

How much the fabric *grammage* may affect cotton combustion?

Jenny Alongi · Fabio Cuttica ·
Federico Carosio · Serge Bourbigot

Received: 21 May 2015 / Accepted: 22 July 2015 / Published online: 31 July 2015
© Springer Science+Business Media Dordrecht 2015

Abstract The present article is addressed to investigating the effect of different fabric *grammages* (mass per area unit) on cotton combustion. To this aim, 100, 200 and 400 g/m² cotton fabrics were tested when exposed to (1) two different heat fluxes (25 and 35 kW/m²) under a cone calorimeter, (2) a methane flame in horizontal or vertical flame spread tests or (3) a propane flame in Limiting Oxygen Index tests, and (4) when pyrolysed and further oxidised in pyrolysis-combustion flow calorimetry (PCFC). The collected results demonstrated a precise relationship between fabric *grammage* and cotton combustion behaviour. Indeed, when exposed to a 35-kW/m² heat flux, the higher the fabric *grammage*, the higher the total heat release during combustion was; the opposite trend was observed when the same fabrics were pyrolysed and further oxidised in PCFC. This finding was ascribed to the different scenarios described by these instrumentations; indeed, the cone calorimeter was able to

reproduce cotton combustion in a well-ventilated context in the presence of air (thus, oxygen), while PCFC only represented the combustion of pyrolysis products. However, both techniques indirectly evidenced a linear dependence of char formation as a function of fabric *grammage*: the higher the fabric *grammage*, the larger the amount of char formed was. The same trend was also observed during horizontal and vertical flame spread tests. In conclusion, the present article is intended to show how cotton combustion may be affected by fabric *grammage* as well as how such behaviour is influenced by the experimental conditions in which it is investigated.

Keywords Cotton · Combustion · Cone calorimeter · PCFC · LOI · Flame spread tests

Introduction

Among the fabrics employed in the market, cotton has always been an object of great interest. Its economic significance in the global market is shown by its majority share (over 50 %) among fibres (micro-denier such as polyesters and polyamides, elastomeric and lyocell fibres) for apparel and textile stuffs (Gordon and Hsie 2007; Wakelyn et al. 2006). One of the most important issues to overcome for using it in numerous applications is its high flammability (Horrocks 1983). Many studies have been carried out by academia and industries in this century, as recently

J. Alongi (✉) · F. Cuttica · F. Carosio
Dipartimento di Scienza Applicata e Tecnologia,
Politecnico di Torino and Local INSTM Unit, Viale
Teresa Michel 5, 15121 Alessandria, Italy
e-mail: jenny.alongi@polito.it

S. Bourbigot
Unité Matériaux et Transformations (UMET) - CNRS
UMR 8207, R2Fire Group-Ecole Nationale Supérieure de
Chimie de Lille CS 90108, 59652 Villeneuve d'Ascq,
France

reviewed (Alongi and Malucelli 2015; Lowden and Hull 2013), in particular for replacing the main market targets represented by Proban[®] and Pyrovatex[®] (Horrocks 2011; Alongi et al. 2013b). Among the most innovative solutions, the use of green flame retardants from bio-sources has been also explored (Basak et al. 2014, 2015a, b, c; Malucelli et al. 2014), opening a “new era” for seeking environmentally friendly flame retardants.

Overall, the surface deposition of flame retardants or of a specific coating is the most common methods exploited for reaching the above goal at both the scientific and industrial scale (Alongi et al. 2014a). However, according to a close examination of the scientific literature, generally, no one seems to be interested in the role of cotton as a substrate with its own physical properties, concerning *grammage* and texture, as examples. Indeed, usually, flame-retardant properties are measured as a relative comparison of untreated and treated fabrics, without taking into account that the flame retardants employed in the study may be efficient for thin and not thicker fabrics. However, this aspect can be of fundamental importance, in particular in the case of fabrics or films. On the other hand, also the fabric texture can play a key role, in particular when a flame retardant is designed for blocking cotton combustion when exposed to a flame, regardless of the gas type, flame length or application time. Thus, the spontaneous question that comes to mind is: *how much does the fabric grammage affect cotton combustion?* In addition, *how can cotton combustion be investigated? What instrumentation can be used and what do the collected results mean?* The present article is meant to answer all these questions, highlighting the role of fabric *grammage* on the resulting cotton combustion behaviour by employing different types of instrumentations, thus providing basic and solid knowledge useful for future studies. More specifically, the flammability of 100, 200 and 400 g/m² fabrics has been explored by exposing cotton to: (1) an irradiative heat flux (25 or 35 kW/m²) during cone calorimetry tests; (2) a methane flame during horizontal or vertical flame spread tests; (3) a propane flame in specific conditions of oxygen concentration during LOI tests; and (4) pyrolysis and further oxidisation during PCFC tests. Collected results demonstrated a precise and direct relationship between fabric *grammage* and cotton combustion behaviour.

Materials and methods

Materials

Three scoured cotton fabrics having 100, 200 and 400 g/m² *grammage* (hereafter coded as COT100, COT200 and COT400) were purchased from Fratelli Ballesio s.r.l. (Torino, Italy), washed in Marseille soap, ethanol and diethyl ether, and subsequently dried before testing them. The weave design characteristics of the employed fabrics are described in Table 1, which reports the *grammage* of each fabric, basic weaves and number of threads per unit length assessed by the ISO 3572 (1976) and UNI EN 1049 (1996) standards, respectively. In addition, some pictures of the three fabrics are reported in Fig. 1 (a, b and c for COT100, COT200 and COT400, respectively).

Characterisation techniques

The surface morphology of the treated samples was studied using a LEO-1450VP scanning electron microscopy (SEM; beam voltage: 5 kV). Pieces of fabric surfaces (5 × 5 mm²) and cross sections were cut and pinned up with conductive adhesive tape and gold metallised.

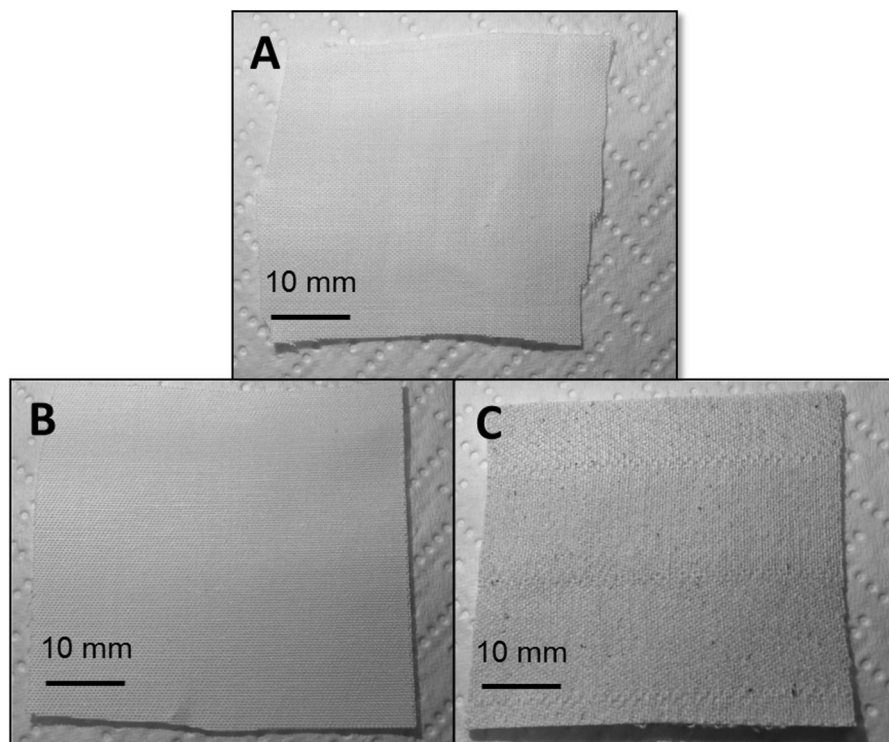
Thermogravimetric analyses (from 50 to 800 °C with a heating rate of 10 °C/min) in both nitrogen and air (60 ml/min for both atmospheres) were carried out for investigating the thermal stability of the different fabrics. To this aim, a TAQ500 thermogravimetric balance was used, placing the samples in open alumina pans (12 mg). The experimental error was 0.5 % for weight and 1 °C for temperature.

LOI tests were performed with a FIRE oxygen index apparatus according to the ASTM D2863 standard (2006). The experimental error was 0.5 %.

Table 1 Weave design characteristics of the fabrics employed in the present study

	<i>Grammage</i> (g/m ²)	Basic weaves	Number of threads per unit length
COT100	100	Twill	Warp 28 Weft 31
COT200	200	Twill 3/1	Warp 27 Weft 30
COT400	400	Twill 4/1	Warp 25 Weft 52

Fig. 1 Pictures of COT100, COT200 and COT400 (**a**, **b** and **c**, respectively)



PCFC (Fire Testing Technology) was used according to the ASTM D7309 standard (2013). More specifically, the cotton specimen (12.0 ± 0.1 mg) was pyrolysed (60 °C/min) under nitrogen (80 ml/min). Subsequently, these latter products were mixed with a 20 ml/min stream of oxygen prior to entering the combustion furnace, where they were burnt at 750 °C for 10 s. The T_{initial} (T at which combustion starts), peak heat release rate (PHRR) and corresponding temperature (T_{PHRR}) and time (t_{PHRR}) and total heat release (THR) were evaluated. These tests were repeated three times, and the experimental error on PHRR and THR was ± 2 %. The instrumental error on T and t was 1 °C and 1 s, respectively.

The resistance to flame exposure was examined by performing horizontal and vertical flame spread tests on rectangular specimens (50×150 mm²): more specifically, a blue methane flame (20 mm length) was applied on the short side of the specimen for 3 s. These tests were repeated three times for each configuration. In the horizontal flame spread tests, the afterflame time (length of time the flame persists after the ignition source has been removed on the basis of ISO 13943 2007, expressed in s), burning rate (mm/s) and afterglow time (length of time that there is

persistence of glowing combustion after both removal of the ignition source and cessation of any flaming combustion, expressed in s) were assessed. More specifically, the burning rate was calculated evaluating the time required to burn the specimen portion between two lines traced at 2.5 and 12.5 cm. The experimental error for these parameters was ± 2 %.

The resistance to a heat flux of square fabric samples (100×100 mm²) was investigated using cone calorimetry (Fire Testing Technology). The measurements were carried out under 25 - or 35 -kW/m² irradiative heat flux in horizontal configuration, following the procedure described elsewhere (Tata et al. 2011) and derived from ISO 5660 (2002), also suitable for cotton fibres (Ceylan et al. 2013). Parameters such as the time to ignition (TTI, s), flame-out time (FO, s), total heat release (THR, kW/m²) and peak of heat release rate (PHRR, kW/m²) were measured. The latter two parameters were also normalised on specimen initial mass and expressed in kg (namely, nTHR and nPHRR). Average [CO] and [CO₂] yields (both expressed in kg/kg) were assessed as well. The experiments were repeated three times for each material investigated to ensure reproducible and significant data; the experimental error was assessed as standard deviation (σ). Prior to combustion

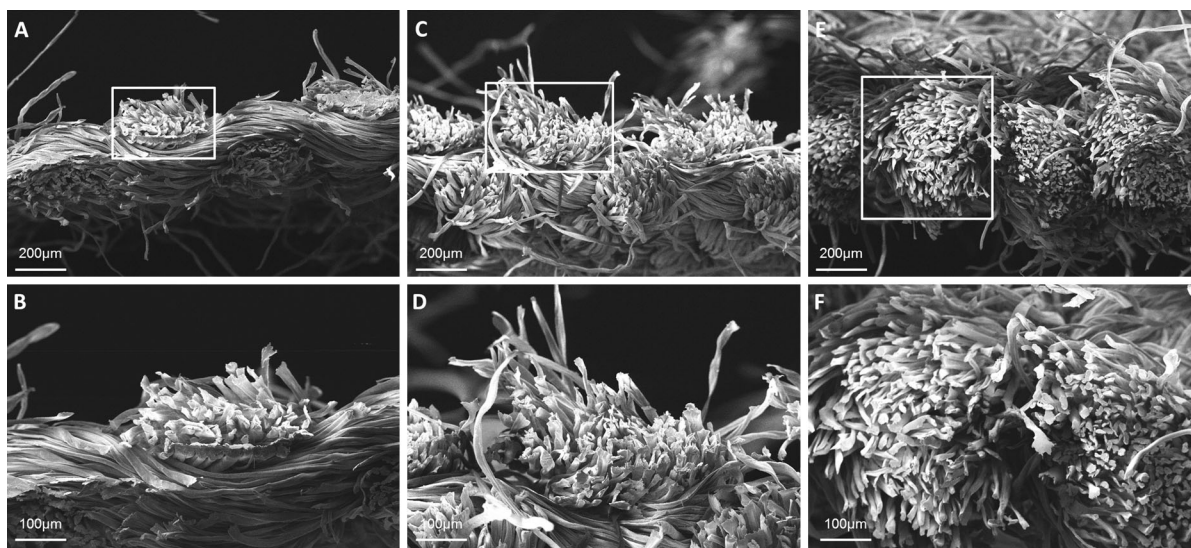


Fig. 2 SEM observations on cross sections of COT100 (a and b), COT200 (c and d) and COT400 (e and f)

tests, all the specimens were conditioned at 23 ± 1 °C for 48 h at 50 % relative humidity in a climatic chamber.

Results and discussion

Fabric texture

Fabric texture of COT100, COT200 and COT400 has been observed by SEM on specimen cross sections and surfaces (Figs. 2, 3, respectively). In detail, COT100 and COT200 have been proven to consist of fibre bundles with comparable sizes (namely, 180 ± 20 and 220 ± 20 μm, respectively), also visible comparing Fig. 2a with c and Fig. 2b with d, respectively. Increasing the *grammage* from 100 to 200 g/m², the bundle number increases as expected. This effect becomes more pronounced in COT400 (Fig. 2e, f), where the disorder level and bundle size are higher (350 ± 50 μm) than those observed in COT100 and COT200. These findings confirm the technical data for the different fabrics reported in Table 1; indeed, the number of threads per unit length is the same in terms of warp, but not in terms of weft (namely, 31, 30 and 52 for COT100, COT200 and COT400, respectively). However, the fibre diameter is almost equal for the three fabrics: namely, 19 ± 5 , 18 ± 4 and 18 ± 5 μm for COT100, COT200 and COT400 (Fig. 3a b, c d, e, f respectively).

Fabric combustion

As already mentioned in the Introduction, cotton flammability was extensively studied in this century and its combustion mechanism was understood (Horrocks 1996; Kandola et al. 1996; Kandola and Horrocks 1999; Mamleev et al. 2009; Bourbigot et al. 2002; Price et al. 1997). This phenomenon depends on the pre-ignition step during which cellulose contained within cotton degrades in air at high heating rates. This occurs through the competition between depolymerisation and dehydration (Horrocks 1996; Shafizadeh and Fu 1973; Shafizadeh and Sekiguchi 1983 and Shafizadeh and Sekiguchi 1984). Depolymerisation induces the formation of highly combustible volatile species (mainly levoglucosan, furan and furan derivatives), which are responsible for cotton ignition (Faroq et al. 1991). On the contrary, dehydration produces the formation of a thermally steady *char* (Morterra and Low 1983, 1984, 1985) having aromatic features (Sekiguchi et al. 1983). The predominance of one of these two processes has recently been proven to be a function of heating rates (Alongi et al. 2013a). Indeed, the higher the heating rate, the more favourable the depolymerisation and thus cotton flammability are. On the other hand, dehydration and thus char formation are favoured when cotton is heated at low heating rates.

In any common standard procedure employed for investigating the combustion of a material, the

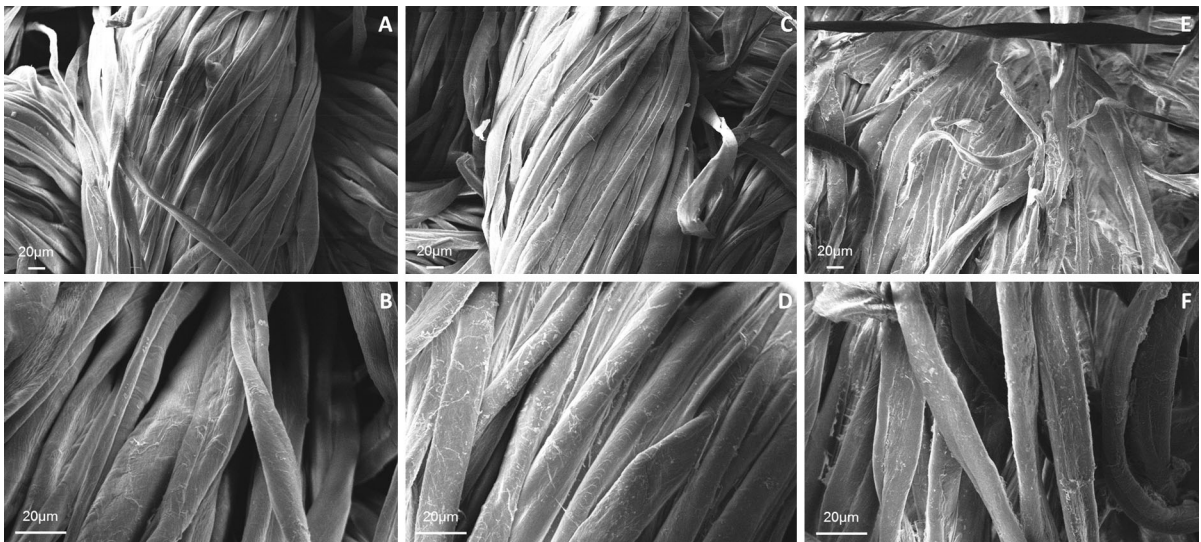


Fig. 3 SEM observations on surfaces of COT100 (a and b), COT200 (c and d) and COT400 (e and f)

adopted heating rate is very high, usually hundreds °C/min as in cone calorimetry, flame spread or LOI tests. This does not occur in PCFC, where the heating rate is generally 60 °C/min (Lyon and Walters 2004; Lyon et al. 2009); in addition, and in contrast with the other aforementioned tests, in PCFC only the combustion of pyrolysis products is carried out (Yang and Hu 2011, Yang and Hu 2012; Yang et al. 2010). Hence, these considerations highlight the different scenarios described by the common characterisation techniques and PCFC, which have found remarkable interest in the last decade, in particular concerning the characterisation of coatings derived from nanotechnology such as layer-by-layer assembly (Alongi et al. 2014a). This does not mean that PCFC is a useless instrument, but it is important to point out that the information derived from it does not describe the same scenario as cone calorimetry or flame spread tests. Indeed, this equipment can be very useful when combined with thermogravimetry, as recently demonstrated (Alongi et al. 2014b), as it is able to measure the combustion heat of pyrolysis products, which is not directly possible by using thermogravimetry.

Under 25 kW/m² heat flux, cotton with different fabric *grammages* behaves in different ways (Table 2). Indeed, COT100 did not give reproducible data: two out of five specimens did not ignite. The amount of material is probably too low (namely, 1.2 g) and thus the amount of volatile species released upon

heating does not reach the flammability limit. Indeed, this does not occur with COT200 and COT400. As a consequence of different *grammages*, COT200 and COT400 exhibited different TTI, FO and combustion durations: the higher the fabric *grammage*, the lower the TTI and FO were. Furthermore, COT400 exhibited a higher THR with respect to COT200 (4.9 versus 1.8 MJ/m²) and an apparently higher PHRR (132.0 versus 69.3 kW/m²), as expected.

The next question arises: *is it correct to compare two specimens having such different masses under cone calorimetry?* First of all, we have to take into account that the cone calorimeter was not designed for fabrics but for thick materials; indeed the balance present in the standard configuration is not as sensitive for thin specimens (Schartel and Hull 2007). In spite of this, we have already demonstrated that it is possible to use this instrumentation for fabrics (Tata et al. 2011) and fibres (Ceylan et al. 2013), but it is necessary to proceed carefully. In this context, for meaningfully comparing COT200 and COT400, it is necessary to normalise parameters such as THR and PHRR on the basis of initial specimen masses rather than surface area exposed. As reported in Table 2, it is clear that the THR trend is not changed (the higher the fabric *grammage*, the higher the total heat release during combustion is) but this does not occur for PHRR. Indeed, taking the experimental error into consideration, the PHRRs of COT200 and COT400 are almost

Table 2 Combustion data of COT100, COT200 and COT400 collected by cone calorimetry

Samples	Mass (g)	TTI $\pm \sigma$ (s)	FO $\pm \sigma$ (s)	THR $\pm \sigma$ (MJ/m ²)	nTHR $\pm \sigma$ (MJ/kg)	PHRR $\pm \sigma$ (kW/m ²)	nPHRR $\pm \sigma$ (kW/kg)	[CO] $\pm \sigma$ (kg/kg)	[CO ₂] $\pm \sigma$ (kg/kg)
Heat flux = 25 (kW/m ²)									
Not reproducible data									
COT100	1.20								
COT200	2.00	62 \pm 0	99 \pm 3	1.8 \pm 0.1	9.0 \pm 0.5	69.3 \pm 4.1	352.8 \pm 27.9	0.1234 \pm 0.0167	1.55 \pm 0.08
COT400	3.90	35 \pm 2	83 \pm 2	4.9 \pm 0.2	12.5 \pm 0.2	132.0 \pm 12.3	338.6 \pm 32.8	0.0563 \pm 0.0039	1.69 \pm 0.04
Heat flux = 35 (kW/m ²)									
COT100	1.20	35 \pm 5	62 \pm 3	1.0 \pm 0.2	8.3 \pm 1.4	60.7 \pm 6	506.1 \pm 50.3	0.0869 \pm 0.0160	1.56 \pm 0.03
COT200	1.90	19 \pm 2	58 \pm 2	2.1 \pm 0.1	11.0 \pm 0.1	92.2 \pm 3.1	476.9 \pm 10.1	0.0805 \pm 0.0074	1.63 \pm 0.05
COT400	3.80	16 \pm 0	53 \pm 1	5.0 \pm 0.1	13.2 \pm 0.2	173.6 \pm 9.9	456.8 \pm 26.1	0.0271 \pm 0.0025	1.74 \pm 0.03

equal; this suggests that the heat release rate measured in the cone calorimeter is not a function of cotton *grammage*, but is a feature typical of the material under investigation.

Another important aspect that should not be neglected is the meaning of THR normalised on mass and expressed in kg; indeed, the cone calorimeter can directly measure the heat release normalised on the specimen mass (this parameter is the effective heat of combustion, EHC), but not in the case of thin materials. Since it is quantified as the ratio between THR and mass at a specific time, the instrumental error for mass would be very high because of the low sensitivity of balance (as already mentioned). Thus, it is possible to overcome this instrumental limit for fabrics by calculating the normalised THR and thus eliminating the mass effect due to samples having a remarkable difference in terms of mass specimen, as in our case.

Furthermore, during combustion, the [CO] yield turned out to be a function of fabric *grammage*, while the [CO₂] yield was almost equal for COT200 and COT400 (Table 2). As reported here, the amount of [CO] yield of COT400 is drastically lower than that of COT200. This may be attributed to a higher formation of char for COT400 than COT200.

The behaviour of these fabrics is ascribed to the different *grammages*, which can affect the irradiative heat transmission during cone calorimetry tests. Indeed, monitoring the transmitted heat flux, it is possible to observe that the lower the fabric *grammage*, the higher the heat flux transmission is, as is visible in Fig. 4a. Thus, COT400 absorbs a higher heat flux with respect to COT200 and COT100 (Fig. 5a). On the other hand, calculating the heat absorbed per mass unit in 1 s (Fig. 5b), it is possible to observe that COT100 absorbs more heat than COT200 and COT400; thus, reasonably, its temperature and heating rate are higher than those of the other two samples. As a result, the decomposition kinetics of the three fabrics may be significantly different as cotton degradation is governed by a precise equilibrium between the volatilisation and charring processes (Alongi et al. 2013a). The collected data demonstrated that the higher the absorbed heat (the higher heating rate), the more volatile species are released and the lower the char formed. In conclusion, the higher the fabric *grammage*, the higher the char formed is.

By increasing the heat flux to 35 kW/m² (Table 2), COT100 gave reproducible data, as did COT200 and

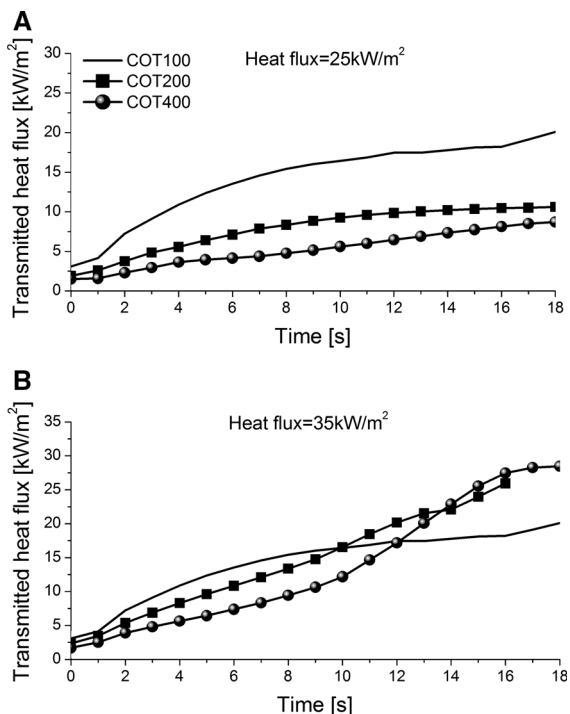


Fig. 4 Transmitted heat flux measurements of COT100, COT200 and COT400 under 25 (a) and 35 kW/m² (b)

COT400. Once again, TTI and FO are non-linearly dependent on fabric *grammage*, as already observed under 25 kW/m². Conversely, THR, nTHR and PHRR increase by increasing fabric *grammage*. Also in this case it was confirmed that the heat release rate is a material characteristic, as demonstrated by nPHRR values, and that the *grammage* affects the heat transmission (Fig. 4b) and adsorption (absorbed heat flux in kW/m² and J/g/s in Fig. 5c, d respectively).

At the end of these tests, independently of heat flux and fabric *grammage*, cotton does not leave any residue. In conclusion, fabric *grammage* may affect the parameters assessed by cone calorimetry, regardless of the adopted heat flux; indeed:

1. the highest TTI was found with the lowest fabric *grammage*,
2. the lowest THR (and nTHR) was found with the lowest fabric *grammage*, and
3. only nPHRR should be taken into consideration for fabrics as it should be an intrinsic characteristic of cotton.

The effect of fabric *grammage* on cotton combustion has also been investigated by horizontal and

vertical flame spread tests. These two configurations are drastically different in terms of combustion kinetics (namely, afterflame time and burning rate); indeed, when cotton is exposed to a methane flame in a horizontal configuration, it burns more slowly than in a vertical configuration, as is clear when comparing the snapshots in Figs. 6 and 7, respectively. More specifically, when cotton is horizontally burnt, it is possible to measure the afterflame and afterglow times and the burning rate. This is almost impossible in a vertical configuration; thus, only a qualitative observation of the test was performed in this work.

In detail, after removing the ignition source, in a horizontal arrangement COT100, COT200 and COT400 burnt slowly under flaming combustion for 51, 78 and 141 s (afterflame time in Table 3), leaving a very thin residue that undergoes glowing combustion for 9, 11 and 34 s, respectively (afterglow time in Table 3; Fig. 6). As expected, by increasing the fabric *grammage*, the combustion duration (intended to be the sum of the afterflame and afterglow times) increases, while the burning rate decreases. Indeed, the more dense and compact the fabric, the more slowly it burns, as is visible when comparing the snapshots in Fig. 6. As a consequence, the time for reaching the 12.5 cm line is very different: 57, 78 and 171 s for COT100, COT200 and COT400, respectively.

From a visual observation, the same behaviour of COT100 and COT200 was found when fabrics were placed in a vertical arrangement. They burnt completely and vigorously in approximately 15 s; the last 3 s involved the afterglow, which totally consumed the remained specimen, leaving no residue (Fig. 7). On the other hand, COT400 exhibited a different behaviour: indeed, after removing the flame, COT400 combustion was focussed on a smaller area with respect to the other two fabrics, as evidenced when comparing the snapshots at 0 s reported in Fig. 7. After 5 and 10 s, combustion involves a smaller area with respect to that burnt in COT100 and COT200. At 15 s, COT100 and COT200 are totally consumed, while COT400 goes on burning for another approximately 5 s in the presence of flame and another 3 min as glowing combustion, leaving a very thin and impalpable residue, the amount of which is however negligible. As was predictable, the burning rate observed in these tests was significantly higher than that observed in the horizontal configuration, but once again was conversely dependent on fabric *grammage*:

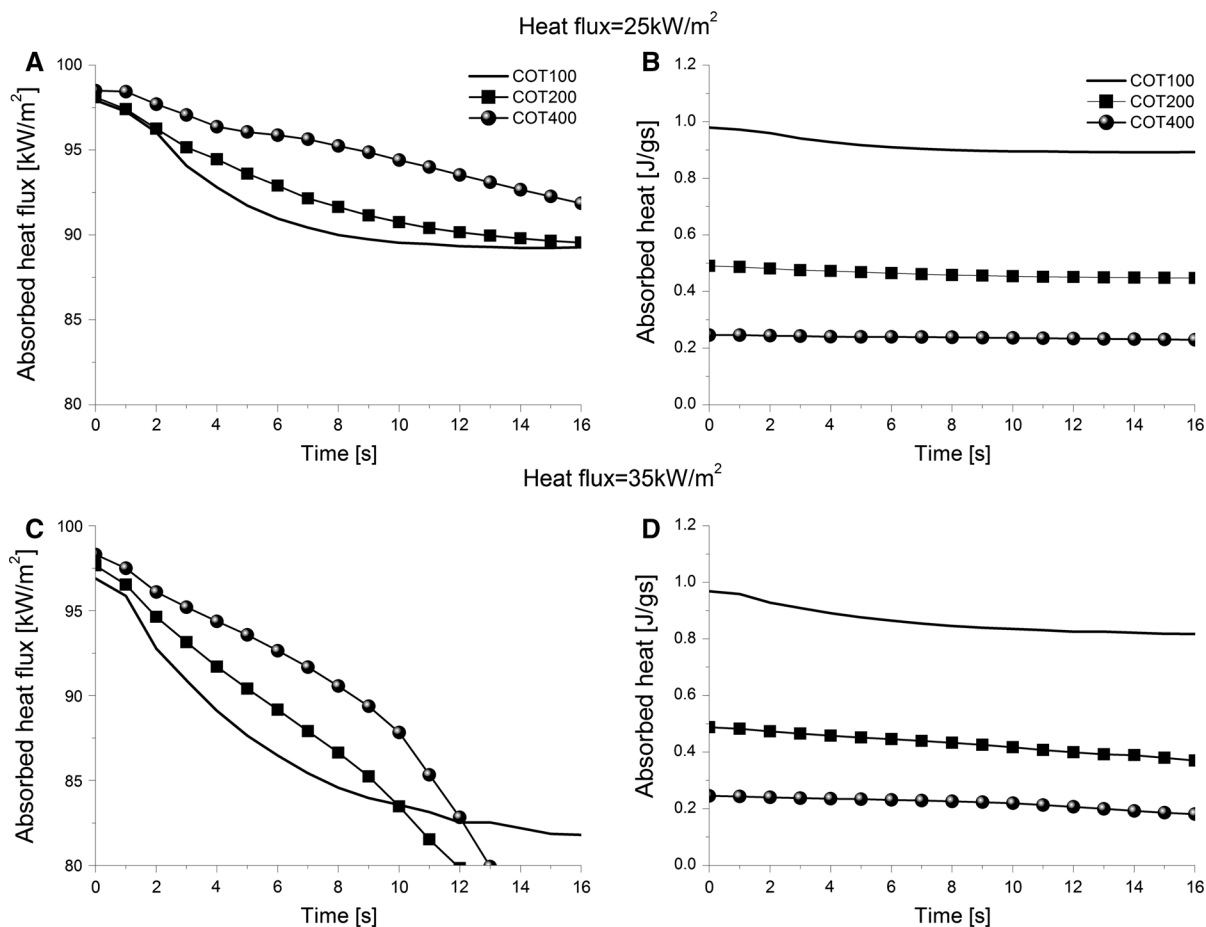


Fig. 5 Absorbed heat flux measurements of COT100, COT200 and COT400 (a and c) and calculated absorbed heat (b and d)

the higher the fabric *grammage*, the lower the burning rate was. Also in these tests there was a precise dependence of cotton flammability (independently of the adopted configuration) on fabric *grammage*; indeed:

1. the highest afterflame and afterglow times and the lowest burning rates were found with the highest fabric *grammage*.

Beyond flame spread tests, the LOI was commonly used for characterising fabrics; indeed, it has long been considered a good flammability index by both industrial and academic researchers, although it is questionable whether the collected results can be correlated with those of any other test and in particular with those of a real fire scenario (Weil et al. 1992). Indeed, it is common opinion that the LOI can only be

considered a useful tool for defining candle-like ignition and is of no value for real-world fire safety. However, in our opinion, it is important to measure this value as a function of fabric *grammage*. As reported in the last column of Table 3, COT100, COT200 and COT400 exhibited an LOI value of 19.5, 20.0 and 21.5 %, respectively. As the LOI represents the minimum concentration of oxygen in a flowing mixture of oxygen and nitrogen that will just support flaming combustion (ASTM D2863), it should represent an intrinsic characteristic of the material that should not depend on the fabric *grammage* (as studied here) and, as a consequence, on the fabric weave (Table 1). This finding raises some doubts about the real meaning of these tests for fabrics. In conclusion, fabric *grammage* may affect the cotton LOI value; indeed:

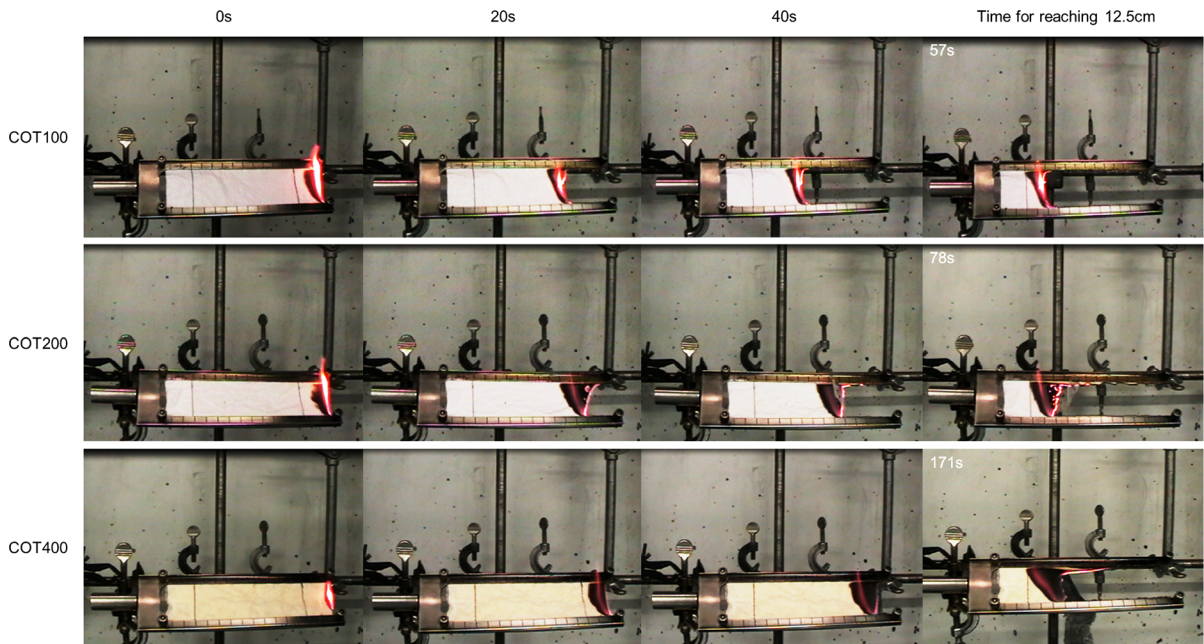
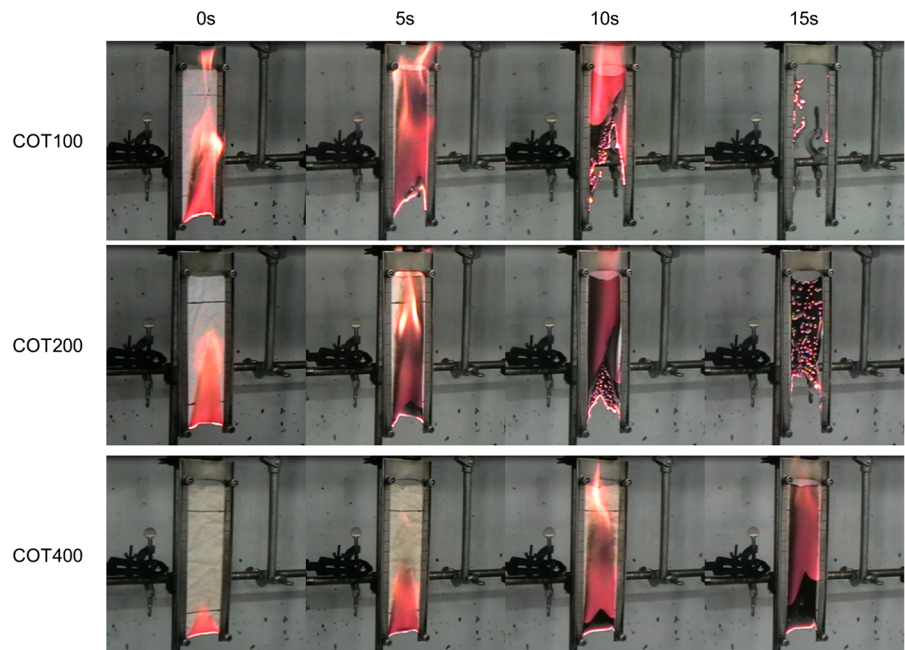


Fig. 6 Snapshots of COT100, COT200 and COT400 at 0, 20, 40 s and the time to reach 12.5 cm during horizontal flame spread tests

Fig. 7 Snapshots of COT100, COT200 and COT400 at 0, 5, 10 and 115 s during vertical flame spread tests



1. the highest LOI was found with the highest fabric grammage.

Referring to the behaviour of cotton when first pyrolysed and further burnt in PCFC, it was possible to observe a completely different behaviour with respect

to that previously described (in particular with the cone calorimeter). Table 4 reports the collected data and Fig. 8a plots the HRR curves of COT100, COT200 and COT400.

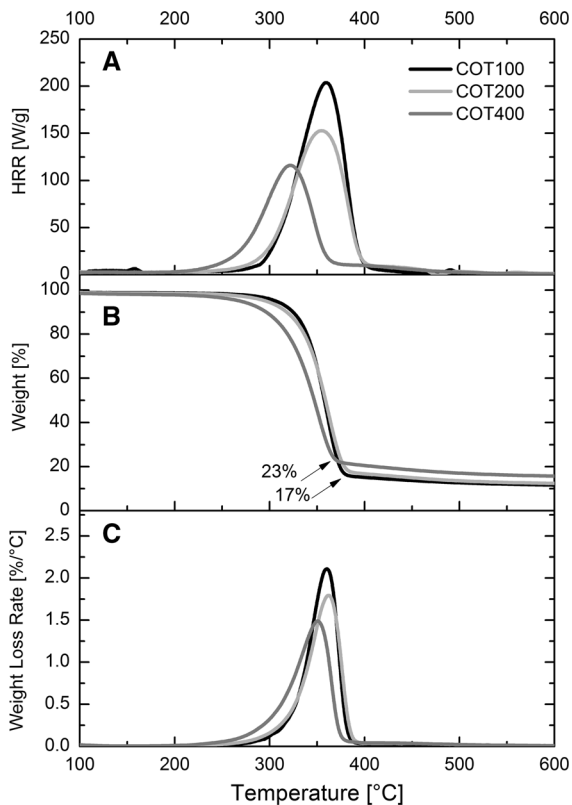
First of all, the $T_{initial}$, THR and PHRR values decreased with fabric grammage increases. This trend

Table 3 Combustion data of COT100, COT200 and COT400 collected by horizontal flame spread and LOI tests

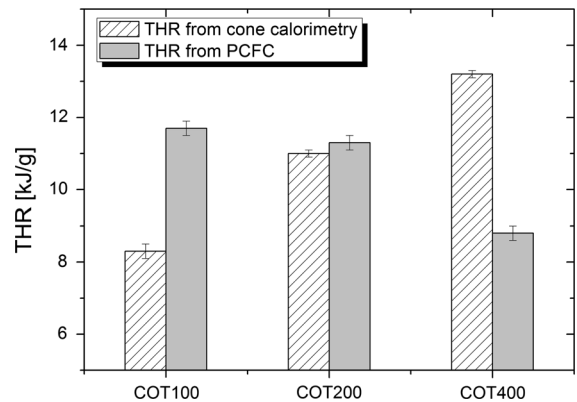
Samples	Weight (g)	Afterflame time (s)	Afterglow time (s)	Burning rate (mm/s)	LOI (%)
COT 100	0.90	51 ± 1	9 ± 3	1.97 ± 0.02	19.5
COT 200	1.45	78 ± 1	11 ± 3	1.28 ± 0.02	20.0
COT 400	2.90	141 ± 3	34 ± 2	0.71 ± 0.02	21.5

Table 4 Data of COT100, COT200 and COT400 collected by pyrolysis-combustion flow calorimetry

Samples	T_{initial} (°C)	THR ± σ (kJ/g)	PHRR ± σ (W/g)	T_{PHRR} (°C)	t_{PHRR} (s)
COT100	273	11.7 ± 0.2	203 ± 4	360	311
COT200	250	11.3 ± 0.2	153 ± 3	354	311
COT400	210	8.8 ± 0.2	116 ± 2	322	278

**Fig. 8** HRR (a), TG (b) and dTG (c) curves (in nitrogen) of COT100, COT200 and COT400

was completely opposite to that exhibited by the fabrics under investigation in cone calorimetry. Figure 9 reports the comparison among THR values collected by these two techniques as an example. In

**Fig. 9** Comparison between THR values curves of COT100, COT200 and COT400 collected by cone calorimetry and PCFC

addition, also T_{PHRR} and t_{PHRR} decreased by the same trend.

This discrepancy between cone calorimetry and PCFC can be easily explained taking into account the char's former character of cellulose contained in cotton fabrics. Indeed, thermogravimetry (Figs. 8b, c, 10, respectively) showed that COT400 forms more char (23 and 35 % in nitrogen and air, respectively) than those left by COT100 and COT200, which is approximately the same (17 and 25 %). This means that a lower amount of volatile species was released by COT400 if compared with COT100 and COT200. Thus, if COT100 produces more volatiles than COT400, more pyrolysis products will be produced and further oxidised in PCFC; as a consequence COT100 THR is higher than COT400 THR (11.7 vs.

Fig. 10 TG (a) and dTG (b) curves of COT100, COT200 and COT400 in air

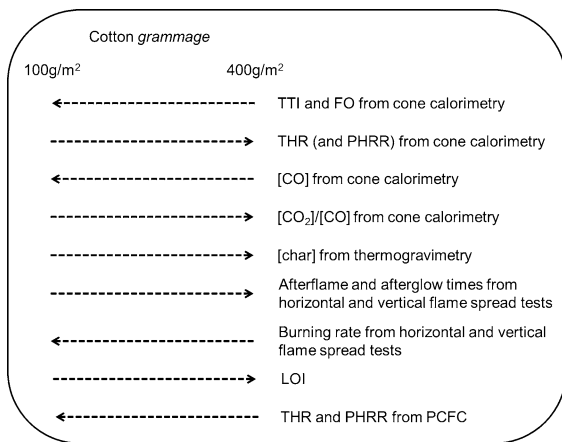
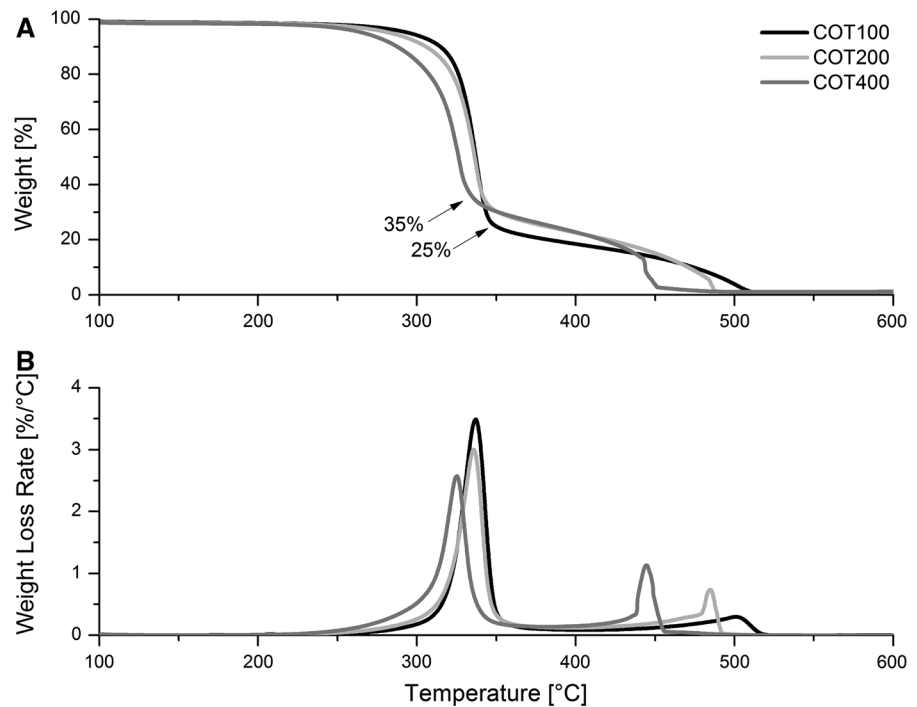


Fig. 11 Relationship between cotton *grammage* and combustion data collected by different characterisation techniques

8.8 kJ/g). Here it is not necessary to normalise THR and PHRR as the measured parameters have been directly given in kJ/g and W/g, respectively. Comparing PHRR values, it is important to highlight that these values are not equal as in the cone calorimeter and are a function of cotton *grammage*, to which the char-forming character is directly connected.

In conclusion, fabric *grammage* may affect the parameters assessed by PCFC; indeed:

1. the lowest THR and PHRR were found with the highest fabric *grammage* (assessed by PCFC), and indirectly
2. the highest char formation was found with the highest fabric *grammage* (assessed by thermogravimetry).

Conclusions

In the present manuscript, cotton fabrics with 100, 200 and 400 g/m² *grammage* were tested by cone calorimetry, (horizontal and vertical) flame spread and LOI tests, PCFC and thermogravimetry (in nitrogen and air). It has been demonstrated that cotton combustion behaviour is affected by fabric *grammage* and the collected results significantly depend on the adopted test method, as graphically reported in Fig. 11. Thus, the present study provides basic and fundamental knowledge for the understanding and proper comparison of cotton combustion data evaluated by different means.

Acknowledgments The authors thank the European COST Action FLARETEX (MP1105) “Sustainable flame retardancy for textiles and related materials based on nanoparticles

substituting conventional chemicals". In addition, the authors want to thank Mr. Andrea Messina and Mr. Alessandro Di Blasio for the PCFC tests and SEM observations, respectively. In addition, we thank Dr. Gabriella Fusi and Centro Tessile e Cotoniero (Busto Arsizio, Italy) for the measurements regarding the fabric weave.

References

- Alongi J, Malucelli G (2015) Cotton flame retardancy: state of the art and future perspectives. *RSC Adv* 5:24239–24263. doi:[10.1039/C5RA01176K](https://doi.org/10.1039/C5RA01176K)
- Alongi J, Camino G, Malucelli G (2013a) Heating rate effect on char yield from cotton, poly(ethylene terephthalate) and blend fabrics. *Carbohydr Polym* 92:1327–1334. doi:[10.1016/j.carbpol.2012.10.029](https://doi.org/10.1016/j.carbpol.2012.10.029)
- Alongi J, Carosio F, Horrocks AR, Malucelli G (2013b) Update on flame retardant textiles: state of the art, environmental issues and innovative solutions. *Smithers RAPRA Publishing, Shawbury*
- Alongi J, Carosio F, Malucelli G (2014a) Current emerging techniques to impart flame retardancy to fabrics. *Polym Degrad Stab* 106:138–149. doi:[10.1016/j.polymdegradstab.2013.07.012](https://doi.org/10.1016/j.polymdegradstab.2013.07.012)
- Alongi J, Milnes J, Malucelli G, Bourbigot S, Kandola B (2014b) Thermal degradation of DNA-treated cotton fabrics under different heating conditions. *J Anal Appl Pyrolysis* 108:212–221. doi:[10.1016/j.jaap.2014.04.014](https://doi.org/10.1016/j.jaap.2014.04.014)
- ASTM D2863 (2006) Standard test method for measuring the minimum oxygen concentration to support candle-like combustion of plastics (oxygen index). *American Society for Testing and Materials International, West Conshohocken (Pennsylvania)*
- ASTM D7309 (2013) Standard test method for determining flammability characteristics of plastics and other solid materials using microscale combustion calorimetry. *American Society for Testing and Materials International, West Conshohocken (Pennsylvania)*
- Basak S (2015) Fire retardant cellulosic textile using banana pseudostem sap. *Pol J Chem Technol* 17:123–133
- Basak S, Samanta KK, Chattopadhyaya SK (2014) Fire retardant property of the cotton fabric treated with herbal extract. *J Text Inst.* doi:[10.1080/00405000.2014.995456](https://doi.org/10.1080/00405000.2014.995456)
- Basak S, Samanta KK, Chattopadhyay SK, Narkar R (2015a) Thermally stable cellulosic paper made using banana pseudostem sap, a wasted by-product. *Cellulose.* doi:[10.1007/s10570-015-0662-7](https://doi.org/10.1007/s10570-015-0662-7)
- Basak S, Samanta KK, Chattopadhyay SK, Narkar R (2015b) Self-extinguishable ligno-cellulosic fabric using banana pseudostem sap. *Curr Sci* 108:372–383
- Bourbigot S, Chlebicki S, Mamleev V (2002) Thermal degradation of cotton under linear heating. *Polym Degrad Stab* 78:57–62. doi:[10.1016/S01413910\(02\)00119-2](https://doi.org/10.1016/S01413910(02)00119-2)
- Ceylan O, Alongi J, Van Landuyt L, Frache A, De Clerck K (2013) Combustion characteristics of cellulosic loose fibres. *Fire Mater* 37:482–490. doi:[10.1002/fam.2147](https://doi.org/10.1002/fam.2147)
- Faroq AA, Price D, Milnes GJ, Horrocks AR (1991) Use of gas chromatographic analysis of volatile products to investigate the mechanisms underlying the influence of flame retardants on the pyrolysis of cellulose in air. *Polym Degrad Stab* 33:155–170. doi:[10.1016/0141-3910\(91\)90015-J](https://doi.org/10.1016/0141-3910(91)90015-J)
- Gordon S, Hsie YL (2007) *Cotton: science and technology*. Woodhead Publishing Limited and CRC Press, Boca Raton (FL)
- Horrocks AR (1983) An introduction to the burning behaviour of cellulosic fibres. *J Soc Dyes Colour* 99:191–197. doi:[10.1111/j.1478-4408.1983.tb03686.x](https://doi.org/10.1111/j.1478-4408.1983.tb03686.x)
- Horrocks RA (1996) Developments in flame retardants for heat and fire resistant textiles—the role of char and intumescence. *Polym Degrad Stab* 54:143–154. doi:[10.1016/S0141-3910\(96\)00038-9](https://doi.org/10.1016/S0141-3910(96)00038-9)
- Horrocks AR (2011) Flame retardant challenges for textiles and fibres: new chemistry versus innovative solutions. *Polym Degrad Stab* 96:377–392. doi:[10.1016/j.polymdegradstab.2010.03.036](https://doi.org/10.1016/j.polymdegradstab.2010.03.036)
- ISO 13943 (2007) *Fire safety—vocabulary*. International Organization for Standardization, Geneva
- ISO 3572 (1976) *Textiles—weaves—definition of general terms and basic weaves*. International Organization for Standardization, Geneva
- ISO 5660 (2002) *Fire test, reaction to fire, rate of heat release (cone calorimeter method)*. International Organization for Standardization, Geneva
- Kandola BK, Horrocks AR (1999) Complex char formation in flame-retarded fiber/intumescent combinations: physical and chemical nature of char1. *Text Res J* 69:374–381. doi:[10.1177/004051759906900512](https://doi.org/10.1177/004051759906900512)
- Kandola BJ, Horrocks RA, Price D, Coleman GV (1996) Flame retardant treatments of cellulose and their influence on the mechanism of cellulose pyrolysis. *J Macromol Sci—Rev Macromol Chem Phys* C36:721–794. doi:[10.1080/15321799608014859](https://doi.org/10.1080/15321799608014859)
- Lowden LA, Hull TR (2013) Flammability behaviour of wood and a review of the methods for its reduction. *Fire Sci Rev* 2:4. doi:[10.1186/2193-0414-2-4](https://doi.org/10.1186/2193-0414-2-4)
- Lyon RE, Walters RN (2004) Pyrolysis combustion flow calorimetry. *J Anal Appl Pyrolysis* 71:27–46. doi:[10.1016/S0165-2370\(03\)00096-2](https://doi.org/10.1016/S0165-2370(03)00096-2)
- Lyon RE, Takemori MT, Safronava N, Stoliarov SI, Walters RN (2009) A molecular basis for polymer flammability. *Polymer* 50:2608–2617. doi:[10.1016/j.polymer.2009.03.047](https://doi.org/10.1016/j.polymer.2009.03.047)
- Malucelli G, Bosco F, Alongi J, Carosio F, Di Blasio A, Molleca C, Cuttica F, Casale A (2014) Biomacromolecules as novel green flame retardant systems for textiles: an overview. *RSC Adv* 4:46024–46039. doi:[10.1039/C4RA06771A](https://doi.org/10.1039/C4RA06771A)
- Mamleev V, Bourbigot S, Le Bras M, Yvon J (2009) The facts and hypotheses relating to the phenomenological model of cellulose pyrolysis: interdependence of the steps. *J Anal Appl Pyrolysis* 84:1–17. doi:[10.1016/j.jaap.2008.10.014](https://doi.org/10.1016/j.jaap.2008.10.014)
- Morterra C, Low MJD (1983) IR studies of carbons-II. *Carbon* 21:283–288. doi:[10.1016/0008-6223\(83\)90092-1](https://doi.org/10.1016/0008-6223(83)90092-1)
- Morterra C, Low MJD (1984) An infrared spectroscopic approach to the characterization of intermediate chars. *Mater Lett* 2:289–293. doi:[10.1016/0167-577X\(84\)90134-4](https://doi.org/10.1016/0167-577X(84)90134-4)
- Morterra C, Low MJD (1985) An infrared spectroscopic study of some carbonaceous materials. *Mater Chem Phys* 12:207–233. doi:[10.1016/0254-0584\(85\)90094-X](https://doi.org/10.1016/0254-0584(85)90094-X)
- Price D, Horrocks RA, Akalin M, Faroq AA (1997) Influence of flame retardants on the mechanism of pyrolysis of cotton

- (cellulose) fabrics in air. *J Anal Appl Pyrolysis* 40–41:511–524. doi:[10.1016/S0165-2370\(97\)00043-0](https://doi.org/10.1016/S0165-2370(97)00043-0)
- Schartel B, Hull TR (2007) Development of fire-retarded materials—interpretation of cone calorimeter data. *Fire Mater* 31:327–354. doi:[10.1002/fam.949](https://doi.org/10.1002/fam.949)
- Sekiguchi Y, Frye JS, Shafizadeh F (1983) Structure and formation of cellulosic chars. *J Appl Polym Sci* 28:3513–3525. doi:[10.1002/app.1983.070281116](https://doi.org/10.1002/app.1983.070281116)
- Shafizadeh F, Fu YL (1973) Pyrolysis of cellulose. *Carbohydr Res* 29:113–122. doi:[10.1016/S0008-6215\(00\)82074-1](https://doi.org/10.1016/S0008-6215(00)82074-1)
- Shafizadeh F, Sekiguchi Y (1983) Development of aromaticity in cellulosic chars. *Carbon* 21:511–516. doi:[10.1016/0008-6223\(83\)90144-6](https://doi.org/10.1016/0008-6223(83)90144-6)
- Shafizadeh F, Sekiguchi Y (1984) Oxidation of chars during smoldering combustion of cellulosic materials. *Combust Flame* 55:171–179. doi:[10.1016/0010-2180\(84\)90025-7](https://doi.org/10.1016/0010-2180(84)90025-7)
- Tata J, Alongi J, Carosio F, Frache A (2011) Optimization of the procedure to burn textile fabrics by cone calorimeter: part I. Combustion behavior of polyester. *Fire Mater* 35:397–409. doi:[10.1002/fam.1061](https://doi.org/10.1002/fam.1061)
- UNI EN 1049 (1996) Woven fabrics—construction—methods of analysis. Ente Nazionale Italiano di Unificazione, Milano (Italy)
- Wakelyn PJ, Bertoniere NR, French AD, Thibodeaux DP, Triplett BA, Rousselle MA, Goynes WR, Edwards JV, Hunter L, McAlister DD, Gamble GR (2006) Cotton fiber chemistry and technology. CRC Press, Boca Raton (FL)
- Weil E, Hirschler M, Patel N, Said M, Shakir S (1992) Oxygen index: correlations to other fire tests. *Fire Mater* 16:159–167. doi:[10.1002/fam.810160402](https://doi.org/10.1002/fam.810160402)
- Yang CQ, Hu Y (2011) Applications of micro-scale combustion calorimetry to the studies of cotton and nylon fabrics treated with organophosphorus flame retardants. *J Anal Appl Pyrolysis* 91:125–133. doi:[10.1016/j.jaap.2011.01.012](https://doi.org/10.1016/j.jaap.2011.01.012)
- Yang CQ, Hu Y (2012) Textile heat release properties measured by microscale combustion calorimetry: experimental repeatability. *Fire Mater* 36:127–137. doi:[10.1002/fam.1093](https://doi.org/10.1002/fam.1093)
- Yang CQ, He Q, Lyon RE, Hu Y (2010) Investigation of the flammability of different textile fabrics using micro-scale combustion calorimetry. *Polym Degrad Stab* 95:108–115. doi:[10.1016/j.polymdegradstab.2009.11.047](https://doi.org/10.1016/j.polymdegradstab.2009.11.047)