Near-limit oscillatory behaviors on wick flames of dimethyl carbonate with trimethyl phosphate additions

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Abstract

The near-limit oscillatory behaviors on wick flames of dimethyl carbonate (DMC) with trimethyl phosphate (TMP) additions have been investigated experimentally. The experiments were conducted under a wick burner in conjunction with the limiting oxygen concentration test (wick-LOC), and the fuels were selected as typical examples of electrolyte solvents and organophosphorus compounds (OPCs) for lithium-ion batteries. The near-limit oscillating flames on the wick configuration were observed into two main characters: side-to-wake or side oscillation in for the side-stabilized flame (full flame) and wake oscillation for the wake-stabilized flame (wake flame). By tracing the flame base movement using a 240-fps camera, stable limit cycle oscillations were found in full flame cases, while the wake flame cannot sustain the oscillation for long and finally leads to global extinction. With the addition of TMP in DMC, the transition from the side-to-wake oscillation to the side oscillation was found in the unstable full flames with a linear increase in frequency and a significant drop in amplitude, while the wake oscillating flames performed increased trends for both. To clarify the dominant mechanism of TMP-added flame oscillations, laminar burning velocities of DMC+TMP mixtures were calculated at the oxygen level of each near-limit oscillating flame. The weakened flame speed with TMP addition revealed the inadequacy of buoyancy-driven mechanisms for the OPC added wick-flame. Then the wick surface temperature was measured adopting a special thermocouple arrangement to validate the thermal-diffusive promotion by TMP addition. Results showed that the heat feedback from TMP-added flames compensated for the low reactivity and provided a faster oscillation near the extinction limit.

Keywords

Wick flame; Flame oscillation; Extinction limit; Electrolyte; Organophosphorus compound

1. Introduction

Near-limit oscillatory behaviors of laminar diffusion flames have attracted attention of fire researchers in the fields of material flammability [1–4], flame spread [5–7], and flame suppression [8,9], which is regarded as a precursor of flame extinction/instability limit. Researches on instability of diffusion flames are important for the fundamental understanding of fire hazard and the classification of the material flammability.

Besides the gaseous fuel combustion, using candle-like flame configuration is a typical way to study the fire of condensed materials (e.g. polymer rod, wire, candle and alcohol lamp). Experimental studies focusing on the near-limit oscillation of a candle-like flames have been conducted since 1970s [1], followed by the further modeling on candle flame oscillations [10]. The near-limit flame oscillatory behaviors of condensed materials differ in the experimental conditions. With carefully controlling of the oxygen, the limit-cycle wick flame oscillation on an ethanol lamp can be found for even hours [11]. While in LOI-type devices, the flame of PMMA rods can only oscillated for a few seconds [2,12].

A glance at literature shows [13–18], that the controlling mechanisms of the near-limit flame oscillations varied with the experimental geometries and fuel types, and their characters of oscillation are affected by the heat losses, Lewis numbers, radiations and gravities in many parametrical studies by numerical or theoretical analysis. The typical near-limit oscillations of diffusion flames have low frequencies (a few Hz). Besides the Kelvin–Helmholtz instability for flickering flames, the mechanisms mainly attributed to the buoyancy-driven instability (especially in normal gravity) and thermal diffusive instability (instinct pulsating) [2,8,17]. These works about the edge flame instability help to understand the near-limit oscillatory behaviors of complicated fire for condensed materials.

In the previous research [19,20], the wick-LOC method has been utilized for evaluating the flammability of electrolyte solvents used in lithium-ion batteries (LIBs). Adding organophosphorus compounds (OPCs) into carbonate solvents is an efficient way to improve the flame-retardancy of electrolytes without changing the structure of LIBs. In the study of flame-retardant effects of OPC additives in carbonate solvents, two types of stabilized flame modes have been found, namely, full flame and wake flame. A critical amount of OPC addition divided the flame stability manner of the full and wake flame regimes where the fluid dynamic and thermal balance alternately took advantage, and the near-limit oscillating flames were observed as well. To explore more insight into the role of OPC addition on the mechanism of wick flame stabilization, oscillation and extinction, experimental studies on the near-limit oscillating flames were conducted. In the present work, dimethyl carbonate (DMC, $H_3COCOOCH_3$) and trimethyl phosphate (TMP, $(CH_3O)_3PO$) were selected as the typical example of electrolyte solvents and OPC additives used in lithium-ion batteries.

2. Experimental approach

The experimental setup for the present work was based on the wick-LOC method apparatus reported by our previous works [19,20], and the schematic is shown in Fig.1. In principle, the setup followed LOI type apparatuses. It comprised three main parts: combustion chamber, gas supply system and fueling system. A cotton wick (7 mm in exposed length and 5 mm in diameter) was inserted in the stainless supporting tube in the centerline of the glass chamber and saturated with the electrolyte solvent. The liquid level of the electrolyte solvent was controlled steadily by the fueling system (right-hand side in Fig.1) utilizing the law of communicating vessels and pressure balance. The wick head can be refreshed continuously through the capillarity effect during the combustion. Thus, the potential distillation of TMP and DMC mixture due to their different boiling points have been considered to be negligible in the previous work [19]. Their evaporation and burning rates are spontaneously controlled by the interactions between the flame and wick, which are affected by oxygen levels and flame modes as well. The N_2/O_2 mixed gas was adjusted by the flow meter and supplied to the chamber from the bottom through the gas supply system. A candle-like wick diffusion flame can be generated and sustain under the specific upward external flow and oxygen concentration. In the present work, the external flow rate was controlled as a fixed value of 10cm/s to provide a laminar flow field; the oxygen concentration was adjusted precisely to find the oscillatory behaviors before the flame stability limits.

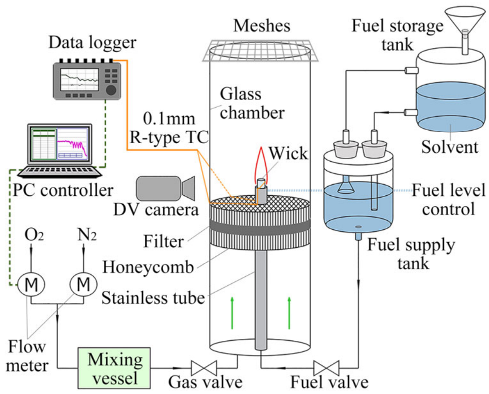

Fig. 1. Experimental setup schematic of the wick-LOC method [19,20].

DV cameras with 30 and 240 frames per second (fps) video rates have been utilized for the observation and further analysis of flame oscillations, which are competent to capture the oscillation phenomena in a few Hz. With the oxygen decrease after ignition, the long-term flame evolutions were recorded by the 30-fps camera including steady combustion, flame shrinking, oscillating, re-stabilizing and global extinction. For the transient phenomena of flame oscillation and extinction, the 240-fps camera was used for further image processing and analysis. During the flame oscillation, the overall image brightness is unstable, especially during the oscillation between full and wake flames. To highlight the flame body and reduce the background noise, the brightness and contrast of the videos were enhanced manually. As shown in Fig. 2, the flame bases, tips and side boundaries were identified by the specific color intensity thresholds along the horizontal and vertical pixels. The thresholds of blue color intensity for pure DMC flames and red color intensity for TMP added flames were selected according to the flame color difference given by DMC and TMP added solvents. The values of the threshold were set individually for different fuels, but it should be the same and proper for different flame modes (transient full or wake flame) during the oscillatory combustion of the same fuel. Then, the movement of flame boundaries can be recorded during the near-limit oscillation. For the near-limit oscillation in this work, the frequencies were determined by the periodic movement of the flame bases.

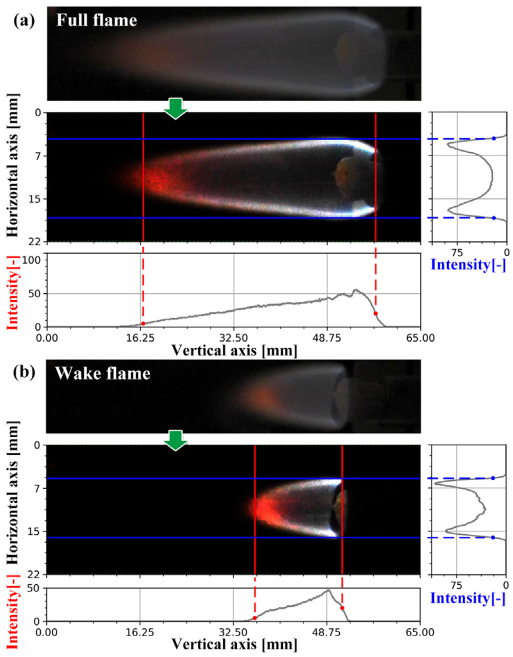

Fig. 2. Image processing for the flame boundaries during oscillatory combustion. (a) transient full flame of

DMC at 16.7% O_2, (b) transient wake flame of DMC at 16.7% O_2.

According to our previous work [20], the thermal balance became more dominant on the low-oxygen stability when the OPC was added. To clarify such an effect on the near-limit flame oscillation, the fine R-type thermocouple was specifically arranged to measure the wick surface temperature, as shown in Fig. 3. Two single wires (0.1 mm) of the thermocouple were wrapped around the wick surface closely to minimize the interference from the flame. The temperatures of four points on the wick surface were measured including center of the top surface, 1-, 3- and 5-mm height of the side surface. The data acquisition rate was 50 Hz which is suitable for flame oscillation with a lower frequency.

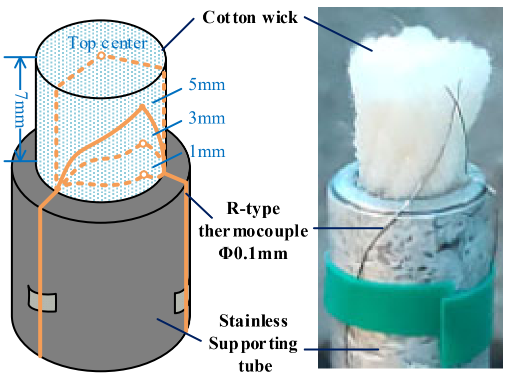

Fig. 3. Temperature measurement on the wick surface using fine R-type thermocouple. Left: schematic of probe arrangement, Right: Actual image.

3. Calculation method

According to the flame stabilization mechanism [8,21], the leading edge of the wick flame with a triple flame structure can be stabilized under the balance of reaction time and residence time (or edge flame propagation and buoyancy-driven local flow). As the propagation speed of edge flame was always proportional to the stoichiometric laminar burning velocity (S_L) [21], it was of interest to investigate the reactivity of near-limit flame bases by calculating the S_L under the near limit oxygen level.

The stoichiometric laminar burning velocity of DMC and DMC+TMP mixtures was calculated by CHEMKIN-PRO [22] using the Premixed Laminar Flow Speed Calculation (PLFSC) model. In the model, DMC and TMP addition in gas phase were homogeneously mixed with oxygen and nitrogen. Under stoichiometric conditions, the ratios of premixed oxygen and nitrogen were corresponding with the oxygen level in each flame oscillation experiment. The reaction mechanism of Glaude et al. [23] was applied for DMC combustion. The mechanism for TMP was taken from Korobeinichev et al. [24] and Jayaweera et al. [25]. In the premixed flame calculation, the initial gas pressure was atmospheric, and the unburnt gas temperature was 298 K. The grid points were set as 250 for a proper resolution (GRAD $\leq$ 0.1, CURV $\leq$ 0.5). Thermal diffusion and multicomponent transport were neglected.

3. Results and Discussion

By observing the flame evolution with the oxygen change under the same external flow of 10 cm/s, the near limit oscillatory behaviors of the wick flame were found and recorded. Figure 4 illustrates the consecutive video images from the 30-fps camera and the schematics of the flame oscillations. In the schematic of Fig. 4, the flame boundaries represent flame shapes in two states during oscillation. The movement of flame tail and flame bases were traced by double-headed arrows in different colors separately. The blue arrows in the wick show the fuel supply direction by capillarity action, and the blue pattern represents the preheat zoon on the wick.

As two types of flame stability limits (full and wake flame) was reported [20], the flame oscillations before full flame limit and wake flame limit occurred correspondingly. Figure 4a shows different oscillatory behaviors of near-limit full flames given by pure DMC (blue flame) and DMC with 5wt% TMP addition (luminous flame). In the typical cycle of oscillation, the flame base of pure DMC was lifted from the initial stabilization point of the wick side to the wake region of the wick, then flashed back to envelop the wick, which is similar to the near-limit oscillating candle flame reported by Chan [1]. By tracking the motion path of flame base qualitatively, the flame base passively drifted downstream along the boundary layer of the side surface and propagated back along a wider stoichiometric line due to the continuous fuel evaporation on the wick surface. Different from the side-to-wake oscillating flame of DMC, the full flame base of DMC+5%TMP oscillated faster in a short-range along the side surface. Sometimes, partially quenching of the flame base to the downstream can cause global extinction immediately but it never reached the wake flame (the LOC is lower for full flame than that of wake flame), as reported in [20]. In general, the geometry of the wick and tube might affect the flame behaviors. By tracing flame base location in Fig. 4, we assume the step between the tube and wick had limited effects on the oscillation behavior. However, if the tube wall became thicker, it might not be neglected.

Figure 4b shows the wake flame oscillation before extinction, DMC and DMC+5%TMP showed the similar manner of oscillation but different scales. The oscillating wake flame looks similar to the near-limit tube flame of liquid fuel [1] or lifted flame of gas fuel [17] in normal gravity. The larger size of the wake flame of DMC+5%TMP represented a larger fuel consumption rate (or stronger evaporation), and this luminous flame due to the phosphorous-containing particles suggested a larger emissivity of the flame body. As DMC and TMP are the typical examples used in the present work, the near-limit oscillatory behaviors above are commonly found in other types of solvent with different types of OPC additions, as shown in the videos of supplementary materials.

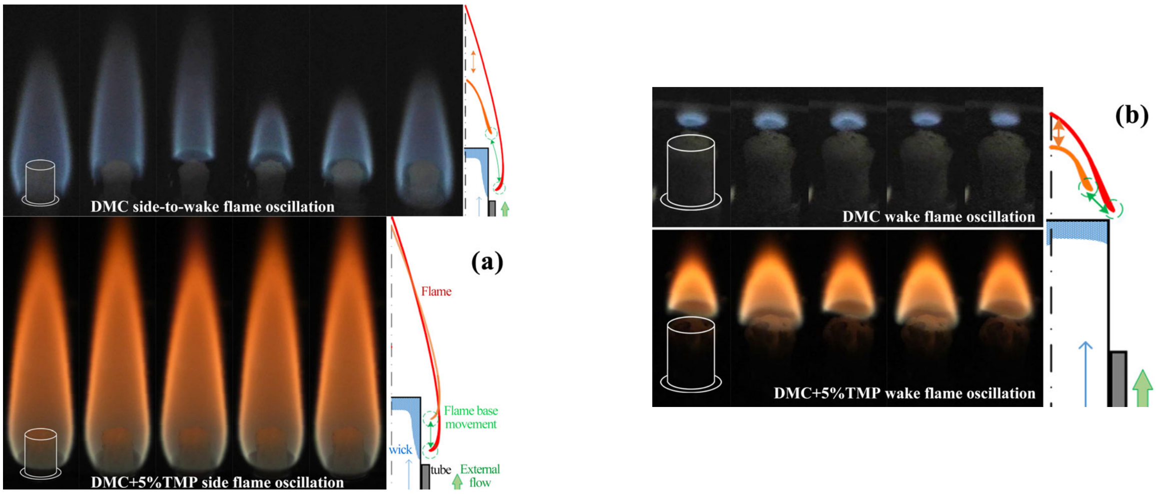

Fig. 4. Sequent images (30-fps) and schematic of near-limit wick flame oscillations. (a) side-to-wake and side oscillation from full flames, Top: DMC (O_2=16.7 vol%), Bottom: DMC+5%TMP (O_2=18.1 vol%). (b) wake flame oscillations, Top: DMC (O_2=15.7 vol%), Bottom: DMC+5t%TMP (O_2=19.5 vol%).

Utilizing the 240-fps camera, the longitudinal movements of flame bases in a representative second were plotted in Fig. 5. To compare the flame base movement of different flames, the 0 mm position in Fig. 5a and 5b were set as the lowest position occurred during each oscillation. In Fig. 5a, no matter frequency or amplitude, the large difference between full flames of pure DMC and DMC with TMP additions is plain to see. The side flame oscillations of DMC with 5% and 10% TMP additions have more than doubled frequency of side-to-wake flame oscillation of pure DMC, while their peak-to-peak amplitudes of the flame base oscillation are one order smaller than that of DMC. Coupling with the buoyancy induced flow and the fuel diffusion around wick geometry, the single cycle of side-to-wake flame oscillation of pure DMC shows a non-asymmetric motion with a faster upward drifting and slower downward propagation. Being consistent with the experimental findings of ethanol lamp flame oscillation [11], the oscillations before the full flame limit can be sustained for a long period, even more than 10 min, with a stable limit cycle. However, the wake flame oscillations cannot persist for a long time, and they were gradually intensified prior to extinction conditions. As shown in Fig. 5b, the amplitudes of different flames are in the same order of magnitude, but the frequency of pure DMC wake flame oscillation is quite lower. All the wake flames oscillated with increasing amplitude and decreasing frequency until extinction, indicating a Hopf subcritical bifurcation of the system [2,26].

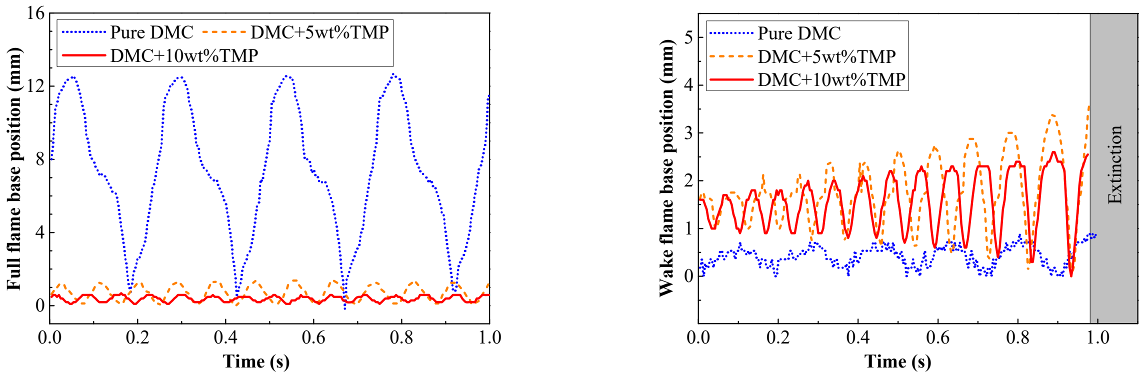

Fig. 5. longitudinal movements of flame base during 1 second oscillation near flame stability limits recorded by 240-fps camera. (a) full flame (b) wake flame.

The addition of TMP in DMC cause the different near-limit oscillatory behaviors for both full and wake flames on a wick configuration. To clarify the effect of TMP addition, the oscillation frequency and peak-to-peak amplitude of were summarized with the change of TMP addition, as shown in Fig. 6. In the figure, each point represents the averaged value of at least four experiments, and the error bars show the deviations from the maximum to the minimum. A boundary at 2 wt% TMP addition was marked according to the switch point of blow-off and quenching regimes found in the previous work [20], and the stability limits of full and wake flames were almost the same. The frequencies of side oscillating flames almost linearly increased from 4 Hz to 14 Hz with the TMP addition from 0 to 10 wt%; while the wake oscillation frequency jumped from 5 Hz to more than 10 Hz with only 1wt% addition of TMP, and the frequency slightly changed with further addition of TMP (up to 10wt%). Different from the common near-limit oscillation frequency (a few Hz) reported in the literature [2,8,10,17], the presence of TMP in the fuel can raise the oscillation frequency to the level of more than 10 Hz, especially for wake flames. By tracking the longitudinal motion of flame bases, the peak-to-peak amplitudes of flame oscillation were plotted in Fig. 6b. The peak-to-peak amplitudes of side flame oscillations declined significantly (from 12 mm to 1 mm) with the addition of TMP. The full flame with high TMP addition can be only sustained in a large volume. Once the full flame size decreases or turns to a wake flame, the combustion will not be sustained, because the heat loss ratio is too large in the flame size change. However, the oscillation amplitudes of wake flames slightly increased with the TMP addition due to the flame size difference. Referring to increasingly oscillating of wake flame before extinction in Fig. 5b, the error ranges of the frequency and amplitude for wake flame (in Fig. 6b) became larger with TMP addition. An obvious deviation of full flame oscillation amplitude was found at 2 wt% TMP addition, because the full flame had both side-to-wake and side oscillations occasionally at the switching point of dominant mechanism where the full flame and wake flame have the same stability limit.

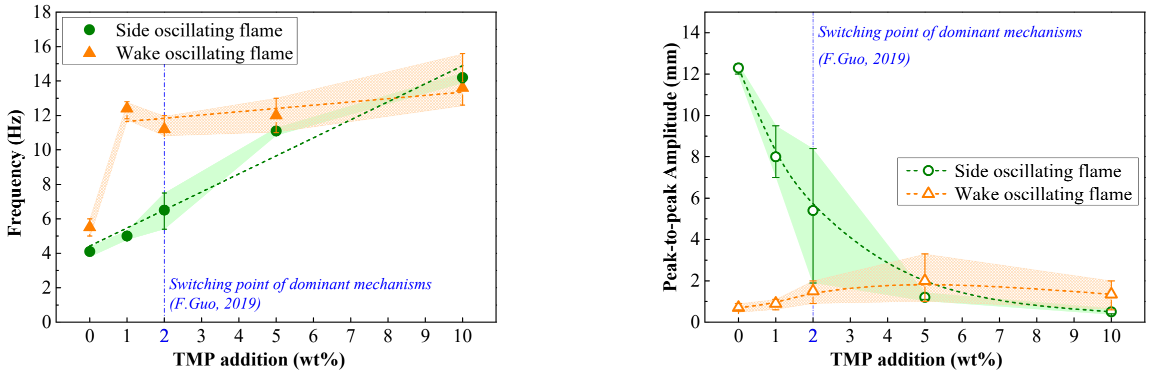

Fig. 6. (a) Frequencies and (b) peak-to-peak amplitudes of near-limit flame base oscillation for DMC with different TMP additions.

To clarify the role of TMP addition on the DMC flame oscillations, the mechanisms controlling the wick flame oscillations should be considered. Different from the flickering flame attributed by Kelvin-Helmholtz instability [2,8], the onset of near-limit flame oscillation always occur when the lowered Damköhler number reaches a critical value [16], and it is significantly affected by the Lewis numbers, heat loss, and equivalent ratios as well [13,15]. In the candle-like wick flame configuration, Chan and T'ien suggested two mechanisms in their early work [1]: one is the oscillatory induced air velocity near the flame base; the other is the periodic fuel evaporation and diffusion, which belongs to the well-known buoyancy-driven instability and thermal-diffusive instability, respectively [2,8,17,18].

Therefore, the laminar burning velocities (S_L) of premixed DMC+TMP gases at each oxygen level of oscillating flames were calculated and plotted in Fig. 7. The upper part of the figure shows the change of S_L change of full and wake flame oscillation with different TMP additions, the lower part presents the oxygen level near the flame stability limits allowing flame oscillations. As the flame edge has a premixed segment, when oxygen reaches the blow off limit of flame, the chemical reaction time becomes comparable with the residence time of local flow. Thus, the same limiting condition should be used to make a fair comparison of the reactivities. The varied limiting oxygen levels in Fig. 7 also correspond to the condition for oscillatory flames in Fig. 5 to Fig. 6. The S_L of both full and wake oscillating flames decreased with the TMP addition, and a cross point appears at 2wt% TMP addition corresponding to the switching point of flame stability limit from the full flame to the wake flame [20]. As found with the flame images in Fig. 4, the buoyancy induced flow should be stronger with the TMP addition because of the increasing flame size. If we considered the flame stabilization mechanism only by the balance of propagation speed and local flow velocity of edge flames, it requires a higher reactivity of edge flame (or reaction kernel [27]) to stabilized the near-limit flame. However, the calculated S_L results gave a contradiction to the hypothesis. It may be attributed to the absence of thermal-diffusive effect on the fuel evaporation. If with the compensation of enhanced fuel evaporation from the wick, the lower S_L can be allowed.

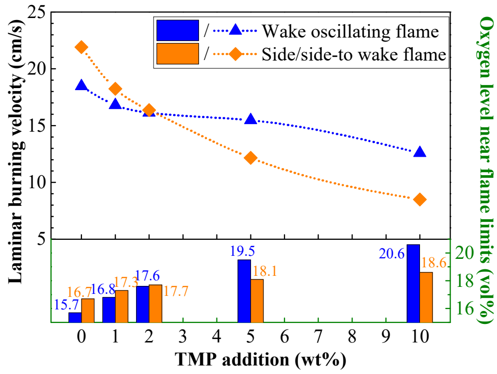

Fig. 7. Calculated laminar burning velocities and corresponding oxygen levels of DMC with different TMP additions prior to the flame stability limits.

To confirm the OPC addition on the fuel evaporation enhancement, the temperature measurement on the wick surface can help. In the wick flame configuration, the evaporation rate is controlled by the heat feedback from the flame and wick surface temperature represents the heat feedback. The flame with OPC addition provides an excessive heat feedback to preheat the wick and intensify the evaporation simultaneously. The higher evaporation rate then supplies more fuel to the flame. It is notable that, even OPC addition were considered to promote the fuel flux, it still increases the LOC of solvent combustion through the dominant chemical inhibition. Figure 8 shows the results of temperature measurement on the surface of the wick during full and wake flame oscillations. The symbols in Fig. 8a are the average temperatures of detected points (indicated at the bottom of the figure) during flame oscillation, the error bars and colored areas indicate the variation range of measured temperature, and the labels reveal the frequency of temperature change. The lines in Fig. 8b represent the intermediate profiles on the center of wick top measured in at least four times, and the colored areas indicate the variation range of measurement results. As the boiling point (BP) of the DMC is around 90 ℃, the wick surface temperature of DMC oscillating flames can hardly reach the BP, and it only has a half value to the BP of pure DMC in the wake flame. The BP of fuel was always adopted for the wick surface temperature in a candle flame modeling [28–30], this choice is reasonable for steady flame analysis but may not suitable for the study of near-limit oscillating flames. By contrast, the wick surface temperature raised significantly (30-40 ℃ for full flames and 20-40 for wake flames) with the additions of 5% and 10% TMP. The higher surface temperature promoted fuel flux, leading to faster periodic fuel evaporation and diffusion surrounding the wick. The variation of the temperature is also consistent with the manner of each flame oscillation, a higher frequency when TMP is added.

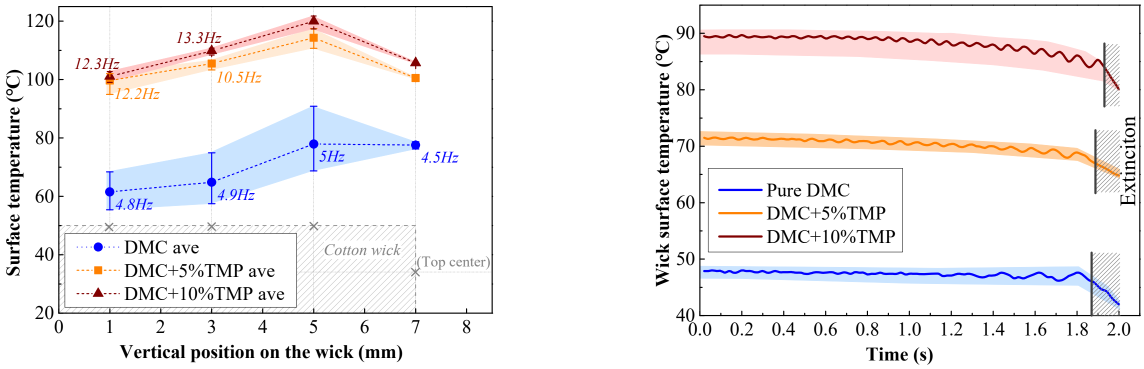

Fig. 8. (a) Averaged wick surface temperatures of flame oscillation prior to the full flame stability limits; (b) Surface temperature profiles on top-center of the wick prior to the wake flame extinction of DMC with 0, 5, 10%TMP additions.

The temperature results supported the hypothesis of thermal compensation to the weakened flame base reactivity by TMP additions. To clarify why the TMP (or other OPCs) can enhance the fuel evaporation, it can be attributed to the chemical and physical effects of OPC involved combustion. As typical flame-retardant additives, the intermediate products of OPCs have chemical inhibition effect in the combustion by capturing the H and OH radicals. However, such exothermic chain termination reactions can potentially cause a higher adiabatic flame temperature [24,31,32]. Besides, the fuel components (like methyl group) of the OPC contribute to the heat release as well, which is called "fuel effect" [33]. Such chemical effects help OPC-added flames have a higher flame temperature (or heat loss). In addition, the particle formation of phosphorous-containing compounds [34] in the flame (reflected by the luminous flame) increase the flame emissivity. Such heat feedback by thermal radiation from the flame to the wick surface can be much stronger than that in the DMC blue flame, which confirmed by the IR emission spectrum in [35]. Such physical effect might be negligible in gas-phase flame when OPCs as suppressants, but it could be considerable in solid combustion scenario when

OPCs as additives. For the oscillation of wake flames, the addition of OPC increased the surface temperature of wick top to enhance the fuel diffusion flux, so the larger wake flame with higher frequency could be found in high TMP-added case. For the full flame oscillation with high OPC addition, the more enhanced fuel flux surrounding the wick surface also made the flame base not have to go far in downstream, because the potential large heat loss ratio of small flame size can lead to global extinction. Therefore, the full flame oscillations with high OPC additions have significant higher frequency and lower amplitude than pure DMC case.

5. Concluding remarks

The near-limit flame oscillations under a candle-wick configuration were experimentally studied with the focus on the addition of TMP (a typical example of OPC additive) in the DMC solvent. Both the side-stabilized and wake-stabilized flames (full and wake flames) performed oscillatory behaviors near the extinction limits. The full flames had stable limit cycle oscillations, while the wake flame oscillations cannot be sustained for long and finally lead to global extinction. With the addition of TMP, the transition from side-to-wake oscillation to side oscillation were found in the unstable full flames, the frequency linearly increased (from 4 to 14 Hz) and amplitude decreased (from 12 to 1 mm) dramatically; however, the wake oscillating flames gave increased trend in both frequency and amplitude.

The laminar burning velocities (S_L) of DMC+TMP mixtures were calculated at the oxygen level of near-limit flame oscillations. The weakened S_L with TMP addition cannot be explained by the buoyancy-driven mechanism sufficiently, and the thermal-diffusive mechanism on the wick can be the compensation of near-limit flame stabilization. The surface temperatures of wick during flame oscillations were measured using a specific thermocouple arrangement, the increased wick surface temperature confirmed the enhancement of fuel evaporation and diffusion in the high TMP addition cases even with flame oscillation.

Even the analytical work on the wick flame configuration is not easy to proceed, the experimental findings in this work can contribute to the candle-like flame modeling and flame-retardant studies of OPCs.

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Figure captions

Fig. 1. Experimental setup schematic of the wick-LOC method [18,19].

Fig. 2. Image processing for the flame boundaries during oscillatory combustion. (a) transient full flame of DMC at 16.7% O_2, (b) transient wake flame of DMC at 16.7% O_2.

Fig. 3. Temperature measurement on the wick surface using fine R-type thermocouple. Left: schematic of probe arrangement, Right: Actual image.

Fig. 4. Sequent images (30-fps) and schematic of near-limit wick flame oscillations. (a) side-to-wake and side oscillation from full flames, Top: DMC (O_2=16.7 vol%), Bottom: DMC+5%TMP (O_2=18.1 vol%). (b) wake flame oscillations, Top: DMC (O_2=15.7 vol%), Bottom: DMC+5t%TMP (O_2=19.5 vol%).

Fig. 5. longitudinal movements of flame base during 1 second oscillation prior to flame stability limits recorded by 240-fps camera. (a) full flame (b) wake flame.

Fig. 6. (a) Frequencies and (b) peak-to-peak amplitudes of near-limit flame base oscillation for DMC with different TMP additions.

Fig. 7. Calculated laminar burning velocities and corresponding oxygen levels of DMC with different TMP additions near the flame stability limits.

Fig. 8. (a) Averaged wick surface temperatures of flame oscillation prior to the full flame stability limits; (b) Surface temperature profiles on top-center of the wick prior to the wake flame extinction of DMC with 0, 5, 10%TMP additions.