

Bench Scale Generation of Smoke Particulates and Hydrocarbons from Burning Polymers

ANNA A. STEC, and JENNIFER RHODES
Centre for Fire and Hazard Science
University of Central Lancashire
Preston, Lancashire, PR1 2HE, UK

ABSTRACT

The soot and hydrocarbon generation behaviour from four common polymers has been investigated under different fire conditions using different bench scale apparatuses. While well-ventilated flaming typically has high CO₂/CO ratios and high combustion efficiency, in under-ventilated flaming, higher yields of the many products of incomplete combustion are generally observed, including carbon monoxide, soot and hydrocarbons.

Hydrocarbon and soot production from polyethylene, polystyrene, polyamide and polyvinyl chloride were investigated using the steady state tube furnace (ISO TS 19700) under well-ventilated, small under-ventilated and large under-ventilated fire conditions; and in the smoke density chamber (ISO 5659-2) under the three standard test conditions; and the results have been compared to published data from the fire propagation apparatus (ASTM E 2058). The steady state tube furnace and the fire propagation apparatus show good agreement, for hydrocarbon and soot yields. Conversely it proved impossible to identify correlations with these tests for the soot and hydrocarbon yields in the smoke density chamber.

To investigate the soot generation further, the particle size distribution was determined using a cascade impactor. This showed the predominance of 1–5 µm particulates for all polymers, except PVC, in under-ventilated conditions, and a smaller yield and a more even particle size distribution for well-ventilated burning. For PVC, no significant difference was observed for different fire conditions, with no clear trend of particle size distribution.

KEYWORDS: smoke, soot, hydrocarbon, equivalence ratio, fire propagation apparatus, smoke density chamber, steady state tube furnace.

INTRODUCTION

The combustion conditions replicated by a physical fire model have a significant influence on the production of different fire effluents [1]. The yield and nature of the fire effluents are a function of the fuels involved and the prevailing thermal and oxidative conditions of fire stages [2,3]. The yields of combustion products in a bench-scale method also depend on apparatus conditions, such as the fuel/air equivalence ratio (ϕ) or CO₂/CO ratio, whether the decomposition is flaming or non-flaming, the temperature of the specimen and the effluents produced, the stability of the decomposition conditions etc.

Most bench-scale methods used for toxicity testing are designed to reproduce a single fire stage or combustion condition, where real-scale fires simultaneously involve different fire stages in different places, which are changing with time [4]. These methods can be grouped according to their physical arrangement; from closed tests, such as those based on the smoke chamber, and flow through tests, such as the stationary and steady state tube furnaces, to the fire propagation apparatus. Alternatively, the methods can be grouped as those with constant combustion conditions, often achieving a prolonged steady-state period, and those with non-constant combustion conditions. Those with constant combustion conditions are best suited to producing data suitable for comparison and modelling, such as the ISO steady state tube furnace method (ISO TS 19700). Other bench-scale methods have non-constant combustion conditions, such as those in closed or semi-closed chambers exposed to a constant source of heat, including the NBS smoke chamber (ISO 5659). Intermediate between these two extremes are those that can produce partially constant combustion conditions such as the fire propagation apparatus (ASTM E 2058). While the simpler tests are used to set pass/fail criteria for materials and products, there is a clear requirement from fire safety engineering to produce data which can be used as input to predictive models of smoke and fire toxicity [4,5].

The stages of a fire, from non-flaming, to well-ventilated flaming, and finally to under-ventilated flaming, have been classified by ISO (Table 1) in terms of heat flux, temperature, oxygen concentration, CO₂ to CO ratio, combustion efficiency and equivalence ratio ϕ (the actual fuel-to-air ratio divided by the stoichiometric fuel-to-air ratio) [6]. While some real life fires may be represented by a single fire stage, other fires may pass through several different stages [7].

Table 1. ISO classification of fire stages, based on ISO 19706 [7].

Fire stage	Heat (kW/m ²)	Max. temp. (°C)		Oxygen (%)		Equivalence ratio ϕ	$\frac{V_{CO}}{V_{CO_2}}$
		Fuel	Smoke	In	Out		
Non-flaming							
1a. Self sustained smouldering	n.a.	450–800	25–85	20	0–20	-	0.1–1
1b. Oxidative, external radiation	-	300–600		20	20	-	
Well-ventilated flaming							
2. Well-ventilated flaming	0–60	350–650	50–500	~20	0–20	<1	<0.05
Under-ventilated flaming							
3a. Small under-ventilated	0–30	300–600	50–500	15–20	5–10	> 1	0.2–0.4
3b. Large under-ventilated	50–150	350–650	>600	<15	<5	> 1	0.1–0.4

The use of CO/CO₂ ratios can only be used to characterise fire stages for materials which do not contain chlorine or bromine since these elements significantly increase the CO yield in well-ventilated fires [6].

EXPERIMENTAL

In this paper, smoke and hydrocarbon yields are compared from different bench-scale methods and compared with results obtained from the large scale. This paper compares smoke and hydrocarbon yields obtained using a standard fire toxicity assessment, in the steady state tube furnace (BS7990 [8] and ISO TS 19700 [9]), together with NBS smoke chamber, (ISO 5659-2 [10]), with published data from the fire propagation apparatus [11] (taken from Tewarson's [12] formulae for the prediction of toxic product yields as a function of equivalence ratio).

Materials

Four classes of polymeric materials were used in these studies:

- Low density polyethylene (LDPE) (Cleflex, Polimeri Europa)
- Polystyrene (PS) (Atofina GPPS 1540)
- Nylon 6.6 (PA 6.6) (Invista)
- Polyvinyl chloride (PVC) (Doeflex-Vitapol).

The materials were selected to provide a range of different toxic products, showing different yield behaviour as a function of equivalence ratio or CO₂/CO ratio.

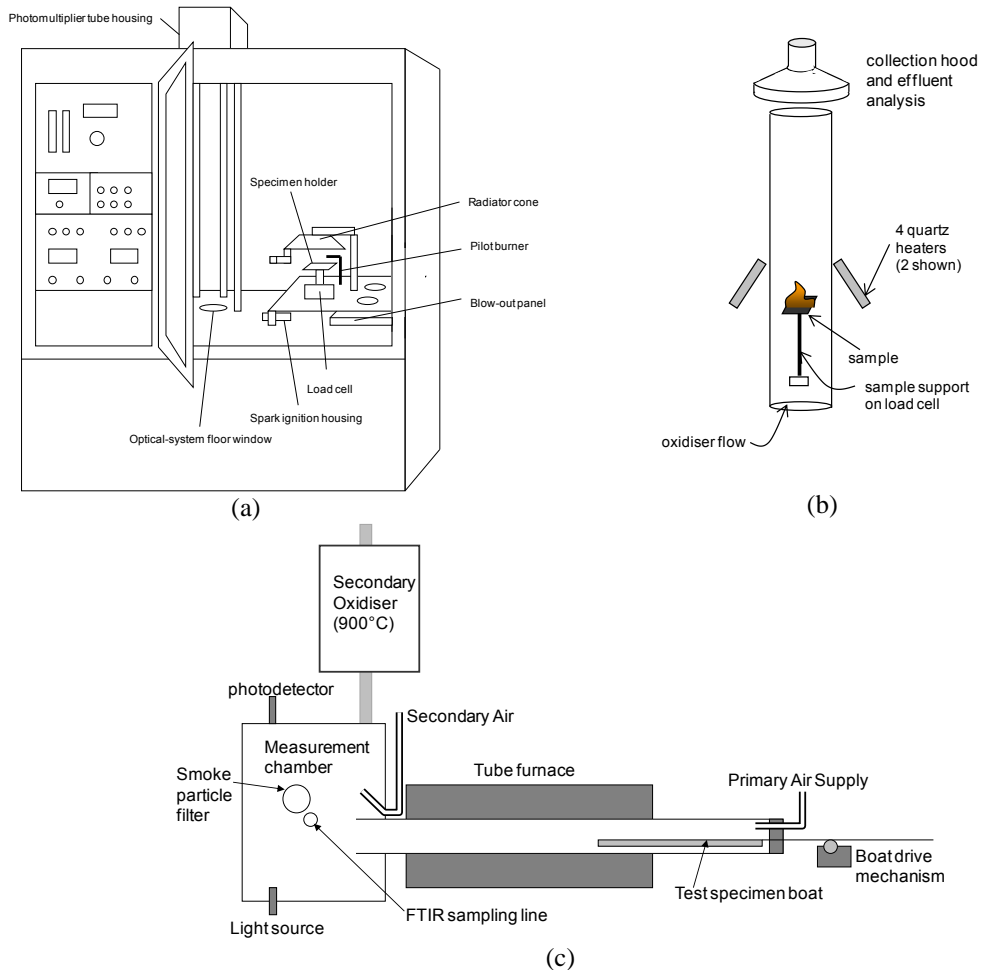


Fig. 1. Test methods used in these studies: (a) NBS smoke chamber; (b) fire propagation apparatus; (c) steady state tube furnace.

The NBS Smoke Density Chamber – ISO 5659-2

The smoke chamber [10,13–15], Fig. 1a, is a well-established piece of equipment, designed to monitor the smoke evolution from burning materials, in order to quantify visual obscuration during a fire. Visual obscuration is measured using a lamp and photodetector at the bottom and top of the chamber, respectively. The method defines smoke generation under flaming (with pilot burner) and non-flaming (without pilot burner) modes, which are reported as the average of the maximum specific optical densities (Table 2) [10,16,17].

Decomposition takes place inside the closed cabinet of volume 0.51 m^3 . In all of these tests, the specimens, 75 mm square and up to 25 mm thick, were exposed to radiant heat (at 25 kW/m^2 or 50 kW/m^2 heat flux respectively) with and without a pilot flame. During the test fire stages may change from 2 to 3a or 3b (see also Table 3) [5]. The flaming fire stage for 25 kW/m^2 irradiation with pilot burner mode may be related between ISO stage 2 (well-ventilated) and stage 3a (small under-ventilated). The test at 50 kW/m^2 irradiation could possibly represent stage 3b (post-flashover flaming) [18].

Three sets of conditions were employed for each polymer using different irradiation conditions, 25 kW/m^2 without pilot flame, 25 kW/m^2 with pilot flame, and 50 kW/m^2 without pilot flame. Before each test samples were conditioned for 24 h at a temperature of $23 \pm 3 \text{ }^\circ\text{C}$ and relative humidity $50 \pm 5\%$ according to ISO 5659-2 test [10]. The gas samples were taken from the NBS smoke density chamber at the time when the maximum optical density was obtained (and determined in preliminary runs) for the burning

polymeric material in order to quantify the products of incomplete combustion using a secondary combustion furnace [4,5].

Table 2. ISO Fire stages represented by NBS smoke density chamber [4].

Heat flux (kW/m ²)	Pilot flame	ISO fire stage
		Depends on test material and thickness and on test duration.
25	No	1b) Oxidative pyrolysis from external radiation
25	Yes	2) Well-ventilated flaming to 3a) Small, Under-Ventilated flaming
50	No	3b) Large Under-Ventilated

Fire Propagation Apparatus – ASTM E 2058

In the fire propagation apparatus (FPA) [11], Fig. 1b, the fire zone is contained within a 172 mm diameter vertical silica tube, allowing better control of the fire atmosphere, and keeping it out of contact with the heaters, which are outside the tube. The effluent flows through an instrumented duct and the rate of heat release is determined from oxygen consumption and the effluent yields can be directly related to the ISO fire stages. A significant advantage of this method is that the air flow and composition in the fire zone is controlled and consequently the apparatus could be used with pre-determined values of equivalence ratio (ϕ) to generate effluent yield data for the different ISO fire stages [12,18–20].

Steady State Tube Furnace – ISO TS 19700

The steady state tube furnace method (SSTF), Fig. 1c, is used in the generation of fire effluent for the identification and measurement of its constituent combustion products, in particular, the yield of different fire effluents under a range of fire conditions according to standards: BS 7990 [8], and ISO TS 19700 [9]. This test was designed to replicate real fire conditions, by using a range of different temperatures and airflow rates [21]. It is possible to reproduce all the different fire stages and types, including low-temperature non-flaming oxidative decomposition, well-ventilated flaming ($\phi < 0.75$) and high-temperature under-ventilated flaming decomposition conditions ($\phi > 2$) [22].

Table 3. Fire conditions corresponding to characteristic stages of burning behaviour.

Fire type defined by equivalence ratio: $\phi = \frac{\text{actual fuel to air ratio}}{\text{stoichiometric fuel to air ratio}}$	Temperature (°C)	Fire type	Primary air flow (L/min)
N/A	350	1b) Oxidative pyrolysis	2
$\phi < 0.75$	650	2) Well ventilated flaming	10 ^a
$\phi > 1.50$	650	3a) Small under-ventilated flaming fires	Twice stoichiometric fuel/air ratio
$\phi > 1.50$	825	3b) Fully developed under-ventilated fires	Twice stoichiometric fuel/air ratio

^a subject to verification of ventilation condition.

The sample is spread evenly along a long silica boat of 800 mm and fed into a tube furnace at a typical rate of 1 g min⁻¹ with flowing air. As it enters the furnace, the temperature (and applied heat flux) increases until ignition occurs. Once the flame stabilises the apparatus forces combustion at fixed fuel feed rates and primary air flow rates. Secondary air is added in a mixing chamber to give a total gas flow of 50 L min⁻¹. The use of a high secondary air flow usually permits the required gas samples to be taken during a single run. Smoke obscuration may also be determined. In addition to analysis of the gases specified in ISO 13344 (CO₂, CO, O₂, HCN, NO_x, HCl, etc.) there is a requirement to determine the total hydrocarbons. This may be achieved by passing part of the air-diluted test effluent through a secondary combustion furnace to allow the determination of the products of incomplete combustion by using a secondary combustion furnace (secondary oxidiser) by further oxidation at 900 °C in excess air over silica wool.

RESULTS

Small-Scale Comparison – SSTF, FPA and NBS Smoke Chamber

Figure 2 compares yields from the steady state tube furnace (SSTF) with predictions based on yields from the fire propagation apparatus (FPA). The relationships proposed by Tewarson [12,20], based on results obtained using FPA for prediction of the yields of fire products as a function of the equivalence ratio, ϕ , have been used to compare the yields of smoke and hydrocarbons for four polymers.

It can be seen that for the most of polymers the smoke yields from the SSTF results show good agreement with FPA predicted results. In Fig. 2a, the low smoke yield from LDPE is consistent in both apparatuses. There is a small increase with equivalence ratio from a yield of 0.06 to 0.1 g/g for the large under-ventilated condition, and the FPA. For the small under-ventilated condition there is a decrease in smoke yield above $\phi = 1.2$.

In Fig. 2b for PS there is also good agreement between the two tests and fire conditions for well-ventilated flaming, but a more pronounced upward trend is observed in the FPA with under-ventilation. The lower smoke yields formed for PS in under-ventilated conditions in the SSTF are consistent with the lower CO yields reported [23].

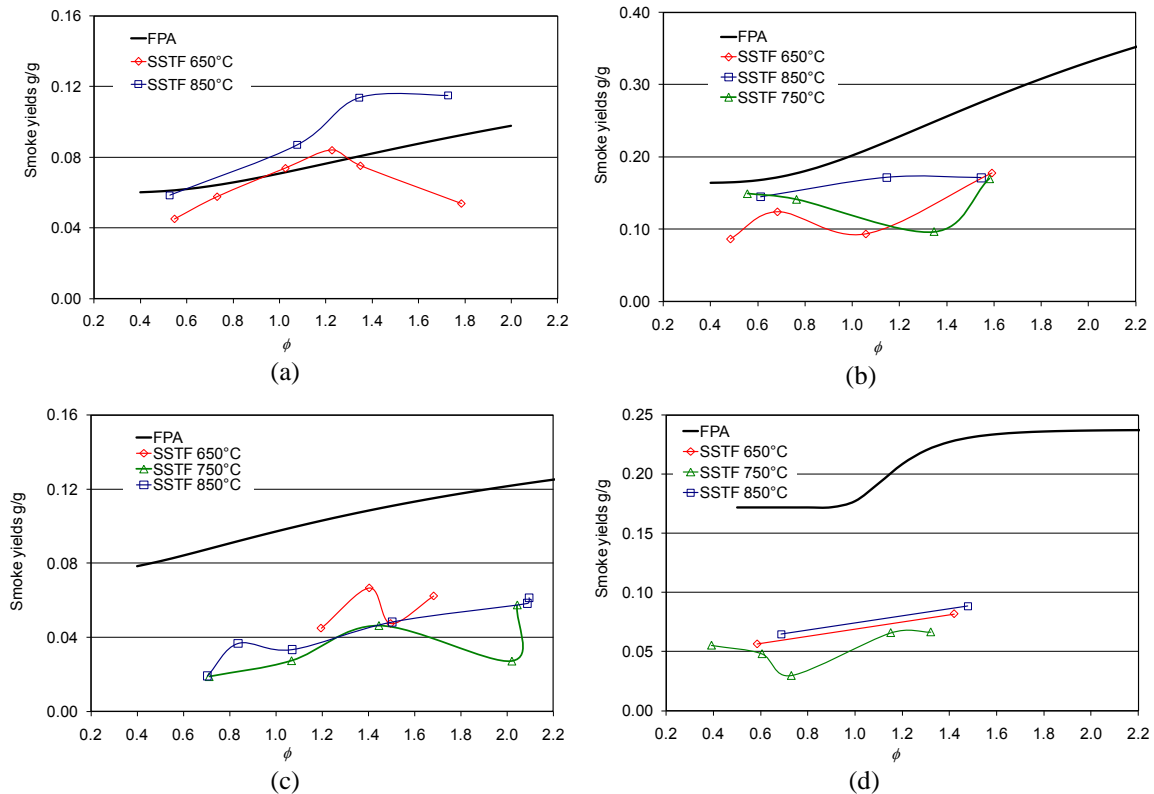


Fig. 2. Smoke yields comparison from the SSTF and FPA: (a) LDPE; (b) PS; (c) PA 6.6; (d) PVC.

Figure 2c shows a progressive increase in smoke yield with ϕ , in both tests and at both conditions for PA 6.6. However, the smoke yields obtained in the FPA are typically double compared to those in the SSTF.

Figure 2d shows greater variation in the smoke yields between the two tests, for PVC. It has previously been reported that the CO yields in the FPA for PVC differ markedly from those in the SSTF in well-ventilated conditions [24]. This is believed to be an artefact of the FPA method, where initial mass loss from PVC is predominantly HCl, giving an apparently low equivalence ratio, but where little fuel is actually evolved.

The hydrocarbon yields from the different polymers show significant differences, as indicated by the different scaling on hydrocarbon axes in Fig. 3. In particular, PS and PVC show much lower hydrocarbon yields in under-ventilated conditions than LDPE or PA 6.6. For most polymers, except PVC, the Tewarson predictions show slightly larger hydrocarbon yields than those found in the SSTF.

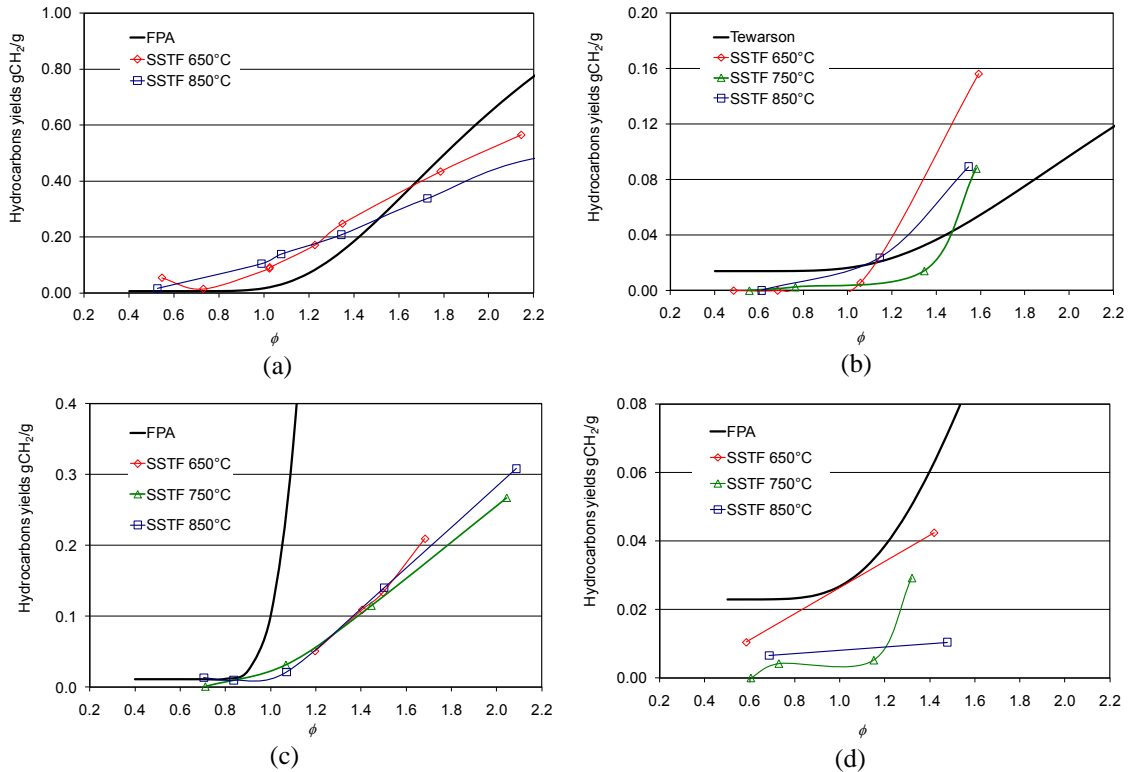


Fig. 3. Hydrocarbon yields comparison from the SSTF and FPA: (a) LDPE; (b) PS; (c) PA 6.6; (d) PVC.

Figure 3a shows very good agreement between the SSTF and FPA under both fire conditions for LDPE. The FPA indicates no hydrocarbon evolution until $\phi > 1$, where the SSTF shows the hydrocarbon yield increasing as $\phi > 0.7$.

Figure 3b shows similarly good correspondence between the two tests for PS. In this case the SSTF shows a greater dependence on increase in equivalence ratio than the FPA.

Figure 3c shows very little dependence on furnace temperature for the increase in hydrocarbon yield for PA 6.6 in the SSTF. The FPA shows an even greater dependency on ϕ , with hydrocarbon yields increasing rapidly when $\phi > 0.9$.

Figure 3d shows reasonable agreement between the two tests for hydrocarbon evolution from PVC, but with almost double the actual yields being observed in the FPA compared to the SSTF.

The fire condition can also be characterised by its CO₂/CO ratio (Table 1 and Table 2). The CO₂/CO ratio is plotted as a function of hydrocarbon yield obtained from the SSTF; and the NBS smoke chamber, for oxidative pyrolysis (25 kW/m² without pilot) in Fig. 4 and for flaming conditions (25 and 50 kW/m² with the pilot burner), in Fig. 5.

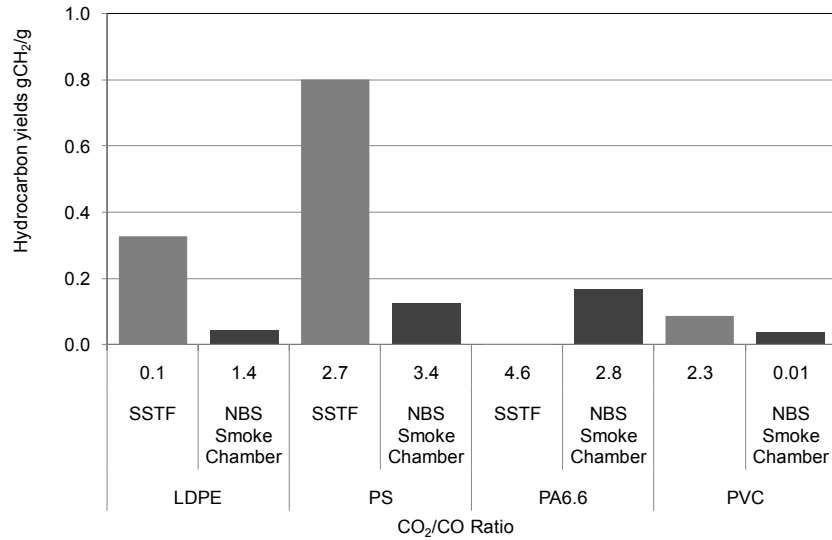


Fig. 4. Hydrocarbon yields comparison from the SSTF and NBS smoke density chamber for the oxidative pyrolysis conditions.

For LDPE samples, the hydrocarbon yield was found to be much more dependent upon combustion conditions, with the lowest yield being generated from 25kW/m² without pilot burner, while almost 7 times more hydrocarbons were generated at 50 kW/m² with the pilot burner. For LDPE, the SSTF results show a significant increase between the hydrocarbon yield of well-ventilated burning and higher yields for under-ventilated fires. Good agreement is shown between hydrocarbons yields obtained in under-ventilated conditions from the NBS smoke density chamber and the SSTF.

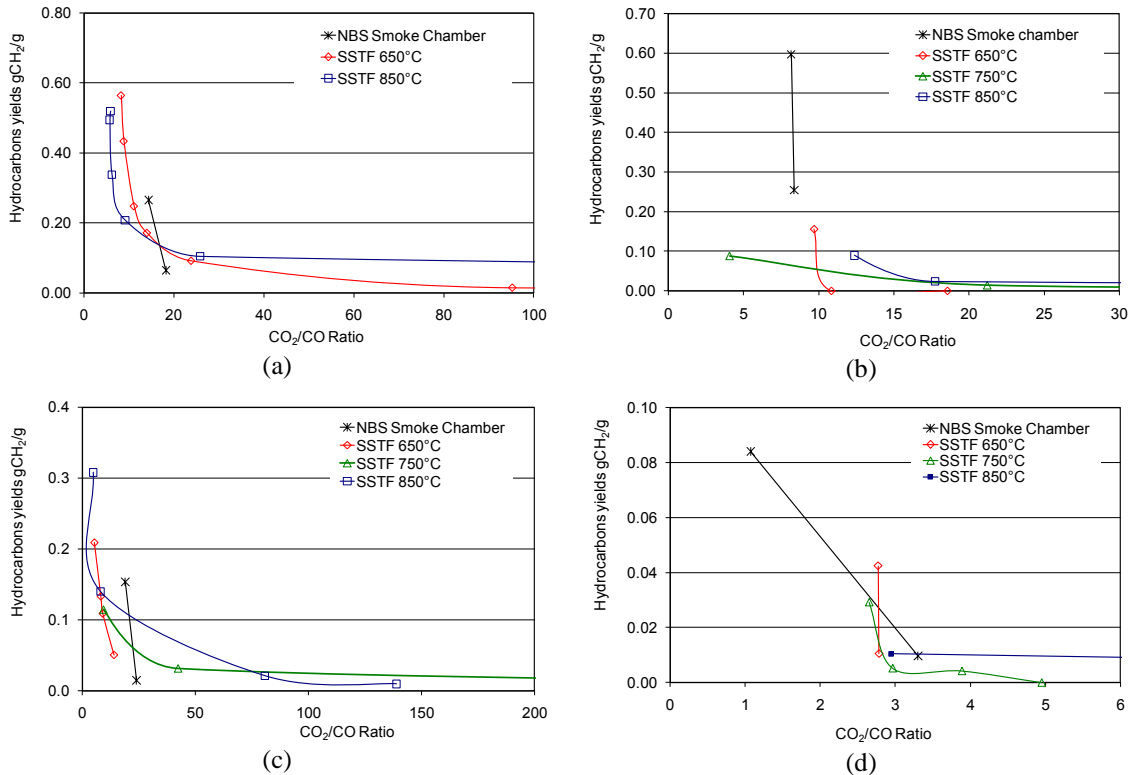


Fig. 5. Hydrocarbon yields comparison from the SSTF and NBS smoke density chamber under flaming conditions (25 and 50 kW/m² with the pilot burner): (a) LDPE; (b) PS; (c) PA 6.6; (d) PVC.

The results for PS from the SSTF give the expected very low hydrocarbon yields under well-ventilated conditions, but high yields in under-ventilated conditions. For PS there is a very high level of organic species in the fire effluent obtained from the NBS smoke density chamber for both ventilation conditions. There is an anomaly between product yields in well-ventilated (25 kW/m² with pilot burner) and under-ventilated fire conditions (50 kW/m² with pilot burner).

In the case of PVC there is little difference between the hydrocarbon yields for the fire conditions in the SSTF. There is a significant increase in hydrocarbons yields between the well-ventilated and under-ventilated flaming conditions for the NBS smoke density chamber. In addition, there is an observed anomaly for the tests under 25 kW/m² with pilot burner where very low hydrocarbon yields are observed.

Similarly to PVC, for Nylon 6.6 all tests show a dramatic increase in hydrocarbon yield from 25 kW/m² to 50 kW/m² for the NBS smoke chamber. However a very similar trend to PVC is observed where the hydrocarbon yields under 25 kW/m² were very low. A consistent trend is observed between the low yields under well-ventilated combustion and the higher yields in under-ventilated combustion in the SSTF.

Cascade Impactor

In order to gain more insight into the formation and production of smoke, a cascade impactor was used to separate the particles by their aerodynamic diameters. It is one of the only techniques which provides a particle size distribution expressed in terms of mass (rather than number) of particles in each size range [25–27]. This is the most relevant parameter for predicting particle transport [28], and health hazard [29].

The cascade impactor was attached to the mixing chamber of the steady state tube furnace. Fire effluent was drawn through at a flow of 2.0 litres min⁻¹ for a period of five minutes during the steady state, and aerosol mass distributions were determined.

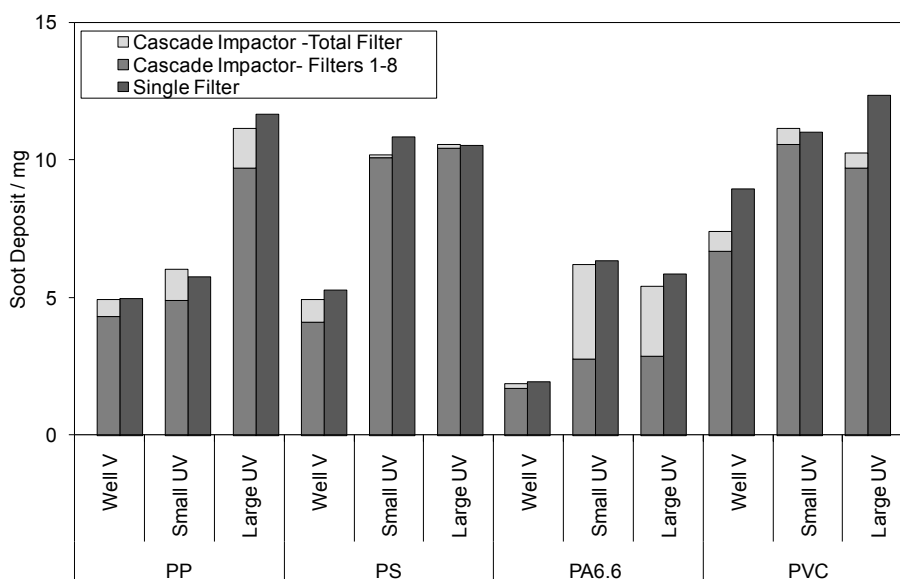


Fig. 6. Total mass of soot collected in the cascade impactor and on the filter for different polymers.

Results are compared for different polymers under various fire conditions in Fig. 6. In order to validate the data the total soot yield was compared to the total distribution of soot collected and separated by the cascade impactor. The cascade impactor total soot estimation was the sum of the soot collected at each stage and included the soot collected by a final porous paper filter. This is located at the end of the cascade impactor filter assembly and collects particles too small to be trapped onto any cascade impactor levels (smaller than 0.5 μm). This was compared to the total amount of soot on the filter paper taken from the mixing chamber, also with a flow of 2.0 L min⁻¹ for five minutes during steady state burning.

The mass determined on the single filter shows good agreement with the total deposited on each of the cascade impactor filters, for each polymers under each fire condition.

For LDPE, PS and PA 6.6 two distinctive patterns of behaviour are observed. In small and large under-ventilated tests there is a predominance of smaller soot particles (1–5 μm) diameter. For PS and PA 6.6 the furnace temperature (650 or 825 $^{\circ}\text{C}$) seems to make little difference to the particle size distribution, where for LDPE the small under-ventilated condition shows intermediate behaviour between well-ventilated and large under-ventilated. In each case the soot yields are lower, and the particle size distribution is very different for the well-ventilated, where there is a more uniform distribution. The exception is PVC, where there is little difference between fire conditions, and no peak in the 1–5 μm range. This is analogous to the higher CO yields under all ventilation conditions observed for PVC, where the radical trapping by HCl prevents the conversion of CO to CO₂ [30]. This implies that the presence of stable halogen radicals also influence the formation and particle size distribution of soot.

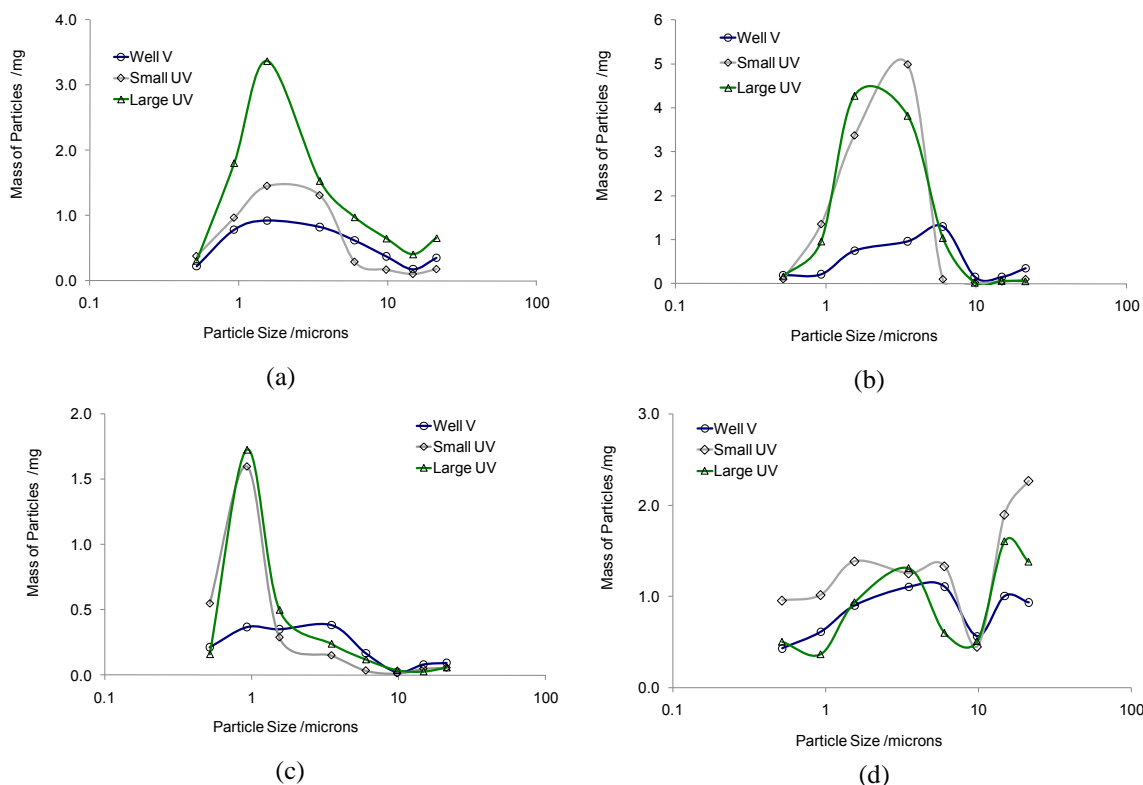


Fig. 7. Soot distribution for different polymers under various fire conditions: (a) LDPE; (b) PS; (c) PA 6.6; (d) PVC.

Figure 7 shows the mass of soot collected on each stage of the filter, expressed in terms of the particle size (or strictly the aerodynamic diameter) using a log scale on the x-axis.

CONCLUSIONS

The predictions reported by Tewarson obtained in the fire propagation apparatus generally correspond well with SSTF, particularly showing similar trends with ventilation condition, but tending to predict slightly higher hydrocarbon yields. Poor agreement with PVC smoke yield in all conditions is believed to be an artefact of the FPA apparatus and particularly the method by which ϕ was calculated.

It is shown impossible to identify correlations with these tests for the smoke and hydrocarbon yields in the smoke density chamber for different polymers.

The particle size distribution data show two clear trends. For LDPE, PS and PA 6.6 there are clear differences between well-ventilated burning and under-ventilated burning, with a peak between 1–5 μm of some of the most dangerous particulates – those able to penetrate deep into the lung – in under-ventilated fires. The differences observed in PVC are intriguing, and given the similarity of behaviour with the independence of CO yield with fire condition suggests that similar mechanism may be involved.

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