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Research paper

Stabilized Superthermite Gelled Kerosene: Towards Advanced Green Propellant Systems

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Abstract: Although hydrazine is the most common liquid propellant fuel, it is highly toxic and cancerogenic. Gelled hydrocarbons could be the greener substitute. Kerosene was gelled using fumed silica nanoparticles (NPs). Reactive metal particles can act as a high energy dense material (HEDM). With this aim, gelled kerosene was loaded with aluminum (Al) NPs. In combustion, SiO_2/Al can induce vigorous exothermic superthermite reaction. Gelled kerosene demonstrated shear thinning behaviour, with high gel stability at 90 g centrifugal acceleration. The silica NPs could form a network *via* hydrogen bonding of Si–OH groups; this network could be broken down under a high shear rate. Aluminized gelled kerosene formulation (8 wt.% SiO_2 + 8 wt.% Al) preserves the shear thinning behaviour, *i.e.* it reached the viscosity of liquid kerosene at a shear rate below 25000 S^{-1}. This value lies within the range of pumping systems in rocket engines. Metallized gelled formulations demonstrated yield stress that is required to avoid phase separation and sedimentation during storage. Stabilised superthermite NPs not only offered enhanced characteristic exhaust velocity by 6% using the ICT thermodynamic code. Furthermore, they could induce vigorous exothermic superthermite reactions.

Keywords: nanothermite, gelled propellant, green propellant, rheology

1 Introduction

Hydrazine is the universal propellant for space exploration and thrusters due to its high specific impulse (I_{sp}) and low flame temperature. Hydrazine can be employed as a monopropellant; as it can be easily decomposed by a catalyst [1]. Hydrazine can also be used in a bipropellant system, it reacts hypergolic with dinitrogen tetroxide [2, 3]. On the other hand, hydrazine is highly toxic and requires high safety precautions. Due to its carcinogenic nature, the European Chemical Agency, considered hydrazine a substance of very high concern, *i.e.* SVHC, [4]. Therefore, hydrazine will be prohibited from use and much research has been directed to replace hydrazine [5, 6]. The demand to develop less toxic, eco-friendly green propellant systems that meet growing mission requirements is continuously increasing [7]. Recently, scientific efforts have focused on replacing conventional propellants with non-toxic and eco-friendly propellants [8]. According to the European Space Agency, green propellants should reduce hazards; in the meantime, they should secure the required performance [9]. Green propulsion strategy can reduce the negative impact on the environment. The demand for high-performance green propellant systems is continually increasing [10]. Hydrocarbon fuels can replace hydrazine in bipropellant systems. Hydrocarbon fuels are considered greener compared to hydrazine-based fuels. However, hydrocarbons have a much lower I_{sp} than hydrazine-based fuels.

Kerosene, Jet A-1, JP-4, JP-5 and RP-1, are the most common hydrocarbon fuels used for bipropellant systems. Kerosene could be the suitable choice for green propulsion technology because it can be gelled with different gelling agents. Gelled propellant is used where the mechanical properties of conventional liquid propellants can be altered by adding a suitable gelling agent [11, 12]. Gelled propellants have the ability to combine the major advantages of liquid and those of solid propulsion systems; in the meantime, they can eliminate any disadvantages [13]. Gel propellants can overcome leakage and sloshing problems as well as they offer safer storage and handling similar to solid propellants [14, 15]. Furthermore, gelled propellants can secure enhanced viscosity and stability (Figure 1).

Figure 1. Enhanced viscosity, and stability of metalized gel propellant [16, 17]

Gelled propellants are generally less sensitive to impact, friction, and electrostatic discharge. Additionally, gelled propellants deliver a I_{sp} comparable to that of liquid systems. The gel chemical structure allows loading the propellant with energetic additives that could ensure enhanced performance. Gelled propellants are non-Newtonian fluids that exhibit shear thinning and thixotropic behaviour. However, gelled propellants behave as viscoelastic solids during storage; they behave as liquid during the feeding and atomization stage [18]. Most liquid fuels and oxidizers can be converted into gels by adding a suitable gelling agent [19]. Gellants are classified into two main types [20]:

- inorganic such as fumed silica and sodium silicate, and
- organic gellant such as cellulose compounds.

Inorganic gellants are considered to be inert materials with a negative impact on propellant performance.

Gelled kerosene can offer the dispersion and stabilization of reactive metal particles, like aluminum, magnesium and boron. Reactive metal particles can increase the combustion enthalpy; therefore, enhanced performance could be achieved [21]. Metallized gelled hydrocarbons are a green competitor to hydrazine; in the meantime, they could secure enhanced performance. Gelled propellants are considered a great step at the onset of green propulsion [8]. The presence of metal additives such as aluminum (Al), magnesium (Mg) or boron (B) can offer the ability to increase I_{sp}, propellant density and system safety [1]. The future missile technology integration program demonstrated numerous benefits of gelled propellant, including extended range, improved kill power, greater mission flexibility, and lower operational costs [6]. In the present work, kerosene which is the common green hydrocarbon propellant, was gelled with fumed silica nanoparticles NPs.

Gelled propellants are exposed to different shear rates during their usage ranging from 0 to 10^{-2} S^{-1} during storge passage by 10^{-2} to 10^{3} S^{-1} during flow until 10^{6} S^{-1} is observed at atomization [20]. Gelled kerosene can act as a shear-thinning material; the viscosity decreases with increase in shear rate. The viscosity of gelled kerosene matched the viscosity of liquid kerosene at a high shear rate. All of rheological properties in addition to stability behaviour of different formulations of gelled kerosene fuel were experimentally tested. Although gelled kerosene show shear thinning behaviour at all different fumed silica formulations, it achieves optimal stability at 8 wt.%. SiO_2 within 90 g centrifugal acceleration stability test. Reactive Al NPs of 70 nm average particle size were effectively dispersed and stabilized within gelled kerosene. Gelled kerosene preserves shear thinning behaviour with nano Al addition. The most viscous metalized gelled kerosene (8% SiO_2 + 8% Al) formulation prepared reached the viscosity of virgin kerosene at a shear rate below 25000 s^{-1}; this is within the range of pumping systems in rocket engines. The ballistic performance of the propellant is calculated theoretically using the ICT thermodynamic code (Institute of Chemical Technology, Germany, 2008). The optimum fuel formulation is achieved at 6 wt.% of Al loaded in kerosene gelled by 8 wt.% of fumed silica taking into account stability, rheological and ballistic performance. Al NPs not only offered enhanced characteristic exhaust using a velocity of 6% but also Al NPs can induce thermite reaction with fumed silica NPs.

2 Experimental

2.1 Materials and samples preparation

Kerosene is considered a good representative for widely used propellant RP-1. High-refined kerosene (Aldrich, CAS: 8008-20-6) was used as the liquid propellant. Analytical grade fumed silica NPs of 10-20 nm average particle size (Aldrich, 99.5%, CAS: 112945-52-5) was employed as inorganic gelling agent for kerosene. Reactive Al NPs (99.9%, US Research Nanomaterials) of 70-nm average particle size were utilized as energetic additives.

The morphology and average particle size of the Al and fumed silica NP_S were investigated by transmission electron microscope (TEM, Philips CM20). Different percentages of SiO_2 gelling agents (7, 8 and 9 wt.%) were investigated. The gelation process depends not only on the gellant type but also it depends on the particle size, mixing time, mixing procedure, and operating temperature. Gelled kerosene (8 wt.% SiO_2) was loaded with Al NPs at different concentrations (4, 6 and 8 wt.%). Gel formulations were prepared by conventional mixing at room temperature, where the fumed silica and metal additives were added stepwise. Mechanical mixing was carried out with a vertical mixer at 100 rpm for about 1 h. The chemical composition of the investigated gel formulations is tabulated in Table 1.

Table 1. Chemical composition of the gel formulations

Sample number	Gel system formulation [wt.%]			Observation
	Dispersion medium	Gellant (fumed silica) [wt.%]	Al [wt.%]	
1	Kerosene	7	–	Thick gel, no separation
2		8	–	
3		9	–	
4		8	4	
5			6	
6			8	

2.2 Evaluation of gel propellant

The gel propellant should meet three main requirements including rheological and stability tests and performance parameters:

- The rheological test was conducted to investigate the viscosity behaviour over a wide range of shear rates and to verify the viscoelastic behaviour of developed gel propellant formulations.
- The ability of gel propellant to withstand the operating conditions of rocket engines was measured by centrifugal tests.
- The performance of gel propellant formulations was theoretically evaluated *via* thermochemical calculations using the ICT thermodynamic code (Institute of Chemical Technology, Germany, 2008).

2.2.1 Rheological test

Characterization of the rheological behavior of non-metallized and metalized gel formulations (Table 1) was carried out by a parallel plate rotational rheometer (Anton Paar MCR-301). The viscosity of the gel samples was measured with shear rates ranging from 0.1 to 1000 S^{-1}. The adopted shear rate range mimics the conditions from storage to injection pass with flow in pipes. The apparent viscosity of the developed gel formulations was affected by temperature. All rheological tests were carried out at 25 ^{o}C to eliminate the temperature effect.

2.2.2 Stability test

The stability of the gel propellant was quantitatively evaluated through accelerated tests. These tests were conducted *via* centrifugal test. This test simulates mechanical loads that could cause gel structure deterioration.

Gel stability was assessed by determining the amount of gel remains after the sample was subjected to centrifugal accelerations for a certain period of time. Bunsen Histam Plus-RH c 18000 Rpm 30065 xG was used to provide centrifugal accelerations that simulate launching and flight conditions. Gel formulations (6, 7 and 8 wt.% of fumed silica) were subjected to 90 g and 1500 g centrifugal acceleration for 10 min. The separated liquid phase was removed, and the remaining gel mass was weighted. The remaining gel mass gives an indication of the gel structure stability.

2.2.3 Thermochemical evaluation

The influence of Al content on gelled kerosene performance was theoretically evaluated by ICT thermodynamic code (Institute of Chemical Technology, Germany, 2008). It was assumed that the pressure inside the combustion chamber was 70 atm and the gas expansion was up to 1 atm [22]. The impact of Al content on gel performance was theoretically evaluated. The rocket engine based on the bipropellant system based on gelled kerosene as fuel and red-fuming nitric acid (75% HNO_3 and 25% N_2O_4) as oxidizer and fuel to oxidizers (F/O) ratio 3:1 was adopted as the case study [23].

3 Results and Discussions

3.1 Nanoparticle characterization

The morphology of starting silica NPs (gelling agent) was investigated using TEM. TEM micrographs demonstrate that the primary particles of the fumed silica before dispersion in kerosene medium are about ~10 to 25 nm (Figure 2).

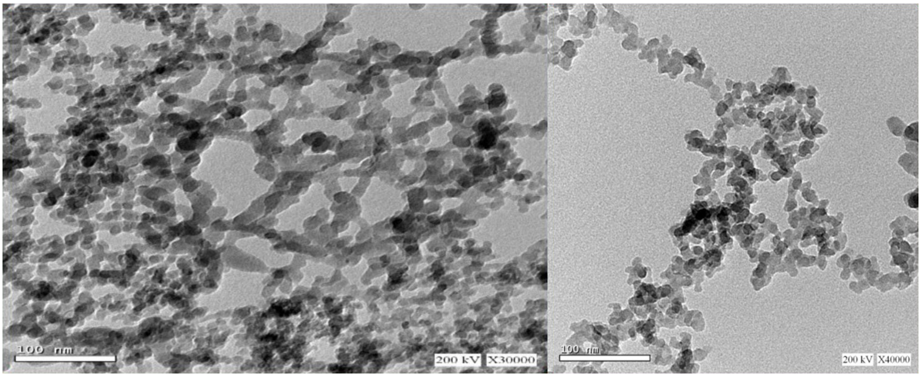

Figure 2. TEM micrographs of the fumed silica gelling agent

TEM micrograph demonstrated the tendency of silica NPs to agglomerate forming 3-D network clusters [24, 25]. Morphology of starting Al NPs was investigated with TEM; TEM micrographs demonstrated spherical Al particles with a mean particle size of 70 nm (Figure 3).

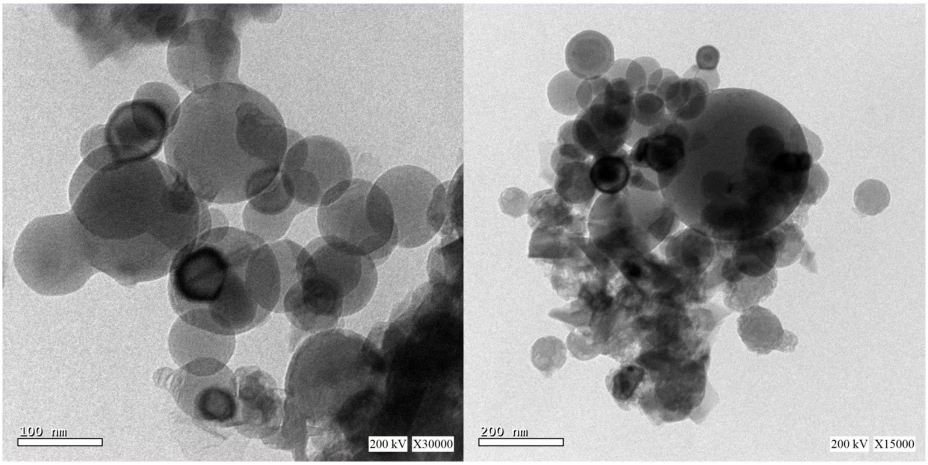

Figure 3. TEM micrographs of Al NPs

3.2 Rheological characterization of gelled kerosene

Gelled propellants are expected to be exposed to different shear rates varied from 10^{-2}, 10^{3}, and 10^{6} S^{-1} during storage, flow, and atomization respectively [20]. The impact of the silica content (7, 8 and 9 wt.%) on the viscosity of the gel was investigated over the shear rate ranging from 0.1 to 1000 S^{-1} (Figure 4).

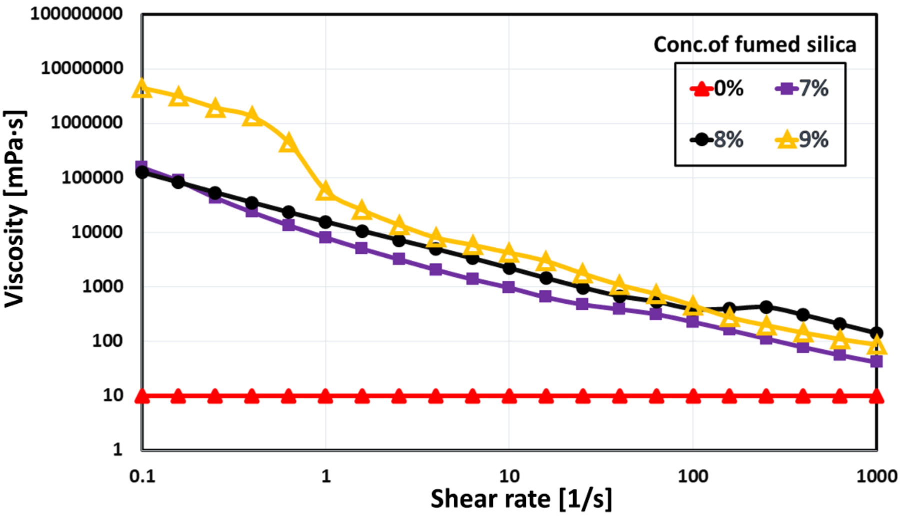

Figure 4. Gel viscosity as a function of gelant concentration and shear rate

Gelled kerosene can act as a shear-thinning material, where the viscosity decreases as the shear rate increases. The influence of fumed silica is more significant at low shear rates compared to high shear rates. It is induced that as the shear rate increases, the apparent viscosity of gelled kerosene continues to decrease until it reaches the viscosity of its liquid base kerosene.

It is obvious that as the amount of fumed silica increases, the gel becomes more viscous. The hydrophilic surface of the fumed silica plays an essential role in the formation of the gel network [26]. The Si–OH groups on the surface of fumed silica particles cause the hydrogen bonding particle-particle interaction, which form the network where the kerosene molecules are entrapped (Figure 5(a)). The viscosity of the gel decreased under the effect of the shear rate due to the breakdown of the fumed silica network (Figure 5(b)).

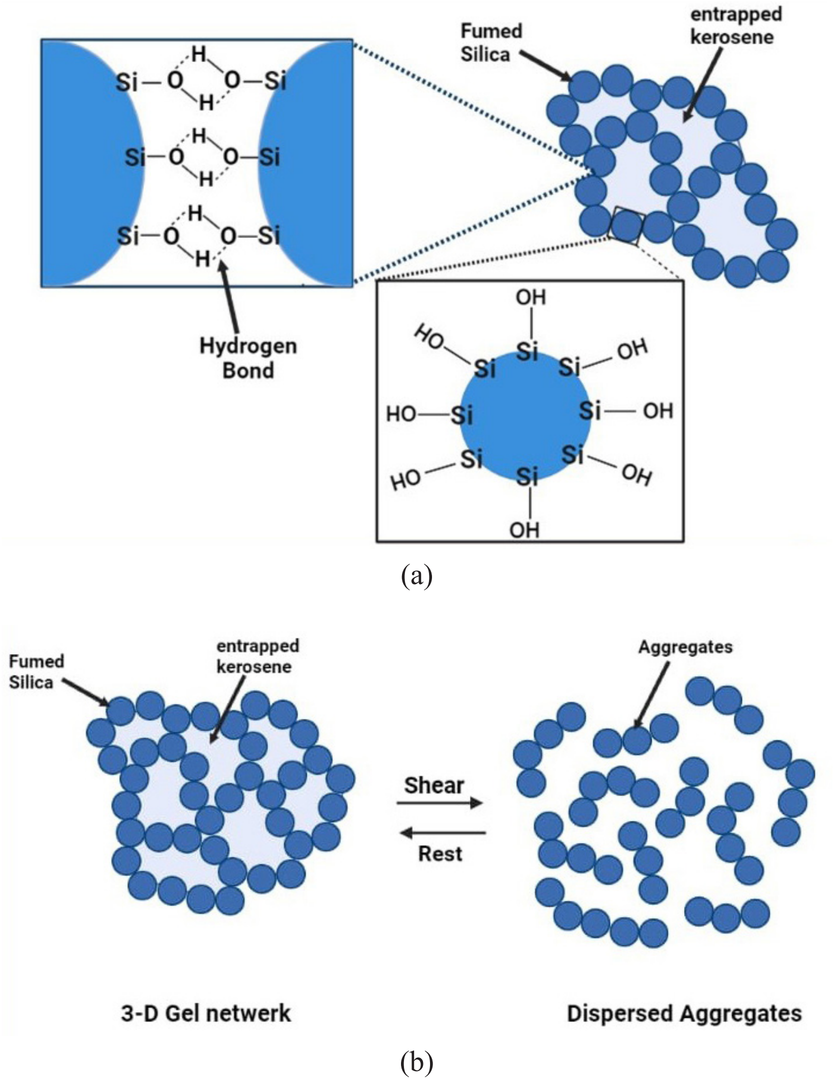

Figure 5. Silica network via hydrogen bonding interaction (a) and impact of shear on silica network (b)

It can be concluded that controlled viscosity of gel can be achieved by controlled shear rate on the gel structure [27]. Reactive metal particles with high heat output can act as an efficient HEDM. Gel propellant can offer a novel approach to disperse and stabilize reactive metal particles; furthermore, it can secure stable colloidal particles.

3.3 Stability of gelled kerosene

Gel stability is an important feature for assessing the ability of the gel to withstand mechanical loads during operation. Gel stability was determined by measuring the physical separation of the gel matrix after being subjected to centrifugal acceleration that mimics flight conditions [28]. The positive impact of the gelling agent content on the stability of gelled kerosene at two different centrifugal accelerations (90 g, 1500 g) is demonstrated in Figure 6. The remaining gel mass was weighed; it indicates gel stability.

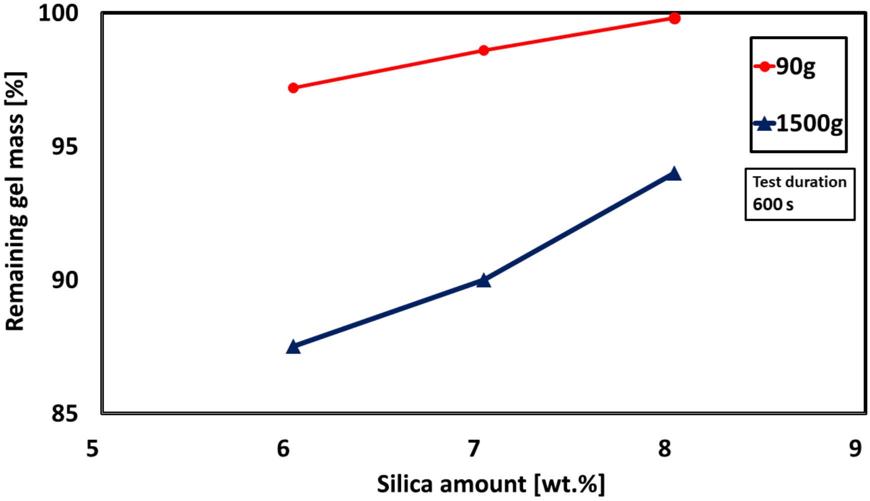

Figure 6. The impact of fumed silica content on gel stability at different centrifugal accelerations

It is obvious that there is an increase in the gel stability with the increase in the silica content. It was noticed that there was no physical separation in 8% fumed silica at the 90 g test. The optimal gelling agent content was reported to be 8 wt.%. Although there was an increase in gel stability with fumed silica content; fumed silica has a negative impact on propellant I_{sp}. Consequently, a gel propellant with balanced stability and performance is required. This can be accomplished through the proper dispersion of reactive metal particles. Reactive metal particles such as Al can act as HEDM; furthermore, it can induce vigorous superthermite reactions with fumed silica NPs.

3.4 Rheology of aluminized gelled kerosene

Al particles can secure combustion heat of 32000 J/g. Consequently, Al can act as an efficient HEDM. On the nanoscale, high interfacial surface area and high reactivity can be achieved [29]. 70-nm Al NPs were effectively dispersed in gelled kerosene. The impact of Al NPs on rheology of the gelled kerosene (based on 8 wt.% silica) was investigated (Figure 7).

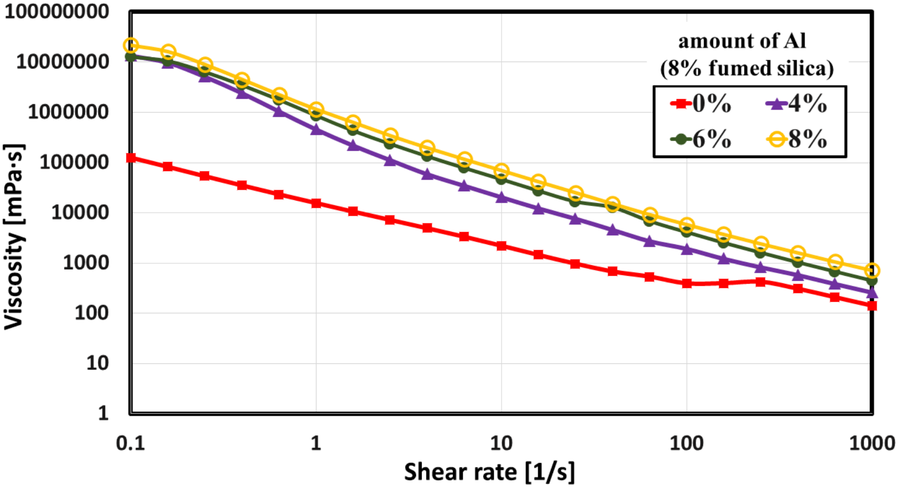

Figure 7. Gel viscosity as a function of metal concentration and shear rate

The influence of metal loading on the gelled kerosene viscosity within the range of shear rates 0.1 to 1000 S^{-1} was assessed. The increase in viscosity of gels is clearly observed with increasing Al content. The dispersed Al particles could act as a secondary gellant [30]. The great influence of metal additives on viscosity is noticed at low shear rates. Despite increasing viscosity, the kerosene gelled propellant loaded with Al metal preserves shear-thinning behaviour.

3.5 Yield stress of gelled kerosene

Proper yield stress is required to avoid phase separation and sedimentation during storage [11]. Gelled kerosene and metalized gelled formulations demonstrated yield stress that is required to avoid phase separation and sedimentation during storage. There was an increase in yield stress with the content of the gelling agent. On the other hand, Al NPs demonstrated a significant impact on yield stress (Table 2).

Table 2. Yield stress of gel system

Gel system formulation	Yield stress [Pa]
Kerosene	0
Kerosene + 7 wt.% fumed silica	8
Kerosene + 8 wt.% fumed silica	22
Kerosene + 9 wt.% fumed silica	40
Kerosene + 8 wt.% fumed silica + 4 wt.% Al	160
Kerosene + 8 wt.% fumed silica + 6 wt.% Al	400
Kerosene + 8 wt.% fumed silica + 8 wt.% Al	600

The dispersion of Al could result in a high resistance to gel flow. Among the flow models, the Oswald-de Waele power law is the most widely used flow model for the gel propellants [16, 20]. According to the power law, the most viscous gel formulation prepared (8% SiO_2 + 8% Al) achieves the viscosity of liquid kerosene at a shear rate below 25000 s^{-1}, *i.e.* within the range of pumping systems currently used in rocket engines (Figure 8). The power law states that shear thinning behaviour is noticed at index value $0 < n < 1$. However, the tested gel propellant formulation has a negative index value ($n = -0.152$). This negative value can be explained by simplifications and restrictions of the power law that consider the gel as time independent. In fact, the gel structure is a thixotropic material whose viscosity decreases as time increases at constant shear rate [11, 31].

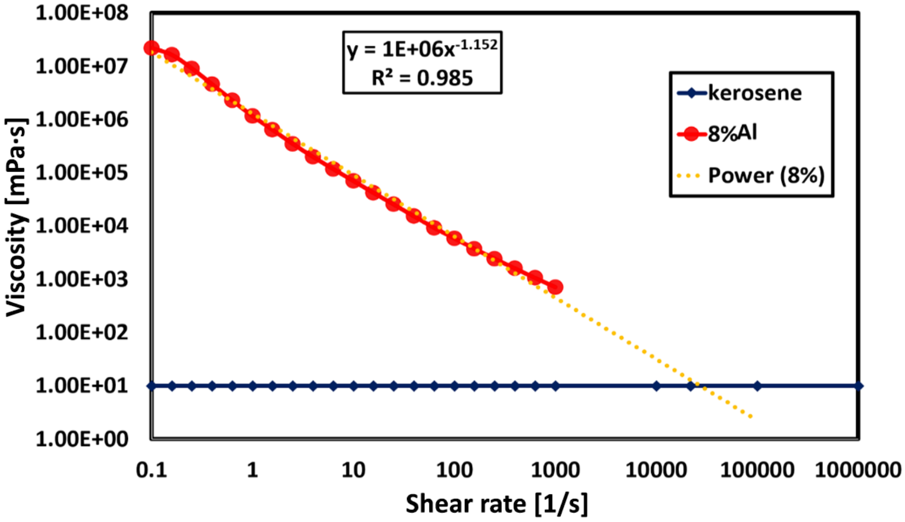

Figure 8. Viscosity of aluminized gelled kerosene based on power law

4 Thermochemical Calculations

The impact of Al NPs on the combustion characteristics of gelled kerosene was theoretically evaluated by ICT thermodynamic code (Institute of Chemical Technology, Germany, 2008). There was an increase in the temperature of the gelled kerosene flame with Al content (Figure 9).

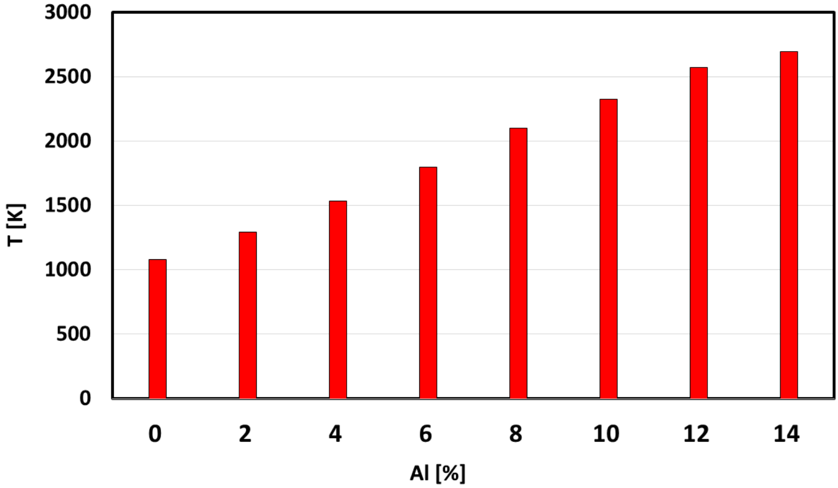

Figure 9. Propellant flame temperature function of Al content

This improvement in combustion is related to the high combustion heat of Al particles (7.34 kcal/g) [32]. The I_{sp} and characteristic exhaust velocity of gaseous products are essential criteria that must be evaluated to determine the ballistic performance of the propellant. Al content of 6 wt.% offered an increase in characteristic exhaust velocity and I_{sp} by about 6%. An increase in Al content was reported to decrease the performance. It can be concluded that the optimum loading level of Al is 6 wt.% (Figure 10).

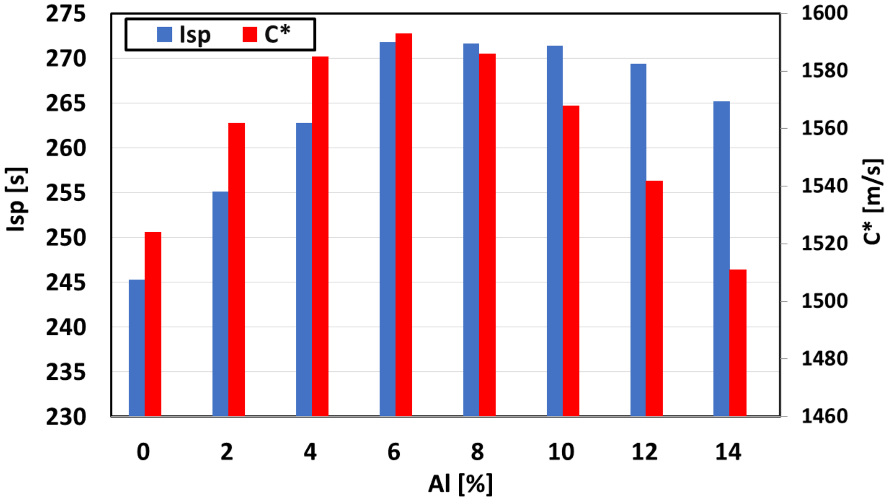

Figure 10. Impact of the content of Al on I_{sp} and characteristic exhaust velocity

The enrichment of the flame temperature and the decrease in the molecular weight of gaseous products could withstand the positive impact on I_{sp} and characteristic exhaust velocity of gaseous products [33]. Although fumed silica serves as an inert gelling agent that compromises the performance, the existence of nano-Al increases the possibility of nanothermite reaction which causes further improves performance by generating 2.15 kJ/g [34-37]. The nanothermite reaction possibility between gellant fumed silica and nano-Al converts inert silica into active energetic material [37].

5 Conclusions

- In this work, a novel aluminized gelled kerosene is introduced as a powerful green candidate that can replace the toxic, cancerogenic hydrazine in the near future.
  Shear thinning gelled kerosene was developed using fumed silica NPs, as an inorganic inert gelling agent of kerosene. Different concentrations of 10 nm average particle size fumed silica NPs were tested.
- The rheological characterization and stability behavior of prepared formulations are quantitively measured. The amount of gellant has a significant influence on the viscosity of kerosene. Through the accelerated stability test, the optimum gel stability was achieved at 8 wt.% fumed silica.

- Silica NPs form a network *via* hydrogen bonding of Si–OH groups that hold kerosene inside; this network would break down under a high shear rate. Reactive Al NPs could act as a HEDM that enhances the combustion enthalpy of gelled kerosene. The most stabilized gelled kerosene (8 wt.% silica) was loaded with Al NPs. The optimum Al content was theoretically reported to be 6 wt.%.
- The most viscous gel (8% silica + 8% Al) demonstrated reached the viscosity of liquid kerosene at a shear rate below 25000 s^{-1}, *i.e.* within the range of pumping systems currently used in rocket engines.
- Al NPs offered an increase in gel performance by 6%. Furthermore, Al could induce vigorous superthermite reaction with silica NPs turning it into reactive particles.

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Contribution

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