Development of Paraffin-Based Fuels for a Vortex Combustion Hybrid Rocket Engine

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Abstract

This work focuses on the implementation of paraffin-based fuels featuring relatively fast regression rates and suitable mechanical properties. The investigated formulations are developed for the future testing in a non-conventional hybrid engine configuration, the vortex flow pancake (VFP). In the fuels, the paraffin wax is blended with a styrene-based thermoplastic copolymer (styrene-ethylene-butylene-styrene grafted with maleic anhydride, SEBS-MA). A wide pre-burning characterization of the fuels is perfomed: it includes thermal, rheological, and mechanical behaviour of wax and of the reinforced blends based on it. The microcrystalline paraffin wax (SW) is the base ingredient of the fuels, while liquid paraffin (LW) is used as additive in some of the tested formulations. Thermal characterization shows that the melting temperatures of the proposed blends is close to the one of paraffin, independently of the SEBS-MA (and LW) addition. From the rheological analyses it is observed that (i) higher mechanical properties at higher temperatures are afforded by the SEBS-MA-blends, (ii) the viscosity value at the same shear rate and temperature increased by increasing the mass fraction of reinforcing polymer, though a reduction is possible by adding LW to the formulation. Tensile tests prove that increasing the mass fraction of SEBS-MA enhances formulation stiffness and ductility. In fact, with respect to W1 fuel, the formulations with 10 wt.% of SEBS-MA features a yield stress increase of 50%, while the yield strain is increased by 70%. Using 5% of LW as additive in a fuel blend with 20 wt.% SEBS-MA yields an increase of 25% in elongation with respect to the formulation without the liquid paraffin.

1. Introduction

Hybrid rocket engines (HREs) offer attractive features for both launch and in-space applications in light of their high impulse, thrust throttling, simplicity, low cost and safety. On the other hand, HREs have two main drawbacks: (i) slow regression rate, and (ii) low combustion efficiency. Paraffin-based fuels feature a combustion mechanism overcoming the slow regression rate of conventional fuels. Vortex combustion offer efficient combustion promoting intense propellant mixing. However, the paraffin waxes alone are not suitable for withstanding loads associated to hybrid rockets operations. Thus, the research activity on paraffin-based fuels aims at providing new formulations with a suitable set of mechanical and ballistic properties.

Several researchers have investigated different strategies to enhance the mechanical properties of paraffin wax fuel grains. Blends of paraffin wax with the addition of low-density polyethylene (LDPE) was studied in Ref. [1, 2] . Tensile tests showed an increase in the ultimate tensile strength and elongation of blended formulations compared to pure paraffin. Kobald et al. [3] analysed the characteristics of paraffin wax (Sasol 6805) with three different additives: stearic acid (SA), nanoclay and a common polymer. The results of the tensile tests reported a maximum tensile strength for the blends with polymer with a 200% increase with respect to pure wax. The addition of nanoclay doubled the elongation at break without improving the tensile strength. In Ref. [4] different additives were tested: SA, polyethylene wax (A-C®6A), ethylene vinyl acetate copolymer (EVA), LDPE, polypropylene (PP) and high density polyethylene (HDPE). Compressive and tensile tests were performed for all the formulations. All the additives improved the mechanical properties of the paraffin wax. The best at improving the compression strength was A-C®6A. LDPE brought the highest increases in tensile strength. Structural properties of pure and 40% aluminised Sasol Wax 0907 were investigated by Veale et al. [5]. The aluminised formulations showed a higher ultimate tensile stress and elastic modulus, while the elongation resulted similar in all the tested conditions.

Bisin et al. [6] compared the mechanical reinforcement of paraffin-based fuels with that of an innovative methodology: the embedment of 3D-printed cellular structures inside the paraffin-based fuel grain. Tested formulations were paraffin wax (Sasol Wax 0907) blended with 5 wt% and 10 wt% styrene-ethylene-butylene-styrene grafted with maleic anhydride (SEBS-MA) copolymer, as well as armored grains featuring 3D-printed gyroids. Three different thermoplastic polymers were used to print the gyroids: polylactic acid (PLA), Acrylonitrile Butadiene Styrene (ABS) and nylon (NY). Blended formulations showed an increase in Young's modulus, yield stress, and yield strain with respect to the pure wax formulation. All the blended formulations still featured a brittle failure mechanism. Armored grains showed an increase in the compression yield stress and strain compared to pure paraffin. The main achievement with armored grains was in the obtainment of a ductile behaviour, in contrast with the brittleness of pure paraffin grains.

This study focuses on in-space propulsion applications. A non-conventional motor configuration, the vortex flow pancake (VFP), burning paraffin-based fuel formulations is considered. The paraffin wax is blended with SEBS-MA. This work is done based on previous research on SPLab [7, 8] in which there is a direct relation between the viscosity of the fuels and the regression rate. Using thermos plastic polymers enhances the mechanical performance of the fuel blends but reduces the regression rate. In this study, liquid paraffin (LW) is being added to the blends with a small mass fraction with respect to the wax keeping the mass percentage of thermoplastic polymer constant. The aim of the LW addition is an enhancement of blended fuel mechanical properties, with a reduction of its rigidity and brittleness.

2. Paraffin-based Blends

In this study, different fuel formulations are tested considering a commercial microcrystalline paraffin (SASOL 0907, SW). The baseline for the study is a reinforced paraffin-based fuel where the micro-crystalline wax is blended with SEBS-MA. The investigated fuel formulations are designed to provide improved mechanical properties with tailored regression rates. According to previous studies performed on conventional engine configuration at lab and intermediate scale [7, 9] The improvement of mechanical properties by SEBS-MA implies a reduction of the ballistic response of the fuel. Thus, to tailor the fuel regression rate while granting suitable mechanical properties, different co-polymer loads were investigated together with various paraffin taken as additives in the SW+SEBS-MA blends. A liquid paraffin (LW) was identified as a suitable additive in the SEBS-MA blends in the light of its possible plasticizing action. An overview of the tested formulations is given in Table 3.

2.1 Materials

SW is a micro-crystalline wax produced by Sasol [10], it has good properties in terms of (low) melt layer viscosity and mechanical properties. The micro-crystalline paraffin waxes are formed by monoclinic crystals which gives their plastic and malleable behaviour, they are also characterized by higher molecular weights, densities and refractive indices when compared to the macro-crystalline ones. Its properties are reported in Table 1.

LW is a liquid paraffin wax, produced by ThermoFisher [11] (Alfa Aesar Nujol). Its properties are reported in Table 1.

Table 1: Paraffin properties [10, 11].

Paraffin Type	Density [kg/m^3]	Congealing Point [°C]	Flash Point [°C]	Boiling Point [°C]
SW	924	83-94	200-240	>370
LW	838	-20	220	>300

SEBS-MA is a commercial thermoplastic elastomer supplied by Sigma-Aldrich [12]. The copolymer contains 30 wt.% of styrene, and it is grafted with 2 wt.% of maleic anhydride (MA), linked to the butylene blocks. The styrene blocks at the extremes of the macromolecule provide the material with thermoplastic behaviour. While the central ethylene-butylene block provides rubber-like properties to the polymer. The material features high tensile strength and ultimate elongation as well as relatively high melt viscosity [12]. Physical properties of SEBS-MA are reported in Table **2**.

Table 2: Physical properties SEBS-MA [12, 13]

Property	Value
Density, [kg/m^3]	910
Melt Index, [g/min] (@230 °C)	21
Ultimate Elongation, [%]	500
Molecular Weight, [g/mol]	9100

Carbon powder (**CP**) is supplied by Sigma-Aldrich in micron-sized powder. The material is characterized by particles size < 20 μm and density equal to 2100 kg/m^3 [12]. The additive was used in order to give to all the blends a black color and therefore enhance the grain surface absorption of radiative heat transfer from the flame [14] and to avoid deep penetration of radiation into the solid fuel.

2.2 Investigated Formulations

Investigated SEBS-MA mass fractions range from 0% to 20%, while, when considering the paraffin contained in the fuel formulations, LW content goes from 0 wt.% to 10 wt.%. As shown in Table **3**, all the tested fuels feature a 1 wt.% CP used as grain opacifier.

Table 3: Proposed paraffin-based formulations

Formulation ID	Composition wt. %				TMD, kg/m^3
	SW	LW	SEBS-MA	CP	
SW	99	0	0	1	929
SWS10	89	0	10	1	928
SWS20	79	0	20	1	926
SW90LW10S10	80	9	10	1	919
SW95LW5S10	84.5	4.5	10	1	923
SW97.5LW2.5S10	86.8	2.2	10	1	926
SW90LW10S20	71	8	20	1	936
SW95LW5S20	75	4	20	1	939
SW97.5LW2.5S20	77	2	20	1	941

3. Experimental Setups and Methods

Three kinds of experimental investigations were conducted to study the thermal, rheological, and mechanical properties of paraffin-wax based blends doped with thermoplastic polymers in order to evaluate a formulation which best meets the requirements to be used as a solid fuel for hybrid rocket engines.

3.1 Thermal Analysis (Differential Scanning Calorimetry-Thermogravimetry)

Understanding the thermal properties (melting and solidification temperatures and latent heats) of solid fuel grains is essential to control the manufacture of the grains. The differential scanning calorimetry (DSC) is widely used to determine the properties of waxes. The heat flow absorbed/released from the tested specimen under constant heating rate is measured. Properties that can be measured are glass transition temperature, melting temperature, the heat of fusion, onset temperature, and end temperature. The Netzsch STA 449 F5 Jupiter unit allows the simultaneous execution of DSC and thermogravimetry (TG).

In a DSC, the difference in heat flow to the sample and a reference at the same temperature, is recorded as a function of temperature. The reference is inert under the tested conditions and exhibits no phase changes. The calorimeter consists of a sample holder and a reference holder as shown in Figure 1. Both are constructed to allow high temperature operation. Under each holder there is a resistance heater and a heat-flux sensor. Currents are applied to the two heaters to increase the temperature at the selected rate.

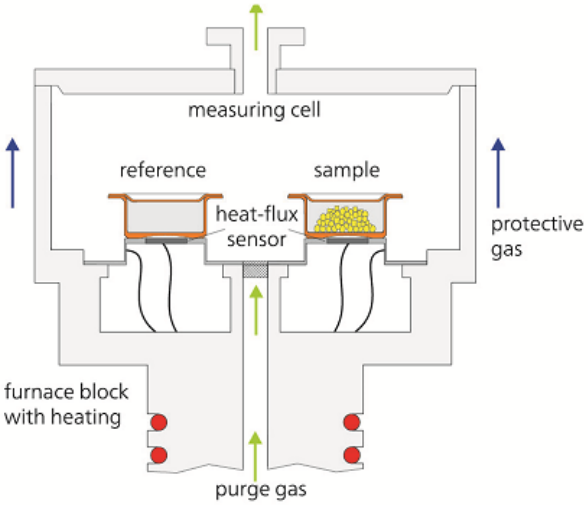

Figure 1 Differential scanning calorimetry (DSC) [15]

3.2 Rheological Characterization

This test was done to investigate the materials response to deformation energy, shear stress, and to study the viscoelastic behaviour of the materials with respect to temperature. These properties can be measured simply with a rotational rheometer. The Rotational rheometer is a high-precision, continuously variable shear instruments in which the test fluid is sheared between rotating cylinders, cones, or plates, under controlled-stress or controlled-rate conditions. Most rheometers depend on the relative rotation about a common axis of one of three tool geometries: Couette apparatus, cone and plate, or parallel plates, as shown in Figure 2.

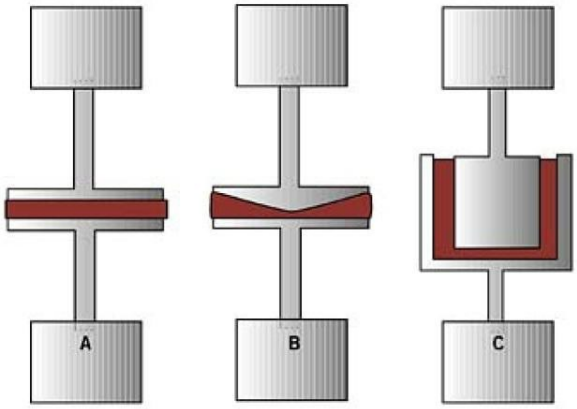

Figure 2 Basic tool geometries for the rotational rheometer:

(a) parallel plate, (b) cone and plate, (c). Couette apparatus [16]

In this study, all the experiments were carried out with a Rheometrics Dynamic Analyzer RDA II to obtain the viscoelastic behaviour of the investigated formulations. A Couette configuration was used for viscosity measurements of melted materials by a steady test. A steady test using Couette is usually performed in controlled-rate mode with the shear rate ranging logarithmically between 10 and 1000 s^{-1}. The used test temperature values ranged between 90 °C and 160°C. While a parallel plate geometry was used to measure the storage modulus of investigated blends though a dynamic test. A dynamic test was performed with plate diameters of 25 mm and sample initial thickness between 2.1 mm and 2.9 mm. A constant strain of 1% has been applied, associated to a shear rate sweep from 0.5 Hz to 50 Hz and imposed temperature sweep from 30°C up to the melting phase with an increment step of 3°C.

3.3 Mechanical Characterization (Tensile tests)

Tensile testing was performed according to the standard ISO 527 for plastic materials [17, 18] using Instron 4302 machine. The machine was equipped with a 1 kN load cell. All tests were performed at normal temperature range (24 ± 1 °C) at a pulling rate of 1 mm/min. This speed was chosen to be consistent with the tensile tests previously performed at the SPLab so to enable the possibility for the comparison of tensile properties of the different formulations tested. The characterization tests were conducted on samples with dog bone geometry with a thickness of 10 mm, compliant with ISO 527-2. Tests were performed in a close environment, under controlled conditions limiting possible issues related to temperature variation of the samples and of the environment.

4. Results and Discussion

This section discusses the results of the experimental analyses. First, thermal properties of paraffin-based blends are discussed. Second, rheological characteristics are studied for both viscosity and storage modulus. Finally, the mechanical tensile characteristics are presented.

4.1 Thermal analysis results

Table **4** shows the output of the DSC_TG runs summarizing the relevant parameters. All the blends have melting points temperature ($\boldsymbol{T_m}$) very close to paraffin, while the melting point of pure SEBS is higher than 340°C. The absence of changes in the melting points is due to the absence of chemical interactions between SEBS-MA and the wax: the components of the fuel re simply mixed, thus their individual behaviour is not significantly changed. On the other hand, increasing the mass fraction of SEBS-MA in the blends increases the degradation onset temperature ($\boldsymbol{T_{on}}$) of the blend, while the degradation end temperature ($\boldsymbol{T_{end}}$) remains almost the same. Increasing the mass fraction of LW inside the blends with different mass fraction of SEBS-MA leads to a decrease in the melting temperature of the fuel. In addition to this, for the LW-doped fuels a decrease on the onset temperature of the blend is noted, while the end temperature remains almost the same. The early onset and melting peak temperature after LW addition are likely due to the presence of light hydrocarbon fractions with small thermal stability.

Table 4: Thermal parameters obtained from DSC-TG measurements

Fuel Id.	T_m [°C]	T_{on} [°C]	T_{end} [°C]
SW	74	347.8	473.9
SEBS	344.4	418.9	467.1
SWS10	75.7	385.4	473.6
SWS20	74.5	391.5	472.2
SW90LW10S10	71.4	341.8	461.2
SW95LW5S10	73.2	358.7	462.8
SW97.5LW2.5S10	75.4	369.8	465.3
SW90LW10S20	67.6	373.5	466.5
SW95LW5S20	69.5	380.3	467.2
SW97.5LW2.5S20	70.1	385.3	467.8

4.2 Rheological results

Several observations obtained from rheological investigations will be discussed in the following two subsections.

Storage Modulus

Figure 3 shows how the SEBS-MA can delay the degradation of the mechanical properties of the blend as the temperature increases. SW show a decrease in the tangential modulus (G') due to the melting of the paraffin (70°C). The formulations with 10 wt.% and 20 wt.% SEBS-MA show a degradation of the mechanical properties at temperature above 70°C. However, the increase of mass fraction of SEBS-MA in the blend does not show any positive effect on maintain the mechanical properties of the fuel at higher temperatures. Under the investigated conditions, no significant difference is noticed when contrasting the $G'(T)$ for SWS10 and SWS20. This suggests that polymer load above 10%

can be beneficial for some of the mechanical characteristics of the fuel, though, given the thermal analysis results, it is not providing further thermal stability. Also, adding a small fraction of LW to the blend with different mass fraction of SEBS-MA does not affect the deformation temperature of the blends as shown in Figure 4 and Figure 5.

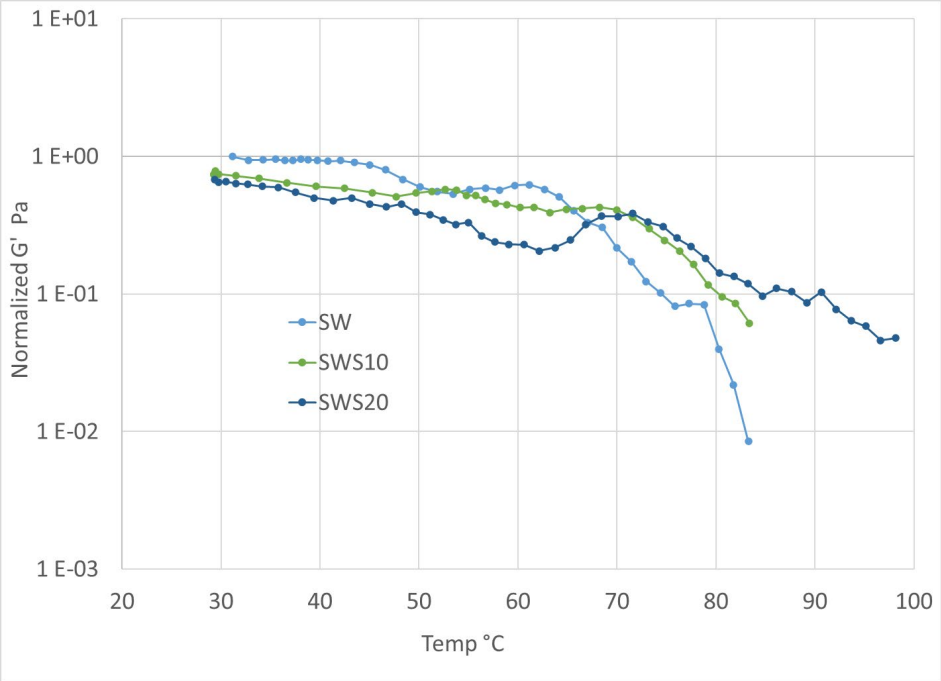

Figure 3 Storage modulus for different SEBS-MA fraction normalized to SW values

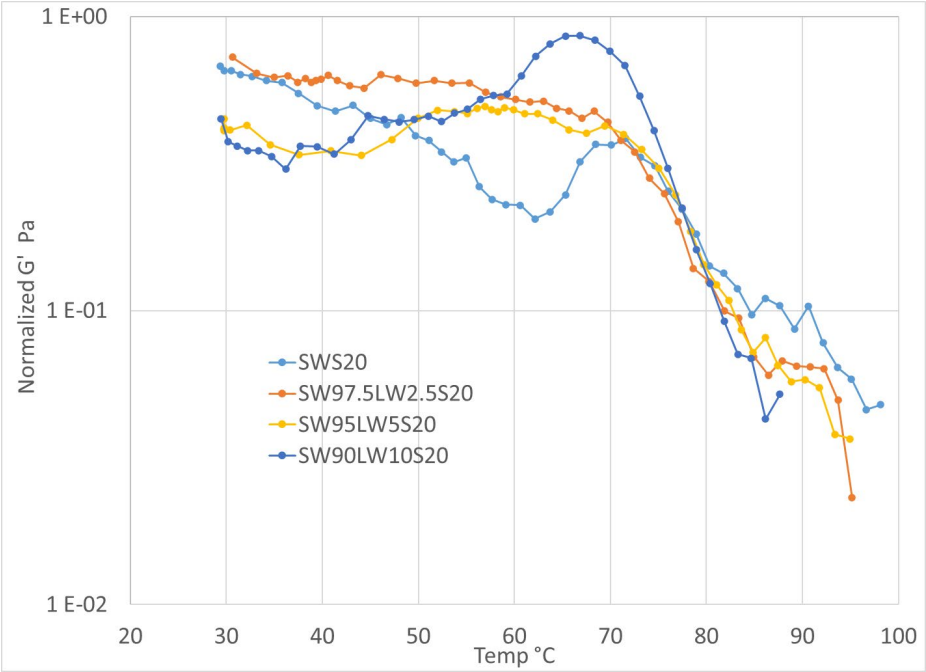

Figure 4 Storage modulus for different LW fractions with 20% SEBS-MA normalized to SW values.

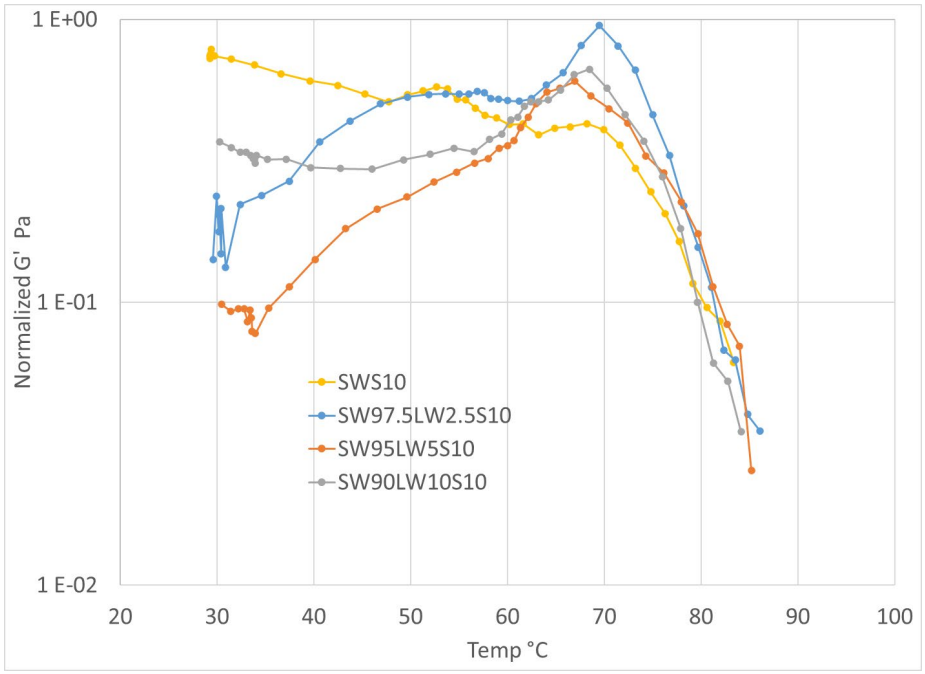

Figure 5 Storage modulus for different LW fraction with 10% SEBS-MA normalized to SW values.

Viscosity

Viscosity measurement is crucial in the case of developing new fuel blends for hybrid rocket engines. Regression rate of the fuel has a strong relation with the melt formulation viscosity value [3, 19-21]. As the viscosity decreases the regression rate should increase. As indicated in Table **5**, the viscosity value at the same shear rate and temperature decreases by increasing the mass fraction of SEBS-MA.

Figure 6 shows a comparison of the effects obtained by loading LW into the 79 wt.% of micro-crystalline paraffin of the blends, always maintaining a 20 wt.% SEBS-MA. Data are evaluated at the temperature of 130 °C. The trends mainly show a reduction in viscosity compared to the fuels with SW alone, as the mass fraction of LW increases, however for small mass fraction of LW (2.5-5 wt.%), the change in viscosity is faint, meanwhile for 10 wt.% LW there is a great decrease of the viscosity (suggesting a possible increment of the entrainment mass transfer and, therefore, on the fuel regression rate). This is also highlighted by the results of Table **5**.

Figure 7 shows the comparison between the SWS10 and the LW-loaded counterparts. The data for the fuels loaded with 10 wt.% of SEBS-MA are evaluated at 110°C, and not at 130°C as in the case with 20 wt.% SEBS-MA, due to hardware sensitivity limitations. Data for SWS10 show smaller values of viscosity than the 20 wt.% SEBS-MA counterpart. This makes the SWS10 an attractive candidate from the entrainment point of view. Small mass fractions of LW in the paraffin binder promote a viscosity decrease that, while negligible (if any), for 2.5 wt.% and 5 wt.%, is pretty high at 10 wt.% (see Table **5**).

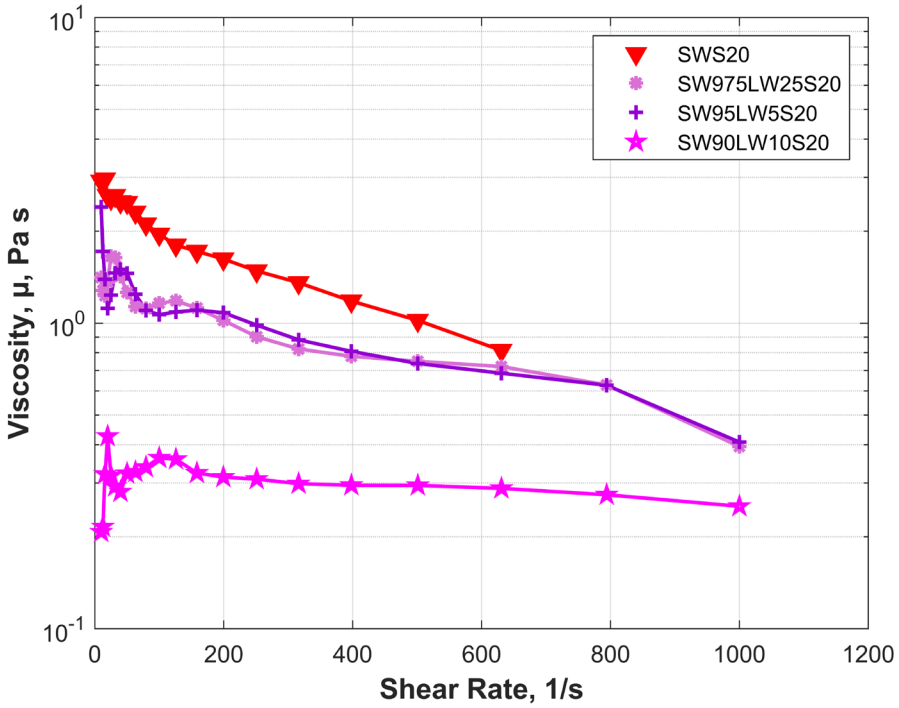

Figure 6 Viscosity for different LW fraction with 20% SEBS-MA

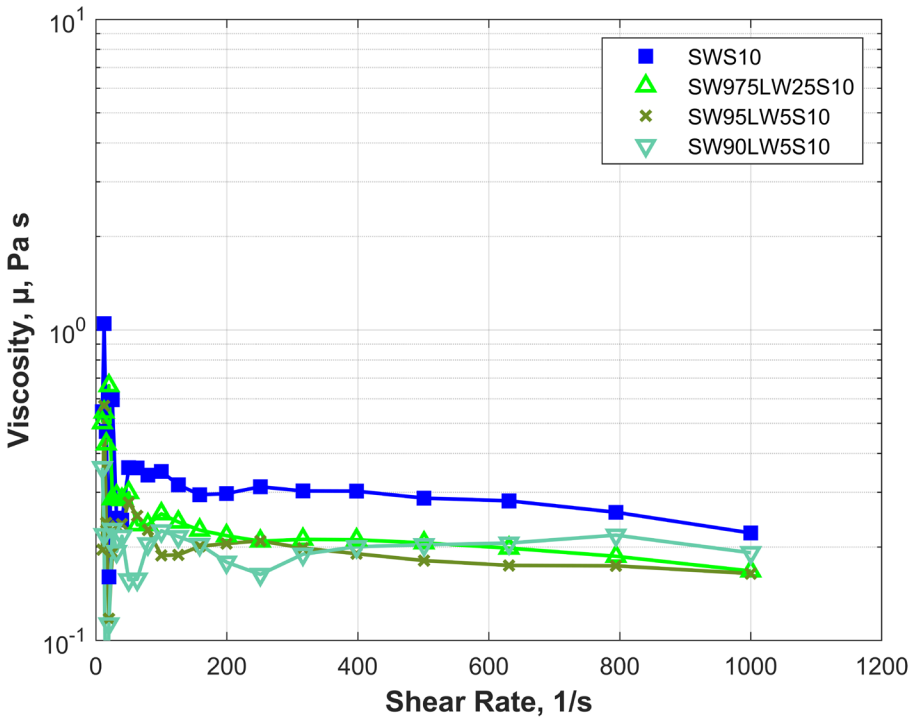

Figure 7 Viscosity for different LW fraction with 10% SEBS-MA.

Table 5 Viscosity values for different paraffin-SEBS-MA blends.

Formulation ID	Viscosity [Pa s]	Shear rate [1/s]	Temperature [°C]
SW	0.004	1000	130
SWS10	0.055	500	110
SWS20	1.021	500	130
SW97.5LW2.5S10	0.206	500	110
SW95LW5S10	0.181	500	110
SW90LW10S10	0.203	500	110
SW97.5LW2.5S20	0.750	500	130
SW95LW5S20	0.738	500	130
SW90LW10S20	0.295	500	130

4.3 Mechanical analysis results

The effects of SEBS-MA on the pure SW have been analysed considering a polymer mass fraction of 10% and 20% with 1 wt.% of carbon powder. As shown in Figure 8 and reported in Table 6, the addition of SEBS-MA translates in an increment of the maximum stress and of the maximum elongation of the formulation. The Young modulus of fuels shows a decrease when passing from SW to SWS20. SWS10 and SWS20 features similar σ_y values. The SWS10 data at break show a reduced scattering with respect to the corresponding results of the baseline. This is probably due to the more efficient mixing of SW with reduced polymer mass fraction (i.e., better dilution and uniformity of the blend). On the other hand, using 5% of LW in W1S20 blends shows a significant increase in the elongation of the formulation (see Figure 9).

Table 6 Tensile properties of paraffin-based fuel blends with SEBS-MA and LW. Data are normalized to SW values.

Fuel Id.	Young modulus	Yield Stress	Yield Strain
SW	1	1	1
SWS10	0.99	1.50	1.70
SWS20	0.91	1.52	2.32
SW95LW5S20	0.79	1.39	2.90

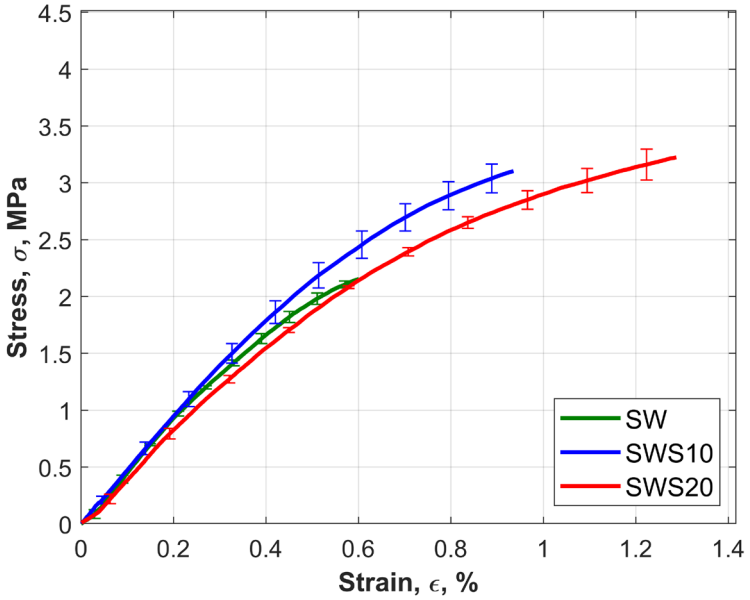

Figure 8: Effects of SEBS-MA on tensile properties of SW.

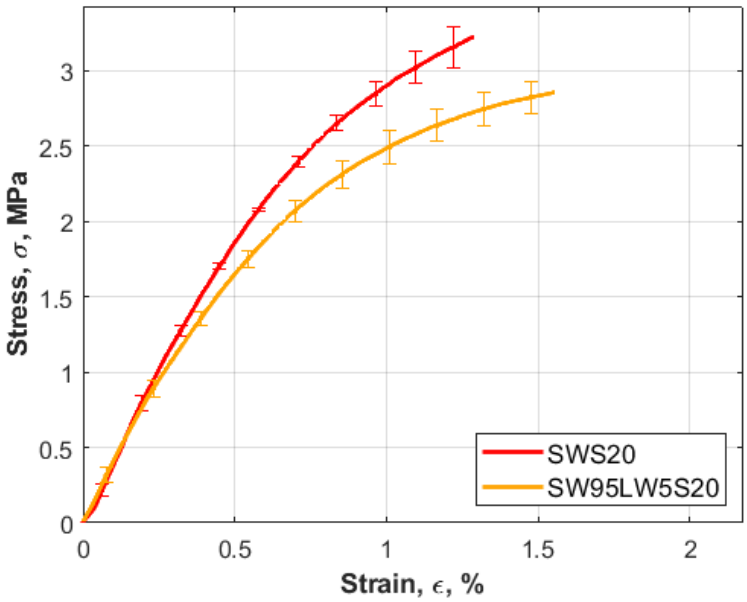

Figure 9: Effects of LW on tensile properties.

5. Conclusions

Investigation of the thermal, rheological, and mechanical behaviour of novel paraffin-based reinforced fuels was done to provide an evaluation of the fuel final properties. The base fuel of this study was Sasol Wax 0907 reinforced with thermoplastic polymer SEBS-MA. LW was used to replace a portion of the paraffin wax to investigate its effect on the performance of the fuel blends. Thermal characterisation showed that increasing the mass fraction of LW inside the blends with different mass fraction of SEBS-MA leads to a decrease in the melting temperature of the fuel. In addition to this, t a decrease on the onset temperature of the blend is achieved, while the end temperature remains almost the same. Rheological behaviours of the blends indicated that higher mechanical properties at higher temperatures are afforded by the blends with SEBS-MA. The viscosity value at the same shear rate and temperature increased by increasing the mass fraction of reinforcing styrenic copolymer, yet it is reduced thanks to the use of LW. Mechanical characterization from tensile tests proves adding SEBS-MA increases formulation stiffness and ductility. The formulations with 10 wt.% of SEBS-MA features a yield stress increase of 50%, while the yield strain is increased by 70% with respect to SW fuel. Using 5% of LW as a percentage of wax amount in W1S20 blends shows an increase of 25% in elongation with respect to the formulation with the same amount of SEBS-MA. LW-including blends offer similar mechanical properties with a melt fuel viscosity reduction possibly offering faster regression rates.

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