hypergolicity and ignition delay study of gelled ethanolamine fuel



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ABSTRACT

An experimental investigation on achieving hypergolicity with a critical catalyst concentration and measuring ignition delay of non-metalized and metalized gelled ethanolamine was performed with hydrogen peroxide as an oxidizer. The experimental results of using high speed camera were obtained by adopting the drop tests in this work. This study represented a sufficient repeatability of ignition delay for hypergolic bipropellant system. Gelled ethanolamine fuel (non-metalized and metalized with nano-sized metal particle substitution) with metal catalysts presented the results in hypergolicity with ignition delays of the order of 1–5 ms in most of the cases, which was comparable to the existing liquid hypergolic bipropellant systems. However, the minimum ignition delay time was recorded for pure-CCAT case. Moreover, the calculated activation energy (E_{vis}) for the gelled ethanolamine fuel for non-metalized and metalized systems was within the range of 0.03–0.06 kJ/mole along with shear thinning behavior. Parameters such as apparent viscosity of the fuels, blended energetic particle, catalyst type along with its critical concentration using the drop impact plays an important role in studying the hypergolicity and ignition delay time of the gel bipropellant.

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1. Introduction

Hypergolic bipropellant systems are advantageous for several propulsion missions, mostly when multiple ignitions are an essential requirement. The term, hypergolicity, is a spontaneous ignition of a fuel and an oxidizer on contact without any external source of ignition [1]. The conventional hypergolic bipropellant systems using UDMH-RFNA and MMH-NTO are very toxic, non-ecofriendly and carcinogenic [2]. The interest in using alternative ecofriendly bi-propellant systems such as alcohol, amine and hydrogen peroxide propellants in rocket propulsion and missile application has been renewed over the past decades [2–7]. The use of catalysts for promoting decomposition, ignition and combustion of hydrogen peroxide oxidizer with organic fuel is one of the emerging topics recently.

Alcohol or amine and hydrogen peroxide based bipropellant system can provide several advantages compared to the current hypergolic systems (UDMH/MMH-RFNA/NTO). Its intrinsic properties are high density, low vapor pressure, less toxic and corrosive, and more ecofriendly. Additionally, the combustion product

is also less toxic in comparison to the conventional hypergolic propellant systems. Current studies have employed the approach of using metal catalyst with amine family and hydrogen-peroxide propellant system for hypergolic ignition. The study is mainly focused on the critical catalyst concentration required for introducing hypergolicity in the formulated gelled fuel system. Gel propellants are modified conventional liquid system whose physico-chemical properties can be altered by the addition of a suitable gelling agent through a three-dimensional network system that transforms the parent fuel/oxidizer into a gel by changing its properties from a free-flowing liquid to semisolid. These gels are non-Newtonian fluids whose rheological properties play a vital role in effective end usage [8–10].

Along with the critical catalyst concentration for hypergolicity, ignition delay time is also a critical parameter for propulsion system design of injector. Ignition delay (ID) is the time between fluid contact and the appearance of first flame. It is a function of parameters such as mixing ratio between fuel and catalyst, kinetics, heat transfer, ambient temperature and pressure. However, in the case of gel propellant system, the viscosity and viscoelastic properties play a vital role, since it has to overcome mixing resistance to initiate a local chemical reaction for ignition [3–15]. Several studies have been performed to understand the effects of the above parameters on ID and hypergolicity of liquid propellant

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Table 1
Summary for the gel fuel system.

| Fuel | Gelling agent | Catalyst for hypergolicity | Oxidizer for hypergolicity |
|-------------------------|--|----------------------------|----------------------------|
| Ethanolamine (liquid) | – | CCAT/MCAT | Hydrogen peroxide |
| Ethanolamine (gel) | PVP + SiO ₂ (6 + 6 wt%) | CCAT/MCAT | |
| Ethanolamine + Al (gel) | [PVP + SiO ₂ (5.5 + 5.5 wt%)] + Al(2 wt%) | CCAT/MCAT | |
| Ethanolamine + B (gel) | [PVP + SiO ₂ (5.5 + 5.5 wt%)] + B(2 wt%) | CCAT/MCAT | |
| Ethanolamine + C (gel) | [PVP + SiO ₂ (5.5 + 5.5 wt%)] + C(2 wt%) | CCAT/MCAT | |

system. However, less effort has been made on gel system itself. Gelled propellant systems (non-metalized and metalized) have a marked difference in terms of their physico-chemical properties in comparison to liquid hypergolic propellants, since they may lead to significant changes in their ignition and combustion behaviors [16–20]. The other parameter, which plays an important role in hypergolicity and ignition delay time of the gelled propellant, is the type of thickening agent, organic or inorganic. Normally, gelling agents are less energetic. However, if the thickening agent can add positive heat of formation to the parent fuel, this would improve the overall performance of the gel propellant in terms of hypergolicity and ignition delay time [1,11–13]. Usually, organic gelling agent participates in the combustion of the parent fuel, while affecting the combustion performance, compared to inorganic gelling agent based gel propellant system. Alcohol and amine fuels give a respectable performance in terms of specific impulse (Isp) (e.g., liquid ethanol with hydrogen peroxide, and ethanolamine with hydrogen peroxide have a calculated Isp of 261 s and 245 s compared to 275 s for hydrogen peroxide with monomethyl hydrazine and 284 s for nitrogen tetroxide (NTO) and MMH. Gelation would help in increasing the density of the gel propellant and in turn the density Isp [21,22].

Previously, the initial testing combinations for hypergolicity were done through drop test. The drop test might provide the best environmental condition for repeatability of experimental results, although the drop test differs considerably from the end propulsion application in which aggressive mixing is involved [21,23–26]. In the meanwhile, for the study of hypergolicity and ignition delay time of the gel bipropellant system, drop impact plays a vital role in providing energy to overcome viscosity barrier between fuel and oxidizer as well as better mixing to initiate ignition.

There are a number of diagnostic tools for studying hypergolicity which have been used in the past. However, this article presents a discussion of hypergolicity and ID experimentation using a high speed camera to determine the propellant decomposition and ID time of gelled ethanolamine fuel with pure and metal catalyst mixture along with the origin of ignition, while keeping the critical catalyst concentration for hypergolicity. Both non-metalized and metalized fuel gel cases were studied by using hydrogen peroxide as an oxidizer which is stored in a controlled environment. The error calculation was made for the experimental reading regarding the weight percent ratios to achieve a good approximation for the calculated ignition delay values [27,28]. This would provide a significant insight into the ability to replicate test conditions.

2. Materials and method

2.1. Materials

For current experimentation, the ethanolamine (99.5% pure, CAS No. 141-43-5, Sigma Aldrich Corp., South Korea) was used as a base fuel, while a hybrid pair of PVP (polyvinylpyrrolidone, CAS No. 9003-39-8, Mol. Wt. 10,000) + SiO₂ (Fumed Silica, CAS No. 112,945-52-5, 0.2–0.3 μm, hygroscopic) was used as a gelling agent. The non-metalized fuel gel sample comprises

6 + 6 wt.% of gelling agent, while the metalized case consists of 5.5 + 5.5 wt.% of gelling agent and 2% by weight of nano metal particles [Al (aluminum, 50–100 nm), B (boron, 200–250 nm), and C (carbon, 50 nm)] (Table 1). All the samples were listed in the nomenclature and hereafter are to be represented as S₁, S_{Al}, S_B and S_C respectively. For both the gel cases (non-metalized fuel gel and energetic gel), PVP and SiO₂ were introduced at a critical concentration for gelation of the parent fuel (ethanolamine). When energetic particles were introduced, the concentration of PVP + SiO₂ was reduced again to achieve a critical concentration in which the system maintains its gel like property and possess shear thinning behavior with very low yield point, as energetic particles do not participate in gelation of the gel system. The addition of these energetic particles provides additional energy to the gel propellant while the gel network provides the homogeneous suspension of these particles, while avoiding the sedimentation and agglomeration of these particles to any surface or container. These energetic systems in turn provide better performance as a homogeneous gel propellant for propulsion application. Below this gellant concentration, the system does not hold gel like behavior (viscoelastic behavior), instead acts as a viscous fluid. The oxidizer used for current experimentation is a propellant grade liquid hydrogen peroxide (90%). To achieve hypergolicity with the base fuel (both in non-metalized and metalized cases), the catalysts such as copper chloride hydrous (CuCl₂·2H₂O, 99.999% pure, CAS No. 7447-39-4, Sigma Aldrich Corp., South Korea) and manganese (II) acetylacetonate (C₁₀H₁₄MnO₄, CAS No. 14024-58-9, Sigma Aldrich) were introduced. These catalysts will be represented as CCAT and MCAT respectively, hereafter.

2.2. Method and experimental setup

In the current experimentation, the main objective was to understand the gel viscosity behavior, hypergolicity, critical catalyst concentration to achieve hypergolicity, ignition delay time of the formulated gel based bipropellant system, and the temperature variation. Rotational rheometer (HAAKE RheoStress 6000, by Thermo-Scientific, Germany) was used for viscosity behavior study. The hypergolicity, ignition delay and the temperature profile of the bipropellant system were monitored and recorded using DSLR and high speed camera, Photron SA-X2, and K type thermocouples by OMEGA Corporation with a bead diameter of 0.127 mm with Teflon insulation. These thermocouples were directly connected to the data acquisition card, PDAQ56 without any additional copper wire connections. Due to its very small bead size of thermocouple, no significantly large errors could be introduced and also radiation has a minimum effect. Figure 1 shows the experimental setup used for the hypergolicity and ignition delay studies.

Injector by “Eppendorf” model “Multipette® M4” was used with a volume dispenser of 5.0 ml (blue) and the dispensing diameter of 0.1 mm. This dispenser was used to introduce oxidizer from the height of 270 mm, directly above the top of the gelled fuel bed on a thin quartz glass slide. The weight percentage of fuel and catalyst in the mixture was in the range of 99.03–99.69 wt% of fuel and 0.31–0.97 wt% of catalyst, respectively. To record the temperature, thermocouples were placed at three different positions as seen in

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