Characterization of Smoke Particles Produced from N-heptane / toluene Test Fire: Influence of Different Mixing Ratio

Jia Liu, Qixing Zhang, Feng Wang, Jun Fang, Yongming Zhang
State Key Laboratory of Fire Science, University of Science and Technology of China, Hefei, Anhui 230026, PR China

Abstract

Experiments were conducted in a fire test room to investigate smoke properties of n-heptane/toluene mixtures blended by liquids in different purity and different mixing ratio. Analytical reagent n-heptane (AR, purity ≥ 99.5 %), chemically pure n-heptane (CP, purity ≥ 97 %) and toluene (purity ≥ 99.5 %) were selected to prepare four kinds of mixtures, mixing by volume and by mass were two modes available. Smoke obscuration coefficients were obtained by obscuration meter AML. Size distributions of smoke particles were measured by DMS 500, then count median diameter (CMD) and geometric standard deviation of diameter (GSD) were calculated. Experimental results revealed that the incorporation of toluene resulted in larger obscuration coefficients and CMD. When fuels were mixed in a same mass ratio, slight gap in purities could also affect smoke obscuration. Fuels mixed by mass could not be employed as test fire, while CP n-heptane could be selected to prepare fuels for test fire.

Keywords: Test fire, Smoke properties, Pool fire, Mixing ratio, Purity

Introduction

N-heptane/toluene mixture is a common fuel of test fire specified in standards that were employed to examine photoelectric smoke detector, such as GB 4715 [1], EN 54 [2] and UL 217/UL 268 [3-4]. In GB 4715, a national standard in China, fuel requirement for “n-heptane fire” is n-heptane mixed with 3 % toluene, and the needs of purity for both liquids are 99 % at least. Relevant requirements in EN 54 are similar and liquids are mixed by volume. Generally, there are two kinds of n-heptane with different purities, which can be divided into AR grade and CP grade. When purity difference is ignored unconsciously, smoke properties might be different to some extent. Mixing ratio of liquids, another point that was easy to be ignored, might also affect smoke
properties. In other words, there might be some discrepancy between smoke emitted by fuels prepared using CP n-heptane or mixed by mass and the smoke specified in EN 54, and detectors identified by these fuels would bring some fire risk to buildings. Thus, it is important to quantify the effects of mixing ratio and purity on smoke properties.

In order to obtain a deep understanding of characteristics of smoke generated by test fire, researchers have carried out many studies [5-10]. However, few researches focused on the effect of mixing ratio and fuel purity on smoke properties. The objective of this paper is to investigate the influence of fuel purity and mixing ratio (mixed by mass or by volume) on characteristics of smoke produced from n-heptane/toluene mixture.

**Experimental**

As shown in Fig. 1, all experiments were conducted in a fire test room as specified in EN 54. Fuel container is also made in accordance with EN 54. DMS 500 Fast Particulate Spectrometer was used to measure size distribution of smoke particles ranging from 4.8-1000 nm.

![Fig. 1. A schematic of experimental set up.](image)

CP n-heptane, AR n-heptane and toluene were selected to prepare fuels. Total mass of fuels were 650 ± 0.6 g. Mixtures for Test 1 were prepared in accordance with EN 54. In Test 2, only AR n-heptane was ignited. As for Test 3 and Test 4, fuels were mixed by mass, mass fraction of n-heptane is 97 % and toluene is 3 %. Mass of each component were shown in Table 1. At least three replicate runs were conducted for each condition to ensure the reproducibility of the results. Result deviations between different runs of a same condition are reflected in later graphs by error bars. Ambient temperatures in all the experiment were ranging from 23.4 °C to 25.6 °C, and relative humidity were 75 % - 90 %.
Table 1. Fuels in different experimental conditions.

<table>
<thead>
<tr>
<th>Experiments</th>
<th>CP n-heptane [g]</th>
<th>AR n-heptane [g]</th>
<th>Toluene [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test 1</td>
<td>-</td>
<td>625.5</td>
<td>24.5</td>
</tr>
<tr>
<td>Test 2</td>
<td>-</td>
<td>650</td>
<td>-</td>
</tr>
<tr>
<td>Test 3</td>
<td>630</td>
<td>-</td>
<td>20</td>
</tr>
<tr>
<td>Test 4</td>
<td>-</td>
<td>630</td>
<td>20</td>
</tr>
</tbody>
</table>

Results and Discussion

Obscuration coefficients

Time series of obscuration coefficients are shown in Fig. 2, starting from 60 s before ignition. About 10 s after ignition, compared with background values from -60 s to 0 s, obscuration value grew obviously. There was response delay because smoke transmission took a little time, and obscuration coefficients reached the largest values short time after flame went out. From 350 s to the end, settlement of large smoke particles causing smoke concentration around AML decreased, that is the mean reason why extinction values declined.

Test 1 has the biggest peak value, followed by Test 4 and Test 3, smoke emitted by AR n-heptane alone in Test 2 has the smallest extinction ability. Comparison of obscuration profiles for Test 1 and Test 4 shows that there is a remarkable difference between the concentrations of smoke emitted by mixtures in different mixing mode, either from the point of view of peak value or its growth rate. In addition, as for fuels mixed in a same mass ratio while using n-heptane with different purities, such as Test 3 and Test 4, there is also an evident distinction in obscuration peak, indicating that even small gaps in purity could affect concentrations of fire smoke.

![Fig. 2. Time series of smoke obscuration coefficients.](image-url)
As mentioned, CP n-heptane and fuels mixed by mass might by selected unconsciously to test detectors. To assess these potential fire risks, extinction profiles for Test 1, Test 3 and Test 4 are illustrated in Fig. 3, as well as lower and upper limits used to check out the effectiveness of test fire. Extinction profiles shown in Fig. 3 are from 0 s to 240 s, in accordance with detectors test duration specified in EN 54. Test 3 couldn’t be used as test fire because obscuration values are less than lower limit some time. Extinction profile for Test 4 is higher than Test 3, but this curve is so close to lower limit that is in critical state. Strictly, Test 4 also couldn’t be employed as test fire. In short, fuels mixed by mass couldn’t be selected as test fire. Finally, based on the high degree of similarity of obscuration profiles from 0 s to 240 s for Test 3 and Test 4, it can be inferred that CP n-heptane/toluene mixed by volume could be selected as test fire, because small gap in purity would not affect extinction ability too much during detector test process (0 – 240 s).

Fig. 3. Obscuration profiles and limits specified in EN54.

**Size distribution**

In Fig. 4, starting from ignition, evolutions of smoke particle size distributions at the position of 3m-circle for one run of Test 1 were shown in different time step. Size distributions of smoke emitted in Test 1, which was strictly complied with EN 54, followed lognormal distribution. Profiles were relatively compact, most particles located in the region from 60 nm to 400 nm. As fire increased, large amounts of particles were released, total number of particles increased significantly. After flame was extinguished, there were no more fresh small particles, and collisions between particles caused agglomeration phenomenon, the combination of these two effects caused diameters of smoke particles in the vicinity of sampling point increased slowly. Generation of large particles through agglomeration also caused concentration decline. It worth noting that some particles were too large and settled
down slowly, as mentioned in previous section, this might also be the reason why concentrations declined at the later stage of test.

Fig. 4. Evolutions of smoke particles size distributions.

CMD and GSD are two parameters used to characterize smoke particles: the former represents mean diameter of particles, while GSD describes the spread of values in distribution. Fig. 5 shows time series of CMD for all four fuels. Values of CMD at time t are average from t-50 to t. From 850 s to 900 s after ignition, values of CMD were: 245-258 nm for Test 1, 232-237 nm for Test 2, 239-254 nm for Test 3, and 242-254 nm for Test 4.

From these differences between mixtures and single n-heptane, it could be inferred that smoke emitted from toluene burning had great influence on smoke produced by blended fuels. At the final stage of experiments, CMD for Test 1 was larger than CMD for Test 4, indicating that fuels
mixed by volume produced bigger particles than fuels mixed by mass, and this could be attributed to mass discrepancy of toluene. As for mixtures prepared in a same mass ratio, such as Test 3 and Test 4, this difference could be ignored, indicating that purity gap has little impact on the value of CMD.

Fig. 5. Time series of CMD values for all four fuels.

From profiles of GSD for all four fuels shown in Fig. 5, it was obvious that GSD decreased gradually over time, indicating that smoke particles became more mono-dispersed [11]. Because there were no more fresh small particles after the combustion was finished. In addition, small particles agglomerated to larger particles through collision, also causing size distributions of smoke particles became much more compact.

Fig. 6. Time series of GSD values for all four fuels.
Conclusions

Major conclusions are as follows:

(1) During the combustion in Test 1, which is strictly complied with EN 54, soot particles were released continuously, total number of particles increased dramatically. After flame was extinguished, there were no more small fresh soot, and collisions between particles caused agglomeration phenomenon. Diameters increased slowly and smoke concentration declined. The decrease of GSD indicating that soot particles were mono-dispersed more and more.

(2) When mixing mode changed from volume ratio to mass ratio, obscuration coefficient and CMD were smaller. The incorporation of toluene had significant influence on smoke properties, and essential reason for the differences caused by mixing mode is because toluene mass in mixtures were different. As for fuels mixed by a same mass ratio, slight gap in purities could also affect smoke obscuration coefficient but purity gap affects size distributions and parameters little.

(3) Obscuration profiles for Test 3 and Test 4 indicated that, strictly, fuels mixed by mass could not be employed as test fire. In addition, purity gap affect smoke extinction very little during detector test process (0-240 s), it could be inferred that CP n-heptane could be selected to prepare fuels for test fire.

Acknowledgement

This work was supported by the National Natural Science Foundation of China under Grant No. 41675024, the National Key Research and Development Plan under Grant No. 2016YFC0800100, and the Fundamental Research Funds for the Central Universities under Grant No. WK2320000032. The authors gratefully acknowledge all of these supports.

References


