Characterization of JP-8/SiO$_2$ and RP-1/SiO$_2$ Gels

P. H. S. Santos*, R. Arnold†, W. E. Anderson‡, M. A. Carignano§, and O. H. Campanella¶

Abstract—**For next generation air-breathing and rocket propulsion systems gelled propellants can be a promising replacement for conventional solid and liquid propellants. Especially for rocket engine operation, the application of a gelled fuel and gelled oxidizer can combine the advantages of conventional solid and liquid propellants without taking into account the specific disadvantages of both individual systems. The present publication describes the rheological behavior of gelled JP-8 turbine fuel and gelled rocket propellant RP-1 when fumed silica is used as a gelling agent. Alongside the determination of an optimal gel mixing process, gel stability and rheological parameters showed a significant influence of the added silica amount.**

Keywords: hydrocarbon, JP-8, RP-1, gel, silica, non-Newtonian, advanced propellants, rocket engine

1 Introduction

Gels are non-Newtonian fluids which have an almost solid like behavior when no shear stress is applied. In general, they consist at least of the original fluid and the added gelling agent. During the gel mixing process, molecular forces like hydrogen bonds or van der Waals forces may create a more or less soft solid structure. The quality and character of the gels are depending amongst other parameters especially on the type and amount of added gelling agent.

Since the requirements for gelled and liquid propellants are similar in terms of chemical stability during storage and thermal stability during the combustion process, investigations in the past concentrated on typical gelled fuels like MMH (Monomethylhydrazine) and UDMH (Unsymmetrical dimethylhydrazine), DMAZ (2-Dimethylaminoethylazide), and also on different types of hydrocarbon fuels like JP-5, JP-8, JP-10 and RP-1. Typical investigated oxidizers are WFNA (White fuming nitric acid), IRFNA (Inhibited red fuming nitric acid) and NTO (Nitrogen tetroxide)[14]. For the production of a gel, two different groups of gelling agents exist: inorganic gelling agents, which remain unburned when the gel is combusted, and organic gelling agents which burn together with the fuel. The most common inorganic gelling agent is silica SiO$_2$, which is especially used to gel nitric acid. Typical organic gelling agents are cellulose-based derivatives like HPC (Hydroxypropylcellulose), HEC (Hydroxyethylcellulose) and HAC (Hydroxyalkylcellulose). Most promising combinations of gelled propellants for future rocket applications are similar to liquid hypergol combinations. However, metallic additives like aluminium, magnesium or boron powder may be added to the propellants to increase the energy density of the propellant. Typical metallized propellant combinations are MMH/Al/IRFNA and MMH/Al/NTO [14, 16].

Advantages of gelled propellants in comparison with standard liquids and solids are almost liquid-propellant-like high specific impulse, wide range thrust control and re-ignitability in addition to solid-propellant-like good storage behavior and safety aspects. Table 1 gives a characterization of liquid, solid and gelled propellants [5, 11].

Table 1: Comparison of different propellants

requirement	liquid	solid	gel
easy to handle	-	+	+
performance	+	-	+
re-ignitable	+	-	+
throttleability	+	-	+
safety aspects	-	+	+
system complexity	-	+	+
storage stability	-	+	+

Storage stability is a very important factor for long-time applications like deep-space missions, and is a further advantage of gelled propellants compared to some liquids. For example, hydrogen peroxide (H$_2$O$_2$) systems have to be vented continuously to prevent overpressure in the tank, which results in a significant propellant loss during storage time [10]. On the other hand, gels are not completely understood by now. Especially the different atomization and burning process needs further investigation. Furthermore, gel production, gel stability during

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P. Santos is with the Department of Agricultural & Biological Engineering, Purdue University, West Lafayette, IN 47907 USA (phone: (765)-496-6330, fax: (765)-496-1115, email: psantos@ecn.purdue.edu)

†R. Arnold is with the Department of Aeronautical and Astronautical Engineering, Purdue University, West Lafayette, IN 47907 USA (email: arnold17@purdue.edu)

‡W. Anderson is with the Department of Aeronautical and Astronautical Engineering, Purdue University, West Lafayette, IN 47907 USA (email: wanderso@purdue.edu)

§M. Carignano was with the Department of Chemistry, Purdue University, West Lafayette, IN 47907 USA. He is now with the Department of Biomedical Engineering, Northwestern University, Evanston, IL 60208 USA (email: cari@purdue.edu)

¶O. Campanella is with the Department of Agricultural & Biological Engineering, Purdue University, West Lafayette, IN 47907 USA (email: campa@purdue.edu)

launch and flight as well as pressure losses in the propellant feed system due to higher viscosities have to be studied in detail to guarantee a safe vehicle operation. For that reason, gels envisaged for the application in a future rocket or airbreathing propulsion system have to show a shear-thinning behavior (viscosity of the gel decreases with an increasing shear rate). Furthermore, thixotropic behavior (viscosity of the gel decreases in time when a constant shear rate is applied) could give some advantages in terms of reduced pressure losses [15, 13].

2 Non-Newtonian fluids

For any fluid the viscosity η is dependent on the applied shear rate $\dot{\gamma}$. For the characterization of Newtonian and non-Newtonian fluids without a yield point, the power-law model (Ostwald-de Waele model) is the most commonly used correlation [8]:

$$\eta = K\dot{\gamma}^{n-1} \qquad (1)$$

K is the consistency index, whereas n gives the power-law index of the fluid. For $n = 1$ equation (1) describes a Newtonian fluid. However, shear thinning behavior is given for a power-law index $0 < n < 1$, and shear thickening (dilatant behavior) for $n > 1$. Fig. 1 depicts the stress behaviour as a function of applied shear rate for typical Newtonian and non-Newtonian fluids with and without a yield point.
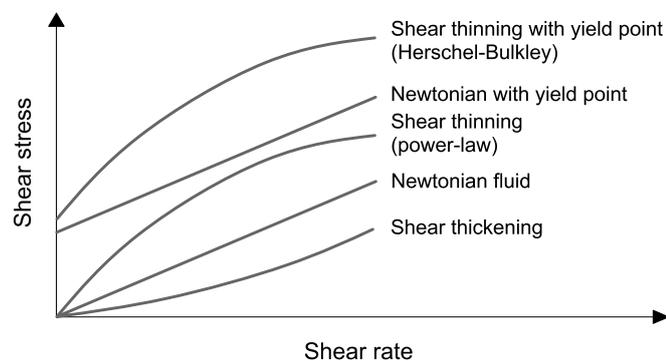
Figure 1: Newtonian and non-Newtonian fluids [13]

For fluids with yield stress τ_0 the following model can be used:

$$\tau = \tau_0 + K\dot{\gamma}^n \qquad (2)$$

3 Materials

3.1 Fumed silica as a gelling agent

Untreated fumed silica SiO_2 (CAB-O-SIL® grade M-5) was used to gel the pure hydrocarbon fuels JP-8 and RP-1 within the present study. In general, silica results from a combustion process of silicon tetrachloride ($SiCl_4$) in a H_2/O_2 flame (see equation (3)). Due to agglomeration a white and fluffy powder is created with an agglomeration size of less than $44 \cdot 10^{-6}$m and a hydrogen chloride level of less than $60\,$ppm after a following calcining process.

The aggregate chains of the silica are hereby a composition of fused together single particles [4].

$$SiCl_4 + 2H_2 + O_2 \xrightarrow{\;2070\text{ K}\;} SiO_2 + 4HCl \qquad (3)$$
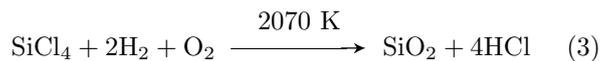
Table 2 gives an overwiew of typical properties of CAB-O-SIL® M-5. Significant characteristics are the high surface area of the fluffy powder and the high melting point of the inorganic silica particles.

Table 2: Properties of CAB-O-SIL® M-5 [3]

B.E.T. surface area (m^2/g)	200
tamped density (g/l)	50
specific gravity (g/cm^3)	2.2
average particle length (10^{-6}m)	0.2...0.3
assay (% SiO_2)	> 99.8
melting point (K)	≈ 1983

The surface of the silica has hydrophilic characteristics and is capable of building hydrogen bonds to create a gel when mixed with a pure liquid due to chemical groups – hydrophilic isolated hydroxyl groups, hydrophilic hydrogen-bonded hydroxyl groups and siloxane groups – which are attached to the surface of the silica during the formation process. Due to hydrogen bonds between single silica aggregates in a dispersed liquid system, increased viscosity and thixotropic behavior is the result. The application of a shear force to the gel weakens the hydrogen bonds between the single silica aggregates and results in a reduced viscosity. However, when the shear stress is removed from the system, hydrogen bonds may redevelop and increase the viscosity of the gel up to its original value before the application of the shear stress, as depicted in Fig. 2. However, viscosity recovery was not detected for the hydrocarbon/silica gels within this study.
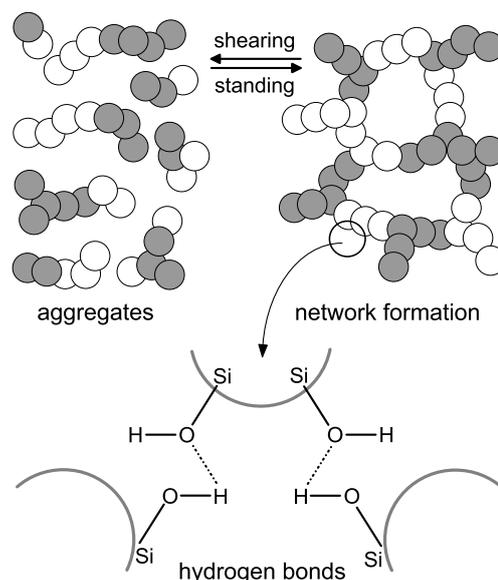
Figure 2: Silica aggregates, hydrogen bonds and network formation (according to [4])

The quality of the gel network between the single aggregates is thereby a function of the following main parameters [4]:

- nature of the pure liquid
- nature and grade of gelling agent
- concentration of gelling agent
- degree of dispersion
- temperature of the system

In combination with non-hydrogen-bond liquids like aliphatics, which show no existence of aromatic rings (e. g. CH_4), and aromatic hydrocarbons and mineral oils, the single silica particels only can connect to each other and not to the liquid part. This results in a best possible gel network and also requires the lowest amount of silica ($3\ldots6\,\%$ by total weight) to obtain desired rheological behavior like increased viscosity and high gel stability. On the other hand, in combination with highly hydrogen-bond-systems like acids, alcoholes and ketones a much higher amount of silica is needed due to undesirable bonds between the silica surface and the liquid system. In terms of a high dispersion of the silica particles within the liquid and the built-up of stable hydrogen bonds, a high shear rate during the gel mixing process and an adequate lenght of shearing time is fundamental, as the comparison of two different mixing types showed significant differences (see section 5.1).

3.2 Hydrocarbon fuels

Two different hydrocarbon fuels were gelled with fumed silica CAB-O-SIL® M-5 for the present investigations, rocket propellant RP-1 and jet propellant JP-8. RP-1 (refined petroleum/rocket propellant) is a rocket propellant, which powers the first-stage boosters of the Delta and Atlas-Centaur rockets, and was also used in the first stage of the Saturn V moon rocket. Due to it's high density in comparison with the high-energetic liquid hydrogen (LH_2), the propellant combination RP-1/liquid oxygen (LOX) is also used in a great many of Russian booster engines (RD-170, RD-180) [17, 18]. JP-8 (jet propellant; NATO code F-34) is the U.S. Air Force primary jet fuel, replacing JP-4 in 1995 because of its less flammable character, better safety and higher combat survivability. Typical properties of the investigated hydrocarbon fuels JP-8 and RP-1 are summarized in Table 3.

Table 3: Fuel properties (at 293 K) [20, 19, 9, 12]

	JP-8	RP-1
gravity (g/cm^3)	$0.755\ldots0.84$	$0.81\ldots0.85$
kin. viscosity (10^{-6}m^2/s)	$< 8^a$	$1.96\ldots2.24$
flash point (K)	min. 310	316
boiling point (K)	$430\ldots573$	420

^{a}at 253 K

4 Measuring system

For the detection of the rheological behavior of the gelled hydrocarbon fuels a rotational rheometer operated in controlled rate mode (CRR) was used. The rotational rheometer imposes strain to the liquid and measures the resulting stress for shear rates up to $1000\,\mathrm{s}^{-1}$. Most common test geometries for rotational rheometers are the parallel plate and the cone-and-plate configuration (see Fig. 3). Whereas the parallel plate offers some advantages like easy sample preparation, the cone-and-plate configuration has been used for the present investigations because it allows a homogeneous strain distribution in the gelled liquid. A temperature control system inside the equipment ensured a constant fluid temperature of $298\,\mathrm{K}$ during all rheological measurements.

(a) parallel plate   (b) cone-and-plate
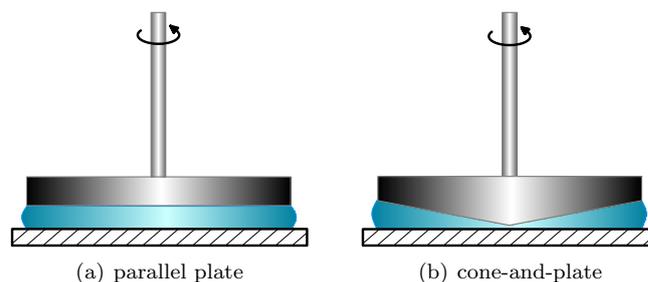
Figure 3: Basic geometries for a rotational rheometer

5 Experimental results

The following section summarizes the experimental results in terms of the gel mixing process, gel quality and stability, as well as a detailed rheological characterization of the gelled hydrocarbon fuels. The mass fraction M describes the gelling agent amount m_{gel} in comparison to the total propellant mass, given by the mixture of liquid m_{liq} and gelling agent m_{gel}:

$$M = \frac{m_{\mathrm{gel}}}{m_{\mathrm{liq}} + m_{\mathrm{gel}}} \tag{4}$$

5.1 Mixing process and gel quality

A comparison between a conventional low-shear and an acoustic high-shear mixing technique (Resodyn ResonantAcoustic® LabRAM) was carried out to determine the influence of the mixing process on the final gel quality and stability. Due to the stiffness of the gel immediately after starting the mixing process, problems arose to keep the liquid in the mixing zone when using a conventional mixing blade or impeller. A high dispersion of the mixture cannot be guaranteed for all mixing conditions. On the other hand, the acoustic mixing uses low-frequency, high-intensity acoustic energy, resulting in a uniform shear level inside the mixing container. For the mixing, the entire system oscillates in resonance, which results in a very high dispersion in the mixing container. Micro-scale turbulences are created inside the medium by the acoustic waves, which gives a very consistent mixing result for highly viscous or even solid materials in

combination with a very short mixing time. Due to accelerations up to 100 g, very high shear rates are applied to the material [6].

Fig. 4 shows the comparison of the high-shear acoustic and low-shear conventional mixing process for JP-8 fuel, using silica concentrations of $M = 4\%$, $M = 5\%$ and $M = 6\%$, respectively.
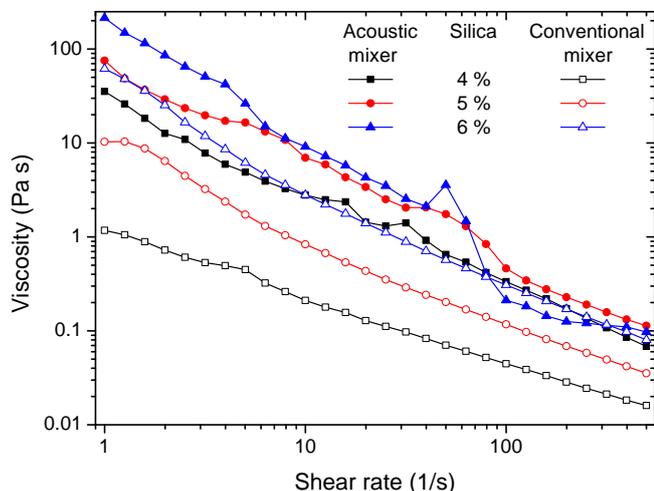
Figure 4: Comparison of high-shear acoustic and conventional mixing process for JP-8 and silica

The different gel mixing parameters for the comparison of the high-shear acoustic and low-shear conventional mixing technique, depicted in Fig. 4 in terms of gel viscosity vs. applied shear rate, are summarized in Table 4. The much lower gel mixing time with the acoustic mixing technique is remarkable.

Table 4: Gel mixing process

	acoustic	conventional
gel composition	JP-8 / silica	JP-8 / silica
mixing time (s)	240	≈ 4000
rotating speed (1/s)	-	≈ 10
resonance frequency (Hz)	61.3	-

Especially for low shear rates $1 \dots 10\,\mathrm{s}^{-1}$ the differences in measured viscosity of gels prepared with the acoustic and conventional mixing technique are significant with a more than one order of magnitude higher viscosity for the sample mixed with the acoustic mixer and for $M = 4\%$, $M = 5\%$ and $M = 6\%$, respectively. For higher shear rates up to $1000\,\mathrm{s}^{-1}$, viscosity differences between the two mixing processes still exist, even though are less significant. The difference in viscosity is due to the high degree of dispersion that the acoustic mixer provides. The viscosity of the gels is significantly reduced once the hydrogen bonds between the silica aggregates is broken. Due to the convincing results, all following gels within this study were produced by the acoustic mixing process.

A typical mixing sequence using the acoustic mixer is pictured in Fig. 5. Accelerations of approximately 50 g and 100 g, respectively, and appropriate resonance frequencies for different propellant mixing processes with varying mixing times in the range of $15 \dots 500\,\mathrm{s}$ for JP-8 and a silica concentration $M = 5\%$ are depicted. Acceleration and resonance frequency attain constant values after a very short mixing time of approximately $10 \dots 15\,\mathrm{s}$. For the lower acceleration of 50 g a slightly lower resonance frequency of $\approx 61\,\mathrm{Hz}$ was found in comparison to the 100 g mixing processes with a frequency of $\approx 61.25\,\mathrm{Hz}$ [2].
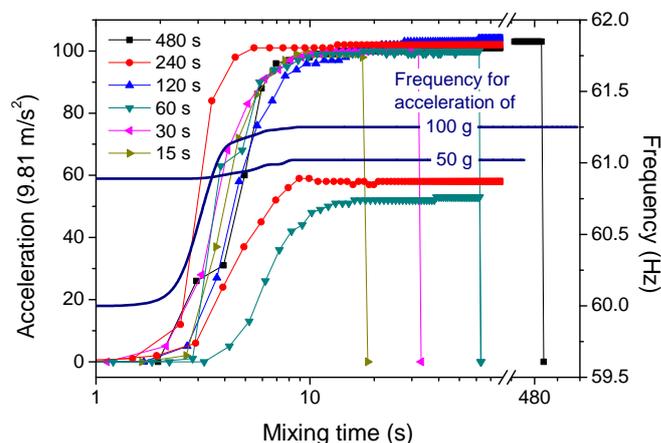
Figure 5: Gel mixing process with acoustic mixing technique (JP-8 with $M = 5\%$ silica)

5.2 Gel quality as a function of mixing time

The influence of the mixing time on the gel quality is significant especially for gels using inorganic gelling agents like fumed silica. Based on the results of section 5.1, JP-8 with a concentration of 5% silica was chosen to investigate the influence of the mixing time on the quality of the gel. Fig. 6 shows the gel viscosity for mixing times in the range from 8 s up to 960 s.

As can be seen very clearly, a higher mixing time does not come along with a higher viscosity of the mixed gel. In contrast however, a shorter mixing time seems to improve the quality of the gel in terms of viscosity, not only for low shear rates in the range of $1 \dots 10\,\mathrm{s}^{-1}$ (see Fig. 6 (a)), but also fewer distinctively for higher shear rates up to $1000\,\mathrm{s}^{-1}$ (see Fig. 6 (b)). Especially for low shear rates, a significant decrease in viscosity can be seen for mixing times exceeding approximately 80 s. Due to very high accelerations during the gel mixing process with the acoustic mixing technique (see Fig. 5) the loss in viscosity for higher mixing times can be caused by interactions of the liquid with the hydrogen bonds of the silica particles [4], which affects and weakens the hydrogen bonds between the single silica particles, as depicted in Fig. 2. Due to the high shear rates due to the high mixing accelerations once the gel is formed, the mixing itself acts like a centrifuge. After the silica network is established, the accelerations of the acoustic mixing system result in a partial breaking of the hydrogen bonds between the silica agglomerates and a reduced viscosity. In comparison however, with a reduced mixing acceleration when using the acoustic mixing tech-
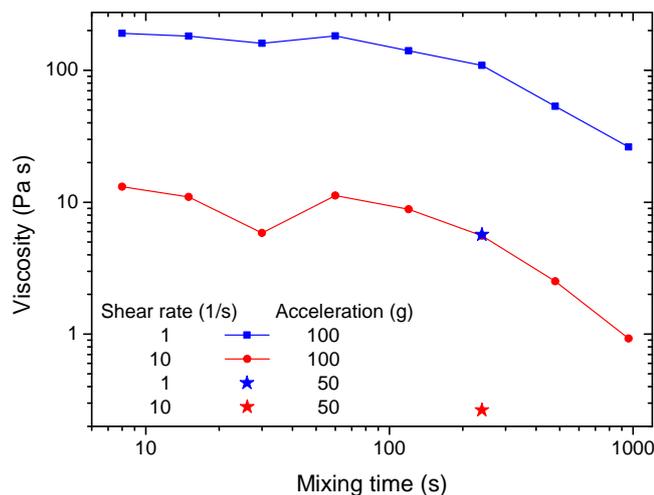
(a) Low shear rate
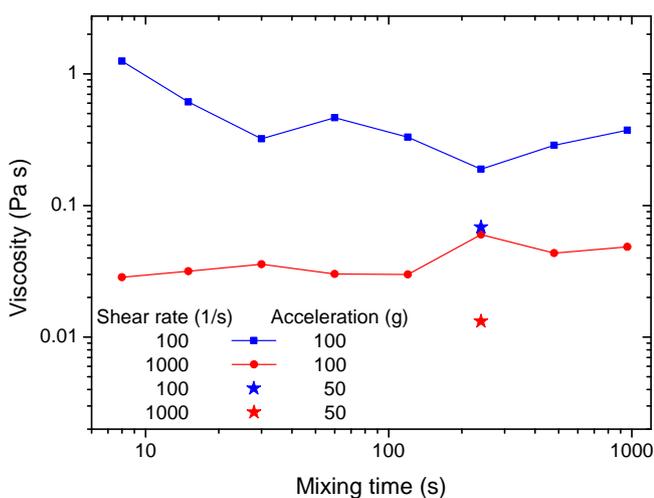
(b) High shear rate

Figure 6: Gel viscosity as a function of mixing time and mixing acceleration (JP-8 with $M = 5\%$ silica)

Figure 7: Gel temperature (JP-8 with $M = 5\%$ silica)
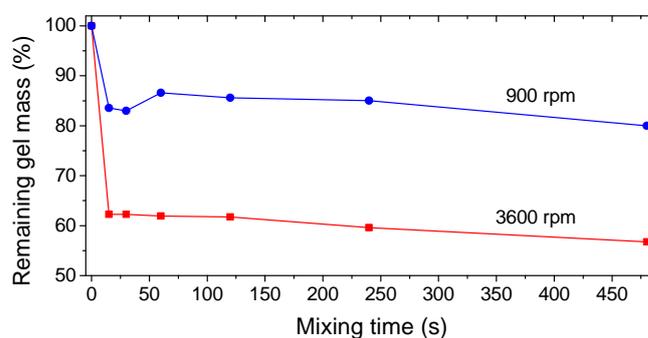
Figure 8: Gel stability as a function of mixing time (JP-8 with $M = 5\%$ silica)

nique of only $50\,\mathrm{g}$ instead of $100\,\mathrm{g}$, it was not possible to produce a gel with similar high viscosities even for a much longer mixing time (see Fig. 6 (a) and Fig. 6 (b)) [2].

Since heating-up of the liquid inside the mixing container due the high-shear mixing process may also result in the measured viscosity reduction for higher mixing times, a thermocouple has been used to measure the gel temperature during the mixing process. The gel temperature during mixing is depicted in Fig. 7. Due to effectively convective cooling however, the temperature increase of the gel inside the mixing container can be neglected even for longer mixing times up to $800\,\mathrm{s}$.

The gel mixing parameters not only influence the resulting viscosity of the gel. In order to characterize the stability of the gel as a function of mixing time, centrifuge tests were conducted with JP-8 and a silica amount of 5%, applying two constant accelerations of approximately $1500\,\mathrm{g}$ ($3600\,\mathrm{rpm}$) and $90\,\mathrm{g}$ ($900\,\mathrm{rpm}$) for a time of $600\,\mathrm{s}$ in each case (see Fig. 8). It is a remarkable result to state that the rheological properties of the gel change significantly

for different mixing times as shown in Fig. 6, however this behavior is not related to the phase separation in terms of gel stability (see Fig. 8). The influence of the mixing time on the stability of the gel seems to be almost negligible with only a slight decrease of the remaining gel mass for a higher mixing time.

JP-8 and RP-1 gels with $M = 4\%$ and $M = 7\%$, respectively, are depicted in Fig. 9. The much stiffer characteristics and the solid-like behavior of the gels featuring a higher gelling agent amount is clearly visible, whereas gels with lower silica concentration are more liquid-like.

Based on these results, a gel mixing time of $60\,\mathrm{s}$ and a mixing acceleration of approximately $100\,\mathrm{g}$ have been chosen for the present study to assure a preferably and reproducible gel quality. These mixing parameters guarantee both, a sufficiently long high-shear mixing time to form the hydrogen bonds and give highest possible gel viscosity, and a minimum negative centrifuge effect due to the mixing itself.

5.3 Rheological characterization

After describing the gel mixing process in section 5.1 and the determination of the optimum gel mixing time and acceleration to obtain a best possible mixed gel in terms of viscosity and stability in section 5.2, the following section will focus into the rheological behaviour of the gelled

(a) JP-8, $M = 4\,\%$ (b) JP-8, $M = 7\,\%$

(c) RP-1, $M = 4\,\%$ (d) RP-1, $M = 7\,\%$
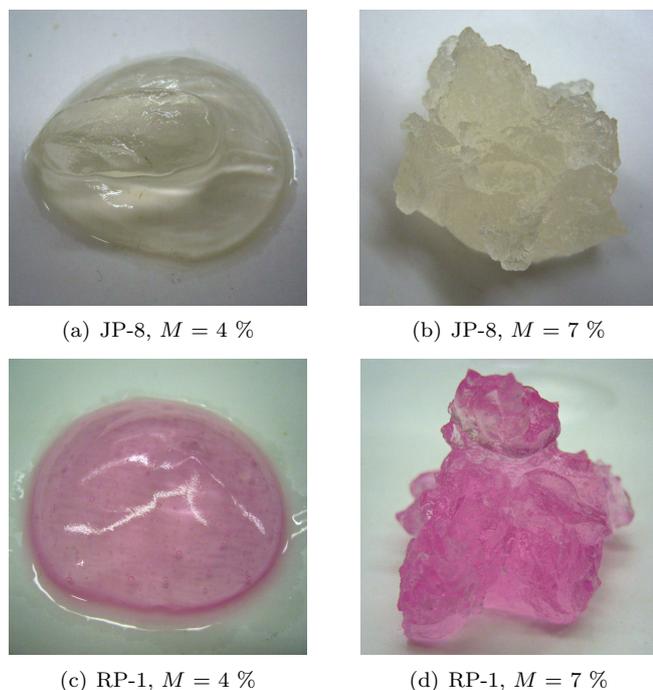
Figure 9: Hydrocarbon/silica gels

hydrocarbons like viscosity η vs. applied shear rate $\dot{\gamma}$ and gel stability as a function of silica amount. JP-8 and RP-1 gels featuring a gelling agent amount M of $4\,\%$, $5\,\%$, $6\,\%$, and $7\,\%$, respectively, were considered [2].

5.3.1 Gel viscosity

Fig. 10 (a) and Fig. 10 (b) depict the comparison of gel viscosities of RP-1 and JP-8, respectively, for shear rates in the range from $1\,\mathrm{s}^{-1}$ to $1000\,\mathrm{s}^{-1}$ as a function of the added silica amount M. A significant increase of the viscosity for both hydrocarbon fuels can be stated with an increasing gelling agent concentration. The influence of the fumed silica is not only visible for low shear rates but remains noticeable but less significantly at higher shear rates. This can be explained by the higher number of carbon atoms in the RP-1 atomic composition (approximately $C_{12}H_{23.4}$) in comparison to JP-8 with an approximately formula of $C_{11}H_{21}$ [7, 12].

5.3.2 Dynamic viscoelastic characterization

In order to investigate the viscoelastic characteristics of JP-8 and RP-1 gels, the storage (G') and loss (G'') moduli were determined for two silica concentrations. In practice, those properties are useful since they describe a gel in terms of strength and weakness. The strain dependence of the storage moduli G' for JP-8 and RP-1 gels with a silica concentration $M = 5\,\%$ and $M = 6\,\%$ at a temperature of $298\,\mathrm{K}$ and a frequency of $5\,\mathrm{Hz}$ can be observed in Fig. 11.

As can be seen in Fig. 11, for both concentrations and hydrocarbon fuels the storage modulus G' remained con-
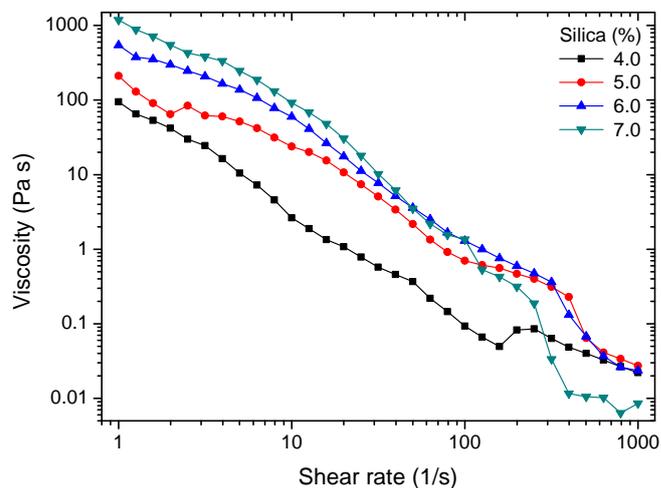
(a) Rocket propellant RP-1
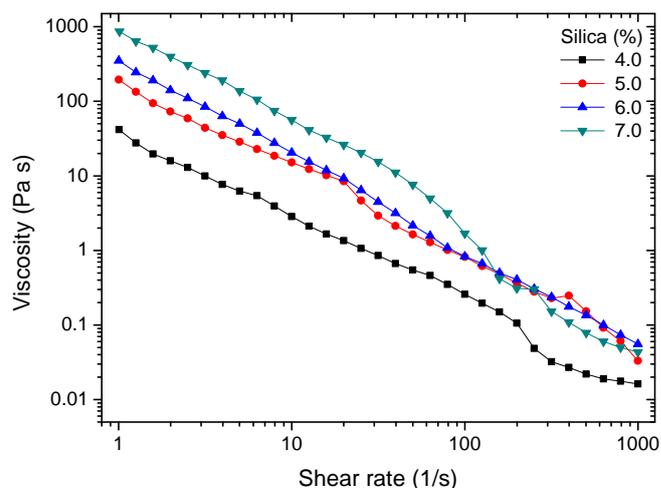
(b) Jet propellant JP-8

Figure 10: Gel viscosity as a function of silica amount

stant at low amplitude deformations. In addition, the effect of silica concentration on the magnitude of storage modulus G' can be clearly observed. Independent on the fuel, gels with $6\,\%$ silica exhibited higher values of storage modulus than the $5\,\%$ gels. Since the strength of a gel can be characterized by its storage modulus in the linear viscoelastic regime, gels with $6\,\%$ silica exhibited also higher strength.

Comparing the RP-1 and JP-8 gels with identical silica concentrations, the strength of RP-1 gels is at least twice as high as the JP-8 gel strength. As presented in section 5.3.1, higher viscosity values for RP-1 gels were also observed when compared to JP-8 gels with the same silica concentration (see Fig. 10).

The range in which the storage modulus G' is not dependent on the strain applied characterizes its linear viscoelastic region. The strain value in which this dependency shows up is denominated as critical deformation value. For JP-8 and RP-1 gels they were around $2 \dots 3\,\%$ and $3 \dots 4\,\%$, respectively.

Moreover, the G' and G'' frequency dependency was investigated, as depicted in Fig. 12. The frequency sweep
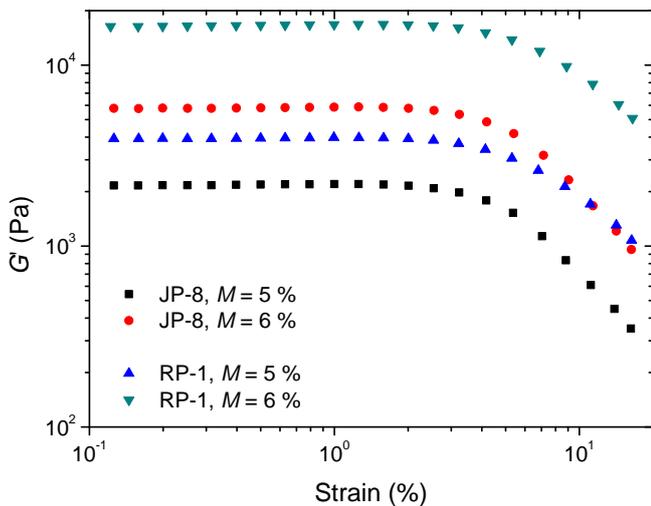
Figure 11: Strain sweep tests for JP-8 and RP-1 gels with 5 % and 6 % silica at 5 Hz and 298 K
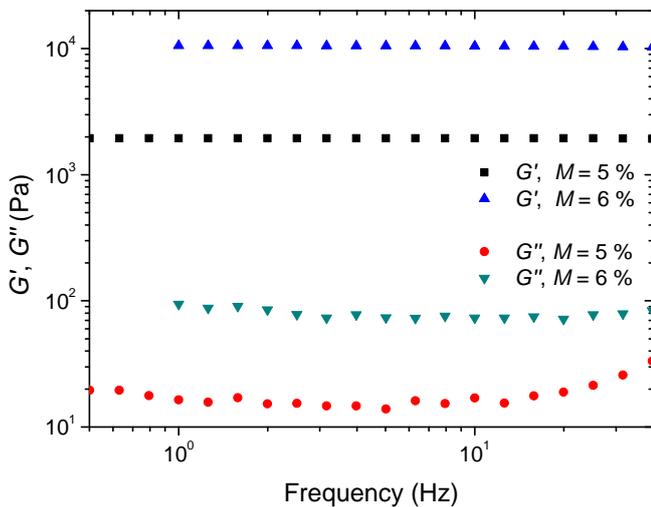
(a) JP-8 gels
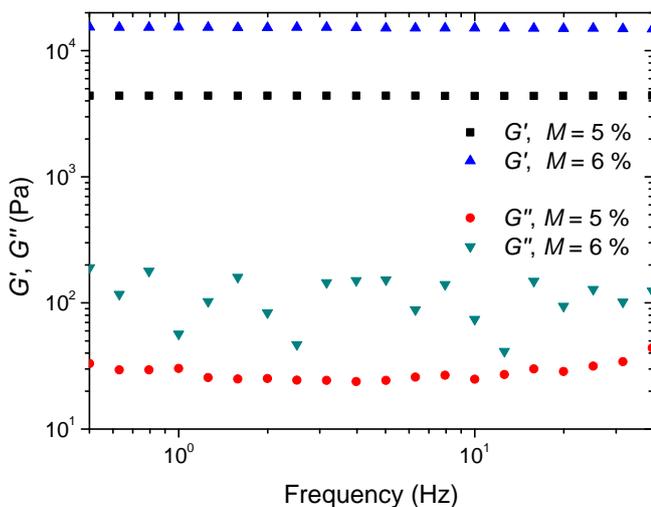
(b) RP-1 gels

Figure 12: Frequency dependency of storage and loss moduli

tests were carried out in the linear viscoelastic region, applying 1 % strain at a temperature of 298 K. The results show that for both concentrations and fuels, the storage modulus G' is significantly higher than the loss modulus G'', which indicates that the gel exhibits a solid-like behavior in the investigated frequency range. Fig. 12 also shows that both storage and loss moduli are mostly independent of frequency over the range studied, which characterizes the behavior of solid gels.

5.3.3   Gel stability

To simulate accelerations and vibrations during a typical rocket launch, which can cause gel separation in the tank and the propellant feed system, centrifuge tests have been conducted. The gel stability as a function of the silica amount M is illustrated in Fig. 13 for a centrifuge operating time of 600 s. After the centrifuge tests, the
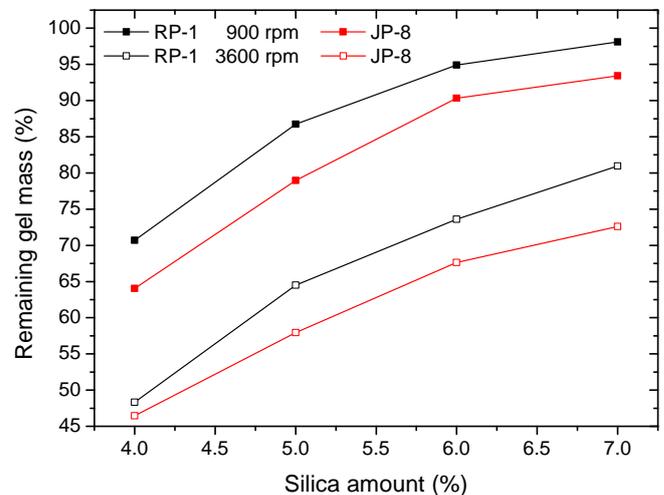
Figure 13: Gel stability as a function of silica amount

separated liquid due to the acceleration was compared with the initial propellant mass to describe the stability of the investigated gels. Fig. 13 indicates, that the 6 % and 7 % gels are quite stable for accelerations of approximately 90 g (900 rpm), whereas for accelerations of approximately 1500 g (3600 rpm) only 70 . . . 80 % of the initial gel mass is remaining. Silica amounts of less than 6 % did not show a stable behaviour during this investigations even for the 90 g accelerations. However, the RP-1 gel seems to be more stable than the JP-8 gel within in the investigated range of added gelling agent and applied accelerations. These results are consistent with the investigated gel viscosity in section 5.3.1, where the measurements have shown a slightly higher viscosity for the RP-1 gel in comparison to the JP-8 gel due to the above mentioned higher amount of carbons.

6   Conclusions and future work

Within the present study, two gelled hydrocarbon fuels, JP-8 and RP-1, have been investigated experimentally. Fumed silica was used as a gelling agent to gel the hydrocarbons. A high-shear acoustic mixing technology was

applied to produce the gels. The influence of the mixing time and the mixing acceleration turned out to be major parameters for the resulting gel quality, whereas a relatively short mixing time in combination with high accelerations resulted in gels with the highest viscosity and stability. The amount of the added gelling agent showed significant influence on the viscosity and stability of the gels for low as well as high shear rates. Future investigations at Purdue University not only will implicate gelled hydrocarbon fuels like JP-8, JP-10 and RP-1, but also consider the rheological behavior of rocket engine fuel MMH (monomethylhydrazine, $CH_3N_2H_3$), using organic cellulose-based gelling agents like hydroxypropylcellulose (HPC) and hydroxyethylcellulose (HEC) [1].

Acknowledgement

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