Autoignition and Combustion Characteristics of Heptane Droplets containing Aluminum Nanoparticles at Elevated Temperatures

Irfan Javed*, Seung Wook Baek, Khalid Waheed

Department of Aerospace Engineering, School of Mechanical, Aerospace and Systems Engineering, Korea Advanced Institute of Science and Technology (KAIST)
291 Daehak-ro, Yuseong-Gu, Daejeon 305-701, Republic of Korea

ijkasuri@gmail.com

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Abstract

Nanofluid fuels, stable suspensions of metallic nanoparticles in traditional liquid fuels, have potential to enhance the volumetric energy density of liquid fuels but they are rarely studied. The objective of the current research work is to develop a fundamental understanding about the droplet combustion behavior of nanofluid fuels. The present experimental study investigated the effects of high ambient temperatures and various concentrations of nanoparticles (NPs) on autoignition and combustion characteristics of heptane-based nanofluid droplet. An isolated, single n-heptane droplet containing various concentrations (0.5%, 2.5% and 5.0% by weight) of aluminum (Al) NPs and hanging at a silicon carbide fiber were suddenly exposed to elevated temperatures (600–850 °C) at atmospheric pressure (0.1 MPa) under normal gravity and its autoignition and combustion characteristics were studied experimentally. The ignition delays, burning rate constants and combustion characteristics of pure and stabilized heptane droplets have been also examined for comparison. The results show that similar to pure n-heptane droplets, the ignition delays of NPs laden heptane droplets were also followed the Arrhenius type of expression and decreased exponentially with increase in temperature. The addition of dense concentrations (2.5% and 5.0%) of Al NPs to heptane increases the physical delay of droplets as compared to pure heptane droplets at relatively low temperatures (below 700 °C) and the ignition was extinct at 600 °C. At relatively high temperatures the physical delay of both types of droplets was comparable. The combustion of nano-Al containing heptane (n-Al/heptane) droplet was not followed the classical $d^2$-law in contrast to the combustion of pure heptane droplet. Regardless of NP concentrations, the n-Al/heptane droplets exhibited disruptive burning behavior, which was characterized by multiple-time expansions and ruptures and known as ‘microexplosions’. The NPs were brought out of the droplet through these microexplosions whose intensity was increased with an increase in temperature; consequently, the burning time and total combustion time of droplet was reduced. At relatively low temperatures (600–700 °C) the burning rate of n-Al/heptane droplets remained equal to pure n-heptane droplets whereas at relatively high temperatures (750–850 °C) the burning rates of nanofluid fuel droplets were substantially higher than those of pure n-heptane droplets.

Introduction

Metals such as Al, boron (B) and carbon (C) have long been of interest to the propulsion and combustion community due to their higher combustion energies. The novel approaches and latest advances in nanotechnology would enable the large scale production of nanomaterials which have shown significant advantages over micron sized particles. When size of metallic particles reduces from micro- to nano-scale, their thermophysical properties often changes significantly. Numerous studies have described decrease in melting points and lower heat of fusion for nano-sized metal particles. Several researchers investigated the effects of addition of NPs to solid fuels and propellants. They have reported many advantages over micron-sized particles such as improved energy density, lower ignition delay, and increased burn rates.

The use of metallic nano powders as energetic fuel additives has received a considerable attention in recent years. The present paper is based on adding NPs to liquid fuels, called nanofluid fuels, which have gotten enormous interest of the combustion and propulsion community. Nanofluid fuels, a new class of nanofluids, are stable suspensions of energetic material NPs in traditional liquid fuels. Nano-powders of energetic materials (such as Al, B, C, iron (Fe) and cerium oxide (CeO₂) etc.) were used as additives and suspended in conventional liquid fuels in a proper way to enhance their ignition and combustion characteristics. Previous studies have shown significant improvements by such nanofluid fuels. Van Devener and Anderson found enhanced catalytic effects for CeO₂/JP-10 nanofluid fuel and the ignition temperature of JP-10 was significantly reduced. Tyagi et al. observed increased hot plate ignition
probability in Al and Al$_2$O$_3$ NPs laden diesel fuel. Sabourin et al.\textsuperscript{(11)} obtained enhanced burning rates and lower ignition temperature by adding functionalized graphene sheets to monopropellant nitromethane. Allen et al.\textsuperscript{(12)} experimentally found 32\% and 50\% reduction in ignition delays of Al/ethanol and Al/JP-8 nanofluid fuels, respectively. Jones et al.\textsuperscript{(13)} got increased heat of combustion with high loadings of Al NPs in ethanol fuel.

Nanofluid droplet combustion, similar to nanofluid droplet evaporation\textsuperscript{(14)}, is a highly complex phenomenon compared to liquid fuel droplet combustion due to its multi-component, multi-phase, and multi-scale nature. A couple of studies were reported in literature regarding the combustion of nanofluid fuel droplet. Gan and Qiao\textsuperscript{(15)} observed a disruptive burning behavior of the droplets with micro- and nano-sized Al particle suspensions in ethanol and n-decane. The disruption behavior of the micro sized suspensions droplets occurred later and with a greater intensity compared to nano suspension droplets for the same surfactant and particle concentrations. Later, Gan et al.\textsuperscript{(16)} detected a simultaneous combustion of both the droplet and the NPs in dilute suspensions of boron and iron NPs in n-decane and ethanol. However, in dense suspensions, most particles were combusted as large agglomerates after the consumption of the liquid fuel.

The above mentioned studies\textsuperscript{(15,16)} give a simple qualitative behavior of nanofluid droplet combustion. In those studies the nanofluid droplet was ignited by a heating wire. Therefore, the effects of NPs on autoignition delay times or the droplet combustion behavior at various elevated temperatures are beyond the scope of those studies. Also, those studies are not able to explain the effects of NPs on combustion rate constants. To the best of our knowledge, no study has been reported regarding the effects of addition of various concentrations of NPs on autoignition and combustion characteristics of nanofluid fuel droplets at elevated temperatures.

Motivated by above, the present research work investigated the autoignition and combustion behavior of heptane-based nanofluid droplets, experimentally. The effects of NPs loading rates and high ambient temperatures on droplet autoignition and combustion characteristics were examined. The n-heptane is selected as a base-fuel because of simplest, highly pure single component liquid hydrocarbon which has abundant experimental data regarding evaporation, autoignition, combustion, and reaction mechanisms. So it would be helpful to investigate the effects of addition of NPs. The Al NPs were used as energetic additives. The ambient temperature was varied from 600–850 °C, which is higher than the autoignition temperature of n-heptane (220 °C) as well as below and above the melting point of the Al NPs (660 °C), and the ambient pressure was kept constant at 0.1 MPa.

### Experimental Methods

The materials and instruments used in current research work were already utilized in experimental investigation of evaporation characteristics of heptane-based nanofluid droplets\textsuperscript{(17)}. All these materials were used in their as-received form without further treatment except the Al NPs whose surfaces were coated with oleic acid (OA). A planetary ball mill (Retsch PM100, GmbH, Germany) was used to modify the surfaces of the Al NPs.

### Preparation of Stable Nanofluid Fuels

The heptane-based stable nanofluid fuels were prepared using a two-step method. In this method the already produced NPs were uniformly suspended in a base fluid through applying various physical and chemical homogenization techniques. Because the metallic NPs were available at industrial scale therefore it is the most economic method to produce numerous types of nanofluids with various concentrations of NPs, either in the laboratory or at a large scale. The post-synthesis surface modification concept was applied to the purchased Al NPs in order to improve their dispersion stability in n-heptane. The surfaces of the NPs were coated by OA using a ball mill and oxide-free, ligand-protected and fuel-soluble Al NPs were prepared. The resulting NP/OA paste was dispersed in heptane through vigorous agitation by hand. A homogeneous suspension was obtained in this way, which remained stable for 2 h with no obvious sedimentation of the NPs. The detailed procedure adopted here has been described in previous research works\textsuperscript{(14,18)}.

### Experimental Apparatus and Procedure

An experimental setup was previously fabricated and installed to investigate the evaporation, autoignition, and combustion characteristics of a single, fiber-suspended liquid fuel droplet at elevated temperatures and pressures, as described in\textsuperscript{(19–22)}. The same experimental apparatus has also been used to study the evaporation behavior of heptane- and kerosene-based nanofluid fuels\textsuperscript{(14,17,18)}. The experimental procedure, and the data reduction and analysis, were also described in detail in those studies\textsuperscript{(14,17,22)}. A schematic view of the experimental apparatus is provided in Figure 1. The experiments were performed with a single nanofluid fuel droplet suspended by a fine SiC fiber (100 μm diameter). The initial average diameter of a droplet was 1.0 ± 0.10 mm. The ambient temperature was varied from 600–850 °C and the ambient pressure was kept constant at 0.1 MPa. High-temperature ambience was provided by a freely falling electric furnace. The oxidizing environment was provided by dry air. The combustion process was recorded by a high-speed charge-coupled device (CCD) camera. A flexible image-processing code was developed using Matlab to obtain the droplets’ diameters from the captured
images. This code was executed iteratively for each image, resulting in the temporal variation of droplet diameter \((d)\) during combustion. If the droplet combustion followed \(d^2\)-law then the combustion rate constant can be expressed as the time derivative of the droplet diameter squared, \(K_c = -\frac{d(d^2)}{dt}\). Thus, the droplet combustion rate constant was obtained from the temporal variation of droplet diameter squared by measuring the slope of its linear regression.

When an isolated hanging droplet present at room temperature and ambient pressure is suddenly exposed to high temperature environment, vaporization starts after an initial heating up period. The fuel vapor generated is transported outward, mixes with the oxidizer gas in the ambience. When fuel vapor/oxidizer mixture of combustible proportion is produced and the fuel activation energy is overcome, ignition takes place\(^{21,24}\). The time lag between high-temperature exposure and the minimum amount of fuel vapors production, necessary for ignition is known as physical delay. And the time span between the availability of ignitable amount of vapors and the overcoming of activation energy is called chemical delay\(^{22}\). It is noteworthy that in the autoignition of homogeneous mixtures, the physical time is significantly reduced as it only involves the time for "complete" mixing of fuel and air\(^{23}\).

The objective of the present study is to observe the effects of high ambient temperatures and NPs concentrations on autoignition characteristics of heptane-based nanofluid fuel droplets since no ignition study has been done so far for such fuel droplets. The autoignition delay time of single component pure fuel (n-heptane) droplets has been studied first as a base line for comparison. This helps to distinguish the effect of addition of NPs on autoignition behavior of heptane-based nanofluid fuel droplets. The initial diameter of the droplets was 1.0 ± 0.1 mm. The temperature ranged from 600 to 850 °C and pressure was kept constant at 0.1 MPa. Ignition delay time is defined as time from the entry of the droplet into the furnace to the ignition radiation\(^{26}\). The occurrence of ignition has been identified visually by the appearance of a flame through the use of high-speed colored photography as studied previously by our group\(^{21}\).

**Results and Discussion**

**Droplet Autoignition**

Spontaneous ignition or autoignition implies to the ignition of a combustible mixture without the support of any external source. The autoignition characteristics have been represented in terms of an ignition delay obtained as a function of initial gas temperature, and the effects of other variables (such as pressure, equivalence ratio, droplet size, fuel type etc.) were included through a parametric investigation\(^{23}\). The ignition delay is generally defined as the time interval between the creation of a combustible mixture and the "appearance" of a flame. The ignition delay has been considered as comprising a physical delay and a chemical delay.

**Fig. 1** A sketch of the experimental setup (1) Pressure vessel, (2) guide bar, (3) furnace entrance, (4) electric furnace, (5) quartz glass window on furnace, (6) thermocouple, (7) temperature controller, (8) lever, (9) dry air vessel, (10) quartz glass window on pressure vessel, (11) LED backlight, (12) silicon carbide fiber, (13) droplet, (14) shock absorber, (15) heating wire, (16) droplet maker, (17) plunger micro pump, (18) CCD camera.

**Pure Fuel Droplet Autoignition**

Figure 2 shows the effect of ambient temperature on the autoignition delay times for pure n-heptane droplets at a constant pressure of 0.1 MPa. The ignition delay time decreased with an increase in temperature as expected and reported by various researchers\(^{26-28}\) in past. At high ambient temperature, the ignition delay time of heptane levels off. The reason is that the chemical delay becomes smaller at elevated temperatures. The pre-evaporation heating time (one of the important parameter in physical delay) of heptane, which is too little now, controls the ignition delay. A simple comparison of ignition delay data along with initial diameters of pure n-heptane droplets between previous results of Takei et al.\(^{29}\) and current study at various ambient temperatures and 0.1 MPa pressure has been also shown in Figure 2. The droplets were selected within a narrow range of initial diameters from 0.925 to 1.104 mm in order to reduce the effect of the initial droplet diameter on ignition delay. As this figure shows, despite lack of data at low temperatures in results of Takei et al.\(^{29}\), the ignition
delays match well each other except for low temperature region in which some difference was observed.

Fuel: n-Heptane

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<tr>
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Fig. 2 Comparison of ignition delay times along with initial diameters of pure n-heptane droplets between present study and previous study by Takei et al. at various ambient temperatures.

**Effect of Ambient Temperature on Autoignition**

In Figure 3, the ignition delay data of n-heptane droplets was presented with an Arrhenius type of expression where the logarithmic ignition delay times were plotted on the ordinate versus the inverse of absolute ambient temperature which was showed on the abscissa. At atmospheric pressure, the autoignition delay times reduced exponentially with an increase in temperature. The experimental results in the figure are correlated with parallel linear lines, which imply that they are formulated with the following relation $r = A \exp(D/T)$ where $r$ is the ignition delay time in seconds, $D$ is a constant and $T$ is the ambient temperature in Kelvin. The above expression is valid for full range of ambient temperatures in the present study, which implies that the proportionality sign can be transferred to equality which leads to the equation $r = A exp(D/T)$, where $A$ is the proportionality constant. The values of the constants $A$ and $D$ for pure n-heptane were calculated from the autoignition delay data obtained experimentally. The values were found to be $A = 0.0219$, $D = 10800$ in current study. The values of $A$ and $D$ calculated from experimental data reported by Takei et al. were 0.0154 and 11380. It is seen that constants’ values of heptane matched well with the reference.

**Stabilized Fuel Droplet Autoignition**

Based on both experimental and numerical investigations, S.K. Aggarwal concluded that the ignition behavior of bi-component fuel sprays is dominated by the more volatile component of fuel. An important implication here is that the ignitability of a low volatile fuel spray can be greatly enhanced by adding a small amount of a volatile component, whereas the ignitability of a volatile fuel spray is hardly affected by adding a significant amount of low-volatile fuel. Based on this conclusion the ignition delay times of stabilized heptane droplets are considered to be same to pure heptane droplets because low concentrations of OA has been added into heptane droplets.

**Autoignition of n-Al/Heptane Droplets**

In this section, the effects of temperature and NPs concentration on autoignition delay times of nano-Al containing heptane (n-Al/heptane) droplets were discussed. Droplets of approximately same sizes (0.907–1.07 mm diameter) were formed and the ignition was detected by the help of high-speed photography (500 frames per second).

**Effect of Ambient Temperature on Autoignition Delay Times**

Fig. 4 Comparison of ignition delay times of heptane-based nanofluid droplets containing 0.5%, 2.5% and 5.0% Al NPs with pure heptane droplets at various ambient temperatures.
Figure 4 gives a comparison of autoignition delay data of n-Al/heptane droplets with pure n-heptane droplets at various ambient temperatures. Similar to pure heptane droplets the autoignition delay times of NPs added heptane droplets were also followed the same Arrhenius type expression and decreased exponentially with increase in temperature. With relatively high loading rates of NPs (2.5% and 5.0%) in heptane, the 1 mm diameter droplets were not ignited at relatively low temperature (600 °C). Their ignition delays also increased as compare to pure heptane droplets at 650–700 °C which were remained close to the ignition delays of pure heptane droplets at 800–850 °C. These results show that the addition of dense concentration of Al NPs to heptane increases the physical delay of droplets as compared to pure heptane droplets at relatively low temperatures. At relatively high temperatures (750–850 °C), the physical delays decreased significantly therefore variation in concentrations of NPs could not be able to effect the ignition delay time of heptane droplet.

**Droplet Combustion**

In this section, the effects of addition of various concentrations of NPs on droplet combustion were investigated at higher ambient temperatures. The combustion rate constant and burning behavior of single component pure fuel (n-heptane) droplets has been studied first as a base line for comparison. This helps to distinguish the effect of addition of NPs on combustion behavior of heptane-based nanofluid fuel droplets. The initial diameter of the droplets was 1.0 ± 0.1 mm. The temperature ranged from 550 to 850 °C and pressure was kept constant at 0.1 MPa.

**Pure Fuel Droplet Combustion**

Figure 6 shows the normalized diameter squared history of combusting pure n-heptane droplets at various ambient temperatures.

The n-heptane droplet of 1 mm initial diameter was not ignited at 550 °C. However ignition and subsequent combustion was observed in the range 600–850 °C. As ignition started, the droplet expanded and then ruptured to a smaller sized droplet and then its diameter reduced by following $d^2$-law. The bubble formation and then its rupture in a pure n-heptane droplet were the effects of fiber which caused it through heterogeneous nucleation.

The total combustion time ($t_{cc}$) which is the sum of ignition delay time ($t_{ig}$) and burning time ($t_b$) has also...
been decreased with an increase in temperature similar to autoignition delay time whereas the burning time of a droplet has been increased up to 750 °C and then remained constant as shown in Figure 7. The combustion rates were calculated from the linear combustion part of n-heptane droplets through using least square method.

![Figure 7](image)

**Fig. 7** A comparison of ignition delay time, burning time and total combustion time of n-heptane droplets at various ambient temperatures and constant pressure of 0.1 MPa.

A comparison between the current values and previous data of combustion rates of n-heptane droplets has been shown in Figure 8. The combustion rates obtained in present study were quite high at relatively low temperatures (600−700 °C) whereas they are comparable to previous values at relatively high temperatures. This is due to difference in droplet diameters and fiber diameters used in both studies. Qasim et al.\(^{(24)}\) suspended droplets with an initial diameter of 1.0−1.2 mm on quartz fibers with diameters of 0.125 and 0.2 mm that were rounded at the tips with bead diameters of 0.25 and 0.35 mm, respectively. However, in our current research work, each droplet was suspended on a 0.1-mm SiC fiber with no bead at its tip, and droplets having similar initial diameters ranging from 0.912 to 1.0831 mm were selected from the experimental data.

**Combustion of n-Al/Heptane Droplets**

**General Combustion Behavior**

Figures 9, 10 and 11 show temporal variations of diameter squared of combusting n-heptane droplets containing 0.5%, 2.5% and 5.0% Al NPs whose surfaces were coated with 0.5%, 2.5%, and 5.0% of OA, respectively, at ambient temperatures ranging from 600−850°C.

![Figure 9](image)

**Fig. 9** Temporal variation of diameter squared of combusting n-heptane droplets containing 0.5% Al NPs at different environmental temperatures.

![Figure 10](image)

**Fig. 10** Temporal variation of diameter squared of combusting n-heptane droplets containing 2.5% Al NPs at different environmental temperatures.

The droplets were selected from the experimental data within a narrow range of initial diameters from 0.953 to 1.124 mm to reduce the effect of the initial droplet diameter.
diameter on combustion behavior. In contrast to pure heptane droplet combustion, the \( n \)-Al/heptane droplet combustion was not followed the classical \( d^2 \)-law. However, the vaporization before ignition followed the \( d^2 \)-law. The most prominent effect of addition of NPs; irrespective of NPs concentrations, is the disruptive burning of heptane-based nanofluid droplets. The NPs were brought out of the droplet by a disruptive behavior of the primary droplet, which was characterized by multiple-time expansions and ruptures. As the temperature increased, the intensity of such micro-explosions was also increased. It is noteworthy that as the concentration of NPs increased from 0.5% to 2.5% or 5.0%, the combustion was not occurred at relatively low temperature (600 °C). However at relatively high temperatures (800–850 °C), the intensity of micro-explosions was increased which reduced the burning time as well as total combustion time.

Figure 12 shows a comparison between burning times of heptane-based nanofluid droplets containing 0.5%, 2.5% and 5.0% of Al NPs with pure heptane droplets at various ambient temperatures. This figure represents that similar to pure heptane droplets, the burning times of heptane-based nanofluids droplets were also increased with increase in temperature. However with 0.5% and 2.5% NPs loading rate, the burning time obtained at relatively high temperature (800–850 °C) was lower than the pure n-heptane droplets. This is due to micro-explosions which were occurred intensively at these temperatures. In contrast, the low values of burning times at relatively low temperatures (650–700 °C) with 2.5% and 5.0% NPs were due to delayed ignition which occurred when droplet diameters were significantly reduced.

Figure 13 gives a comparison between ignition delay times along with total combustion time of heptane-based nanofluid droplets containing 0.5%, 2.5% and 5.0% of Al NPs with pure heptane droplets at various ambient temperatures. This figure indicates that; irrespective of NPs concentrations, ignition delay times are dominant in total combustion time at relatively low temperatures whereas at relatively high temperatures the burning time were higher than the ignition delay and majorly contributing in total combustion time.

**Effect of Ambient Temperature on Burning Rate Constant** As it was mentioned in previous section that the combustion history of \( n \)-Al/heptane droplets did not obey the \( d^2 \)-law, thus the least square method is not applicable for calculation of combustion rate constant. In heptane-based nanofluid, the droplets started to expand before the ignition occurred. Thus the combustion rate constant could not be calculated from droplet ignition diameter (\( d_{ig} \)) and burning time (\( t_b \)). Therefore, for heptane-based nanofluid fuel droplets, the average burning rate was defined as \( K_b = \)
where $K_b$ is the average burning rate constant, in $mm^2/s$, $d_0$ is the droplet initial diameter, in $mm$, and $t_0$ is the total combustion time of droplet, in $s$, which is equal to the sum of the ignition delay ($t_{ig}$) and burning time ($t_b$). Although the droplet diameter squared is not linearly varying with time, however the average burning rate constant defined here is only a measure of the mass combustion rate which provides the combined effect of NPs on whole combustion process (initial heating up period, vaporization, ignition and disruptive burning) of nanofluid droplets.

**Effect of NPs Concentration on Burning Rate Constant**

Figure 14 also indicates that the average burning rates of heptane-based nanofluid droplets containing 0.5%, 2.5% and 5.0% Al NPs with pure heptane droplets at various ambient temperatures.

Fig. 14 Comparison of the average burning rates of heptane-based nanofluid droplets containing 0.5%, 2.5% and 5.0% Al NPs with pure heptane droplets at various ambient temperatures.

Figure 14 compares the average burning rates of $n$-Al/heptane droplets containing 0.5%, 2.5% and 5.0% Al NPs with pure n-heptane droplets along with each other. Similar to pure n-heptane droplets, with an increase in temperature the average burning rate of nanofluid droplets has been increased. However this increasing trend is significantly enhanced with the addition of NPs. At relatively low temperatures (600–700 °C), the burning rate of $n$-Al/heptane droplets remained equal or slightly lower (for 2.5 and 5.0% NPs) than the burning rates of n-heptane droplets. However at relatively higher temperatures (750–850 °C), irrespective of NPs concentration, the burning rates of nanofluid droplets were substantially higher than those of pure n-heptane droplets.

**Conclusions**

The effects of various dilute and dense concentrations of Al NPs on autoignition and combustion of heptane-based nanofluid fuels droplets were investigated. This research work provides basic understandings about the autoignition and some useful data regarding the combustion behavior of these nanofluid fuels droplets. The results obtained are summarized as follows:

- Similar to pure fuel droplets, the autoignition delay time of heptane-based nanofluid fuel droplets was also decreased with an increase in ambient temperature and can be expressed by the same Arrhenius-type empirical relation, $\tau = A \exp(D/T)$.

- The addition of dilute (0.5%) and dense (2.5 and 5.0%) concentration of Al NPs to heptane droplets decreases and increases the ignition delay of heptane droplets, respectively at relatively low temperatures (600–700 °C) whereas at relatively high temperatures (750–850 °C), addition of NPs did not affect the ignition delay of heptane droplets. With the addition of dense concentrations (2.5% and 5.0%) of NPs, ignition was not occurred at 600 °C, at which ignition was observed otherwise.

- Unlike pure n-heptane droplets, the heptane-based nanofluid droplets showed disruptive burning behavior at all tested temperatures (600–800 °C) and did not obey the classical $d^2$-law. The NPs were brought out of the droplet through these disruptions/micro-explosions which were intensified at elevated temperatures.

- The ambient temperature significantly affected the average burning rates of heptane-based nanofluid fuel droplets. The added Al NPs exhibit opposite effects at relatively low and high temperatures. At relatively low temperatures (600–700 °C), the burning rate of $n$-Al/heptane droplets remained equal or slightly lower (for 2.5% and 5.0% NPs) than the burning rates of n-heptane droplets. However, irrespective of NPs concentration, the burning rates of nanofluid droplets were substantially higher than the pure n-heptane droplets at relatively high temperatures (750–850 °C).

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