



Surface engineered nanoparticles dispersed in kerosene: The effect of oleophobicity on droplet combustion



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ABSTRACT

Liquid propellants benefit from solid particle additives that can optimize combustion performance by promoting phase change heat transfer. In this study, aluminum (Al) nanoparticles with and without self-assembled monolayer surface functionalization were combined with kerosene to examine the changes in droplet regression behavior associated with manipulating particle surface chemistry. Aluminum nanoparticles were coated using a long-chain perfluorinated carboxylic acid as the surface binding moiety to induce an oleophobic surface. The resulting particles are thus comprised of self-assembled monolayers of perfluorohexadecanoic acid (PFHD) ($C_{15}F_{31}COOH$) around the alumina (Al_2O_3) shell encapsulating the Al core. The PFHD serves many functions including altering particle wettability and acting as a surfactant that facilitates a stabilized dispersion of particles in kerosene. The Al-PFHD particles are more oleophobic compared with the more oleophilic surface associated with the amorphous alumina shell on aluminum particles. Mixtures with Al-PFHD exhibit a two stage burning behavior with average initial burn rate constant increased by about 121% when compared to pure kerosene. Further analysis using a thermogravimetric analyzer (TGA) showed that Al-PFHD particles in kerosene exhibit evaporation at reduced temperatures that may enhance energy transport during droplet combustion by phase change and convection. These results provide new insight bridging the gap between materials and heat transfer toward achieving the goal of designing particle additives that improve liquid propellant combustion.

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1. Introduction

Regression rate is an indicator of liquid propellant combustion performance and defines the rate that a droplet surface recedes over the course of its combustion [1]. Higher regression rates indicate faster combustion of the liquid fuel, which is usually desirable. An approach for improving the regression rate of liquid propellants is to include particulate additives that affect burning behavior by promoting heat transfer that leads to faster regression rates and improved combustion. Understanding the mechanisms for how particulate additives affect combustion is important for optimizing performance of next generation liquid fuels.

There are three basic approaches for selecting particulate additives to liquid propellants: (1) exploit chemical energy associated with metal fuel particles (such as aluminum [2–4]) toward providing more chemical energy within the mixture that can be liberated upon reaction; (2) identify materials that may spur cat-

alytic exothermic reactions with the propellant (such as iron oxide, Fe_2O_3 , or cerium(IV) oxide, CeO [5]) and may increase the rate chemical energy is liberated upon reaction; and (3) optimize the number of nucleation sites within the liquid to activate phase change heat transfer at lower temperatures and promote improved combustion [4]. The first two approaches are basically driven by chemical mechanisms to enhance either overall energy liberated or the rate that energy can be liberated during droplet combustion. The third approach relies more heavily on accelerating combustion through heat transfer driven by nucleation sites within the multi-phase mixture. Phase change can enhance heat transfer because latent heat is typically much larger than sensible heat. For example, the latent heat needed to boil 1 gram of kerosene ($h = 251$ J/g) is about 25 times larger than the heat required to increase the temperature of the same amount of kerosene by 5 K (10 J/g). In addition, phase change is frequently accompanied by fast and large changes in specific volume that results in enhanced heat transfer due to convection. These processes will significantly contribute to enhancing droplet combustion.

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While the approach taken in this study uses aluminum (Al) particles, the mechanisms associated with activating phase change heat transfer is the focus of this study. The hypothesis is that high heat transfer rates associated with surfaces that promote phase change and nucleate boiling will improve combustion more than simply the addition of nanoparticle aluminum (i.e., nano Al) additives. Toward this end, two different particle additives were examined: aluminum particles compared with the same aluminum particles with a surface functionalization to alter the wettability of the particle.

The quasi-steady process of nucleate boiling involves the temporal sequence of nucleation, bubble growth and detachment. Heat is exchanged optimally when the solid particle surface is in contact with both the vapor and liquid phase [6]. Therefore, a strategy for designing optimum particle surfaces relies on controlling the surface structure to promote contact with both the continuous (liquid) and discrete (vapor) fluid phases. One way to achieve a prescribed surface architecture is by processing techniques that alter surface chemistry; such as by depositing low-surface energy materials (e.g., fluoropolymers) that will induce surface hydrophobicity (i.e., water repelling) and oleophobicity (i.e., oil repelling) properties, and promote multiphase interfaces. In this case, the substrate is amorphous alumina, the passivation shell on aluminum particles. A perfluoro-organic acid was chosen as the coating species to induce greater oleophobicity that will affect nucleate boiling of kerosene and improve droplet combustion [7]. This approach could also be extended to manipulate particle hydrophobicity to impact more polar liquid fuels.

The purpose of this study is to exploit the surface chemistry associated with a perfluorinated self-assembled monolayer (SAM) coating on Al nanoparticles toward optimizing nucleate boiling and improving droplet combustion. This is accomplished by experimentally measuring droplet surface burn rates for kerosene combined with 0.5 wt.% nano Al particles, and kerosene combined with the same concentration of nano Al particles functionalized with perfluorohexadecanoic acid (PFHD) SAMs. Further characterization diagnostics include thermal gravimetric analysis (TGA) for evaluation of phase change behaviors; as well as Fourier transform infrared spectroscopy (FTIR) for evaluation of surface features of Al-PFHD particles; and Laser Flash Analysis (LFA) to quantify the differences in thermal properties of the sample mixtures. Results shown here extend the usefulness of surface engineered materials to applications involving liquid propellants, a medium not previously studied using particles with SAM coatings to control surface wettability as a parameter affecting combustion.

2. Experimental

2.1. Materials

The base propellant used for this study is kerosene ($C_{12}H_{26}$), from Sigma Aldrich (St. Louis, MO). Kerosene is refined from petroleum oil and a commonly used surrogate for aviation fuel [8]. Kerosene is non-polar and has oil-like properties as opposed to water-like properties. Therefore, particle surface engineering must be designed to produce oleophobic rather than hydrophobic properties. This is most commonly achieved with fluorocarbon coating materials.

The aluminum (Al) particles have an 80 nm average diameter and were procured from Nova Centrix (Austin, TX). The Al particles are passivated with an alumina (Al_2O_3) shell with an average thickness of 3 nm, and were well characterized by Kappagan-tula et al. [9]. In addition to this baseline Al powder, the same Al powder was also functionalized with self-assembled monolayers (SAM) prepared from a perfluorinated carboxylic acid, specifically, perfluorohexadecanoic acid (PFHD). The procedure for accomplish-

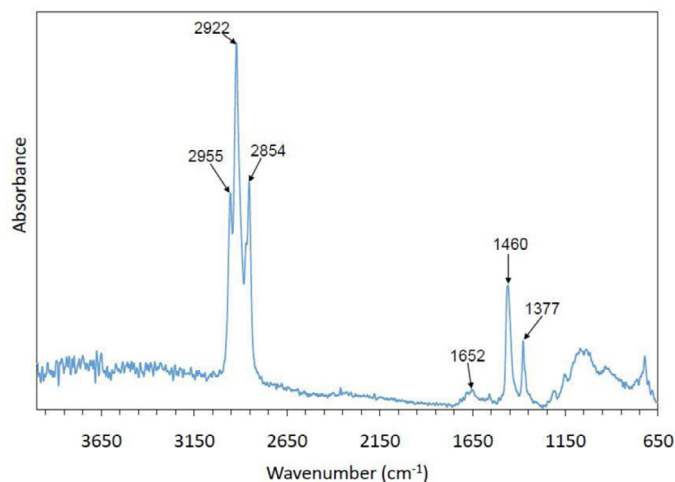


Fig. 1. Spectrum of Al-PFHD in kerosene using ATR-FTIR.

ing similar functionalization is well described in [10] and [11] to obtain particles hence referred to as Al-PFHD, with 35 wt.% acid concentration consuming an average corona thickness of 5 nm (similar to [9]). The powder product was washed three times in diethyl ether to remove any acid that was not bonded to the alumina shell. The dried powder was reclaimed for further experimentation as described below.

The Al-PFHD powder was examined using Fourier transform infrared spectroscopy (FTIR) with a Tensor 27 manufactured by Bruker Optics (Billerica, MA). Samples of roughly 10 mg quantity were loaded on an attenuated total reflectance (ATR) accessory and scans were made at a resolution of 1 cm^{-1} . The analysis was performed to characterize PFHD bonding to the Al particle surface, which is an extension to previous FTIR analysis of perfluorotetradecanoic acid (PFTD) coated Al particles [10].

Figure 1 shows peaks in the $1300\text{--}1500\text{ cm}^{-1}$ region associated with C–F stretching modes and the $2800\text{--}3000\text{ cm}^{-1}$ region is associated with O–H stretching modes [10]. The presence of C–F groups suggests $C_{15}F_{31}COOH$ remains intact upon absorption to the alumina surface. Another observation is an absence of the carbonyl stretching mode at 1754 cm^{-1} but there are multiple O–H stretching modes $2854\text{--}2954\text{ cm}^{-1}$ such that the carboxylic acid may absorb to the alumina surface through scission of O–H bonds and consequently results in the formation of a carboxylate species. This was observed previously for PFTD on Al [10] such that the bonding behavior for Al and PFTD appears similar to Al and PFHD.

The mixtures were analyzed for their bulk thermal properties using a Netzsch Laser Flash Analyzer (LFA) 447 at 25 and 50 °C for each sample. Approximately 0.3 cm^3 of kerosene, Al with kerosene, and Al-PFHD with kerosene were prepared and analyzed. Multiple tests with thirty measurements per test were evaluated and averaged to ensure repeatability of the measurement. This analysis was performed to understand how the bulk thermal properties of the mixtures vary as a function of particle composition. Table 1 includes the measured average thermal diffusivity for each sample at 25 and 50 °C. The variation in measured thermal diffusivity is relatively constant among all samples and consistent with reported literature for kerosene [14].

2.2. Burn rate constant measurements

Droplet burning behavior was experimentally observed using a modified apparatus as explained Datta et al. [12] and Bello et al. [13]. A $50\text{ }\mu\text{m}$ diameter quartz fiber with a circular cross-section acted as the support for fuel droplet suspension. It is also noted

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