European Journal of Engineering and Applied Sciences

ISSN: 2651-3412 (Print) & 2667-8454 (Online)
Journal homepage: http://dergipark.gov.tr/EJEAS
Published by Çorlu Faculty of Engineering, Tekirdağ Namık Kemal University

European J. Eng. App. Sci. 2(2), 17-21, 2019

Research Article

Investigation of Combustion Characteristics of Fire-off Treated Cotton and Co/Pet Fabrics

Raziye Atakan[1,*], Gülay Özcan[1]

[1]Istanbul Technical University, Faculty of Textile Technologies and Design, Department of Textile Engineering, Gumussuyu, İstanbul, Turkey

Received: 03.07.202019 *Accepted: 20.12.2019*

Abstract: In this research, combustion behaviors of cotton and cotton/polyester (Co/PET) blend fabrics, which can be used in apparel and home textiles treated with phosphor-nitrogen synergetic flame-retardant (FR) agent, called Fire-off, were investigated. Different blend ratio and areal density of fabrics were used in the experiments and fabrics were ranked before and after treatment in terms of burning hazard. Untreated 35/65% blend Co/PET fabric was found most hazardous in accordance with fire risk. However, it became the safest after FR treatment with Fire-off. Cone calorimetric data, besides, showed that the treated cotton and Co/PET blend fabrics generated less heat of combustion and obtained better FR properties, which can be also confirmed by increase of LOI. Considering the decreased values of effective heat of combustion (EHC), peak heat release rate (pHRR) and total heat release rate (THR) for all samples, it is concluded that Fire-off is effective for preventing the flame spread.

Keywords: *Co/PET, Combustion behavior, Cone calorimeter, Fire risk, Heat release.*

Fire-off'un Pamuk Ve Pamuk/Polyester Kumaşlarda Yanma Davranışının İncelenmesi

Özet: Bu çalışmada, fosfor-azot sinerjisi içeren bir güç tutuşurluk kimyasalı olan Fire-off ile apre uygulanmış, giyim ve ev tekstilinde kullanılabilecek pamuk ve pamuk/polyester (Co/PET) karışımı kumaşların kapsamlı yanma davranışları incelenmiştir. Deneysel çalışmalarda, farklı karışım oranlarına ve farklı gramaja sahip kumaşlar kullanılmış olup kumaşlar apre öncesi ve sonrası yanma risklerine göre sıralanmıştır. İşlem görmemiş %35/65 Co/PET kumaş yangın riski açısından en tehlikeli kumaş olarak saptanmış, fakat bu kumaş Fire-off apresi sonrası en güvenli hale gelmiştir. Konik kalorimetre verileri, ayrıca, işlem görmüş Co/PET kumaşların daha az yanma ısısı ürettiğini ve LOI değerlerindeki artış ile de doğrulanan daha iyi bir güç tutuşurluk özelliği kazandığını göstermiştir. Bütün kumaş tipleri için apre sonrası düşüş gözlenen efektif yanma ısısı, maksimum ısı yayılım oranı ve toplam ısı yayılım oranı değerleri gözönüne alındığında ise Fire-off'un alev yayılımını önlediği sonucuna varılmaktadır.

Anahtar kelimeler: *Co/PET, Yanma davranışı, Konik kalorimetre, Yangın riski, Isı yayılımı.*

1. Introduction

Natural (cotton) and synthetic (polyester, polyamide) fibers based textiles are ubiquitous in our daily life. They are commonly used in typical indoor applications such as carpets, curtains, upholstered furniture and bedding as well as in apparel. However, they are highly flammable and represent a potential fire hazard [1]. For assessment of the potential burning hazard of fabrics, the source of danger to life and injury should be investigated at each stage of burning process. Unlike single fibre based textile materials, analysis of the combustion process of blended fabrics is difficult. Since flammability behavior of fabric blends is complex and flame retardant (FR) test methods such as horizontal, vertical flammability tests, LOI, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) cannot quantify and classify the fabrics for their potential burning and injury hazard because of providing only partial information about burning behavior of materials [2, 3]. The cone calorimetric technique has been successfully exploited for determining combustion characteristics of fabrics due to providing similar

* Corresponding author.
E-mail address: ratakan@itu.edu.tr (R. Atakan)

combustion environment with the actual combustion environment. The method demonstrates burning behavior of a material when irradiated by a heat flow, which is developed by the flame spread. For instance, the heat release rate (HRR) refers to the "average level of heat" material that releases in fire situation. The bigger the HRR, more violently material burns. Peak heat release rate (pHRR) presents the "maximum heat release rate of material". It reflects the maximum intensity of the burning and thus is regarded as the most important parameter for fire safety assessment. It depends on time to peak (TTP), which is related to ignitability of the specimen [4-6]. The total heat release (THR) represents the "total heat release during the whole combustion process". Time to ignition (TTI) indicates the "time of sustained flame forming" and it is identified visually. The shorter TTI of materials leads to more accessible ignition, to spread to the surrounding elements and higher risk of fire. The fire growth rate (FIGRA) can be calculated dividing pHRR value to the time to peak (TTP) (pHRR/TTP). The lower FIGRA leads to more delay in flashover. Thus, reduced FIGRA provides enough time to people to evacuate in real fire scenario [3, 6, 7]. Therefore, the calorimetric data are useful to analyze the burning behavior in assessing of the material in terms of fire prevention [8-10].

In our previous study [11], flame retardant (FR) agent (PVP (PR)-P-DCDA) was synthesized using polyvinyl alcohol, phosphoric acid, hydrophilic polyester resin (PR), and dicyandiamide (DCDA). Lightweight polyester, cotton, and 50/50% cotton/polyester (Co/PET) fabrics were subjected to FR finish with different concentrations of PVP (PR)-P-DCDA via pad-dry-cure method. Vertical Flammability test and LOI were performed to investigate FR properties of samples according to different concentrations of FR bath. Surface morphology, thermal decomposition, and chemical structure of fabric samples were investigated to characterize samples before and after treatment. According to obtained TGA results, Fire-off treatment lead to reduce in decomposition temperature of fabrics and favor stable char formation. Vertical burning test demonstrated that the treated fabrics were neither ignited nor showed afterglow. 100% polyester fabric exhibited significantly increased LOI values (from 22.5% to 33%) at even low (8–9%) add-ons. PVP (PR)-P-DCDA lead to reduce in char length and mass loss for cotton and Co/PET fabrics. Nevertheless, they achieved LOI of 26 with higher concentrations than polyester. PVP (PR)-P-DCDA has been commercialized by Eksoy Chemical Company under the name Fire-off. In another part of the study [12] the mechanical properties of Fire-off treated fabrics have been also investigated in order to detect any change on strength, elongation, pilling and abrasion values, whiteness index of fabrics. Results showed that Fire-off treatment show a definitive trend of decreasing air permeability, whiteness and increasing elongation, abrasion, pilling properties. To complete its product assessments, combustion behavior of Fire-off in terms of burning hazard could be investigated.

This present work has been undertaken to investigate combustion processes among different areal density and different blend ratio Fire-off treated Co/PET fabrics by cone calorimetry. Experimental fabrics were selected to be used in apparel and home textiles, which are the application fields of Fire-off. Fire risk order of fabrics were determined before and after FR treatment calculating FIGRA (pHRR/Time to pHRR ratio) in accordance with burning hazard potentials.

2. Experimental

2.1. Materials

The experiments were conducted with scoured Co/PET and cotton fabrics, given in Table 1, supplied from Ata Textile in Turkey. They all represent those used in apparel and home textiles.

Table 1. Fabrics used in the study.

Fabric Type	Areal Density	Fabric Structure	Yarn Counts	Thickness
100% Cotton	227 g/m^2	2/1 S twill	Nm 36/1 x 36/1	0.38 mm
50/50% Co/PET	292 g/m^2	3/1 S twill	Nm 24/1 x 20/1	0.56 mm
35/65% Co/PET	300 g/m^2	3/1 S twill	Nm 20/1 x 20/1	0.59 mm
50/50% Co/PET	227 g/m^2	3/1 S twill	Nm 36/1 x 30/1	0.46 mm

*Cotton: T_c: 400ºC, ΔH_m: 19 kj/g, LOI: 18.4 %
*PET: T_g: 80-90ºC, T_m: 255ºC, T_c: 480ºC, ΔH_m: 24 kj/g, LOI: 20-21.5 %

As chemical, P-N synergetic FR agent (PVP (PR)-P-DCDA) called Fire-off, which was commercialized by Eksoy Chemical Company [11], was used for FR treatments of fabrics.

2.2. Fabric Finishing Procedures

FR bath with 350 g/L Fire-off was prepared in water. The fabric samples were treated via 2 dip-2 nip impregnation with a laboratory padder in at room temperature with 95±2% wet pick-up. After padding, they were dried at 100ºC for 3 min, and subsequently cured in an oven at 180ºC for 3 min. The add-on (%) of FR was calculated using equation 1.

$$\text{Add-on (\%)} = \frac{\text{Last weight of specimen- Initial weight of specimen}}{\text{Initial weight of specimen}} \text{x100} \quad (1)$$

2.3. Evaluation of Flame Retardancy

2.3.1. LOI Test

LOI test identifies "the minimum volume (%) concentration of oxygen in a mixture gas of O_2 and N_2 that will sustain combustion of a material". Textile materials demonstrating LOI values up to 21 have a rapidly burning behavior. On the other hand, they burn slowly in LOI values between 21 and 25. LOI values of more than 26 points out that materials have flame retardant features [13, 14]. Limiting Oxygen Index (LOI) test was performed for cotton and Co/PET blends according to BS EN ISO 4589-2[15] standard using Concept Equipment Oxygen Index Module (Figure 2a). Before starting to test, specimens were conditioned for 24 h under the temperature of 20±2°C and relative humidity of 65±5%). The gas pressure of O_2 and N_2 was set to be 2.5 bar. The gas flow was controlled and calibration has been performed automatically.

2.3.2. Cone Calorimetry

Combustion behaviors of untreated and Fire-off treated fabric specimens with 100 mm x 100 mm dimensions were investigated using cone calorimetry (Fire Testing Technology FTT, UK), and all the tests were carried out according to BS ISO 5660-1 [16]

under the heat flux of 35 kW/m^2. The cone calorimeter is intended for testing actual commercial products. Thus the specimen thickness should be, as much as possible, the thickness of the finished product [17]. The instrument is restricted to testing specimens not thicker than 50 mm. On the other hand, the strong influence of low sample thickness should be taken into account when thermally thin samples are investigated. The samples used here (thickness: less than 1 mm) are classed as thermally thin samples according to the ISO 5660 standard for cone calorimetry testing. Before the test, specimens were conditioned in accordance with related standard. In the test, some fabrics may curl or melt during heat flux exposure. Thus, changing specimen configuration is a challenge to record accurate and reproducible calorimetric data [3]. In order to stabilize fabrics, a cross wire grid was used for testing fabric specimens as depicted in Figure 1. The fabrics were placed and maintained in horizontal configuration in a sample holder. Parameters such as Time to Ignition, (TTI, s) Time To Flame out (FO, s), heat release data such as Peak Heat Release (pHRR, kW/m²) and Total Heat Release (THR, MJ/m²), Effective Heat Combustion (EHC, MJ/kg) were measured. The complete test consisted of three runs for each fabric type to guarantee reproducibility of results.

Figure 1. Sample preparation and retaining grid geometry.

3. Results and Discussion

3.1. Evaluation of Flame Retardancy

3.1.1. LOI Results

Table 2 shows LOI tests results along with associated with FR add-ons of cotton and Co/PET fabric samples.

Table 2. LOI values of untreated and treated fabrics.

Fabric Type	LOI of untreated fabrics	Add-on (%)	LOI of treated fabrics
100% Cotton (227 g/m^2)	18.5	22.41	27.3
50/50% Co/PET (292 g/m^2)	18.8	24.51	26.7
35/65% Co/PET (300 g/m^2)	18.6	24.21	26.6
50/50% Co/PET (227 g/m^2)	18.7	25.54	27.1

It is evident in Table 2 that treated samples with ~22 to 25 % add-ons showed LOI values higher than 26, which indicates their flame retardant properties. As it is also obvious that Fire-off treatment induce a remarkable increase in LOI values of cotton (18.5 to 27.3), and Co/PETs (18.6 to 26.6) as compared to the scoured fabrics.

3.1.2. Cone Calorimetry Results

Regarding to heat release data, heat release rate (HRR) curves of each fabric are depicted in Figure 2 and the detailed data obtained from the cone calorimeter are listed in Table 3.

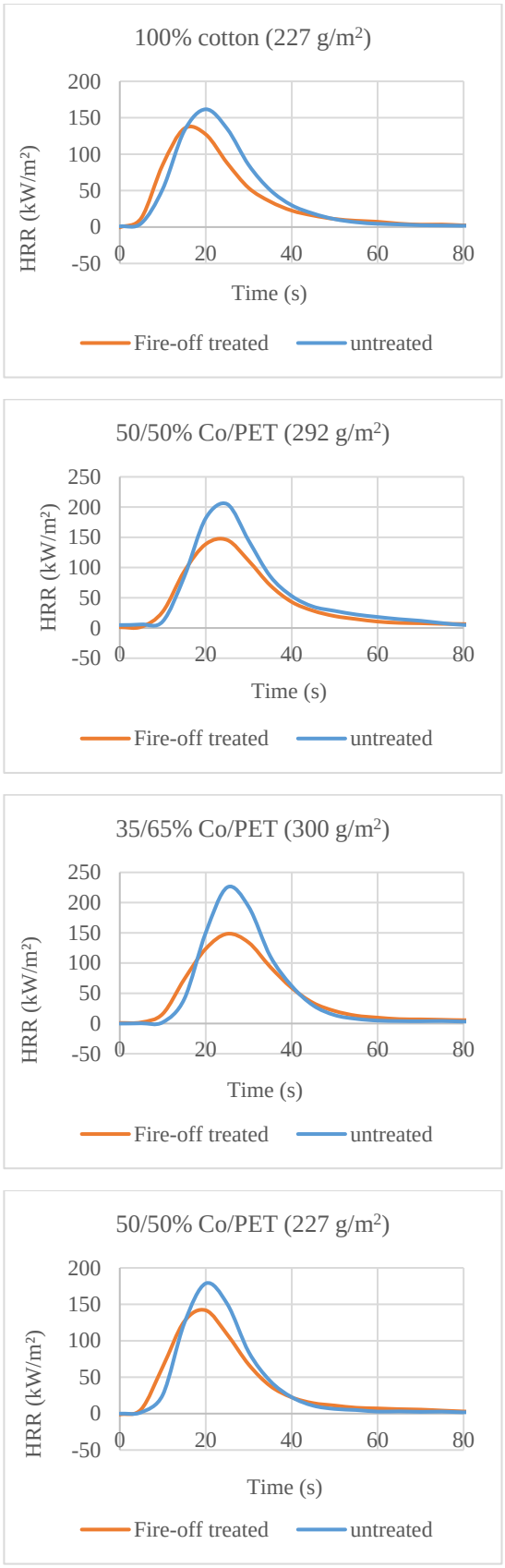

Figure 2. HRR curves of untreated and treated fabrics with Fire-off.

Based on the ignition and flame out times of samples (TTI and FO), it is clearly seen from the Figure 2 and Table 3, ignition times of treated fabrics are slightly earlier or similar compared to untreated fabrics under the heat flux of 35 kW/m^2 in cone calorimetry. On the other hand, Fire-off treatments clearly

reduced the flame out time of cotton fabrics from 54 s to 30s. This result is in line with the LOI values of samples, increase in LOI values of samples before and after treatment is the maximum for cotton (from 18.5 to 27.3, 47% increase). Therefore, it can be said that the presence of Fire-off in the fabrics do not delay ignition times of cotton and Co/PET fabrics. On contrary, treated fabrics extinguish earlier or similar compared to untreated ones. These results are also in line with the TGA data obtained in our previous study, which shows decreases in the decomposition temperatures of cotton, polyester and Co/PET blends [11].

Table 3. Combustion data by cone calorimeter.

Untreated Fabrics:

Fabric Type	**TTI (s)**	**FO (s)**	**THR (180)**	**EHC (120)**	**pHRR**	**Time to pHRR**	**FIGRA**
100% Cotton (227 g/m^2)	10	54	3.65	15.89	158.20	20	7.9
50/50% Co/PET (292 g/m^2)	13	52	4.40	16.08	205.01	23	8.9
35/65% Co/PET (300 g/m^2)	11	34	4.81	15.37	233.16	23	10.1
50/50% Co/PET (227 g/m^2)	10	31	3.42	12.28	178.31	20	8.9
CV % within the replicates:	*0-0.1*	*0-0.3*	*0-0.1*	*0-0.5*	*0-0.03*	*0-0.1*	-

*EHC (120): Average Effective heat combustion over 120 seconds.
**THR (180): Total heat release over 180 seconds.

Fire-off Treated Fabrics:

Fabric Type	**TTI (s)**	**FO (s)**	**THR (180)**	**EHC (120)**	**pHRR**	**Time to pHRR**	**FIGRA**
100% Cotton (227 g/m^2)	10	30	3.18	12.65	134.65	17	7.9
50/50% Co/PET (292 g/m^2)	10	40	4.18	11.35	157.43	25	6.3
35/65% Co/PET (300 g/m^2)	10	42	3.96	11.52	148.76	25	6.0
50/50% Co/PET (227 g/m^2)	8	31	3.15	11.24	139.65	20	7.0
CV % within the replicates:	*0-0.14*	*0.01-0.1*	*0.03-0.09*	*0-0.1*	*0-0.07*	*0-0.14*	-

*EHC (120): Average Effective heat combustion over120 seconds.
**THR (180): Total heat release over 180 seconds.

As Table 3 shows, Fire-off treatment modifies the flammability behavior of cotton and Co/PET fabrics by decreasing the THR (3.65 MJ/m^2 to 3.18 MJ/m^2 for cotton, 3.42 MJ/m^2 to 3.15 MJ/m^2 for the same areal density Co/PET). Since THR decreases, pHRR decreases as well (namely, 158 kW/m² to 135 kW/m² for cotton: 14% and 178 kW/m² to 140 kW/m² for same areal density Co/PET: 21%). It is also obviously seen from the HRR curves in Figure 2, Fire-off treatment shows lower rate of pHRR compared to untreated fabrics in the study. The reduction in pHRR and FIGRA for treated fabrics was highest for 35/65% Co/PET (233 to 149 kW/m^2 and 10.1 to 6 kW/m².s). This might be due to high polyester content in the blend.

For treated samples, with regard to effect of areal density of Co/PET blends in terms of pHHR and THR (Sample 2 and 4), heavier Co/PET (Sample 2-292 g/m^2) showed higher pHRR and THR value (157 kW/m² and 4.17 MJ/m^2) than those (140 kW/m² and 3.15 MJ/m^2) of lightweight Co/PET (Sample 4-227 g/m^2). This shows that weight of the samples also effects pHHR and THR; the heavier Co/PET fabrics release more heat during combustion, as expected. However, it is safer with lower FIGRA value (6.28 kW/m².s versus 6.95 kW/m².s). This finding demonstrates that fabric weight has a positive effect on burning hazard of treated Co/PET fabrics. Comparing to equal areal density cotton and Co/PET fabrics (Sample 1 and 4), Co/PET sample ignite earlier and showed higher pHRR as expected. However, FO and THR of samples were closely similar, which indicates similar combustion behavior. These results are also in line with LOI results (cotton: 27.3 and Co/PET: 27.1).

Since the cone calorimeter was developed in order to approximate an ideal performance-based bench scale fire testing method, some of the results even allow an accurate description of the materials' properties, such as the effective heat of combustion (EHC)[18]. It is also an important parameter that can reflect the combustibility of fabrics. It is seen in Table 3, fourth column that EHC values of the treated fabrics decreased compared to untreated fabrics (12.65 MJ/kg vs.15.89 MJ/kg for cotton, 11.35 MJ/kg vs. 16.08 MJ/kg for 50/50 Co/PET, 11.52 MJ/kg vs. 15.37 MJ/kg for 35/65 Co/PET), which means the reduction of heat released from volatile portion.

In our previous study, it is indicated that Fire-off treatment on the cotton, polyester, and CO/PET fabrics reduced the flammability via dehydration into char, thus the mechanism is mainly in solid phase. [11]. For this study, considering the decreased values of effective heat of combustion (EHC) for all samples, it is reasonable to conclude that that Fire-off is effective for preventing the flame spread.

4. Conclusion

In the present work, cotton and Co/PET fabrics to be used in apparel and indoor applications were subjected to FR treatment with organophosphorus based FR system (Fire-off) via impregnation method. Combustion behaviors of cotton, 50/50%, and 35/65% Co/PET blended fabrics were investigated by cone calorimetry under the heat flux of 35 kW/m^2 to evaluate their fire risk. Burning hazard potentials of fabrics were compared before and after the treatment. The cone calorimeter results showed that the HRR, THR and EHC values of the treated fabric decreased. In conclusion, Fire-off treated fabrics obtained better flame retardancy with generating less combustion heat, which can be also confirmed by the decrease of FIGRA and increase of LOI value (~27%). Untreated 35/65% blend Co/PET fabric was found most hazardous in terms of fire risk. However, it became the safest after FR treatment with Fire-off. These findings point out that Fire-off treatment is effective on both solid phase and exothermic reaction of the combustion.

Acknowledgments

The authors gratefully acknowledge the funding by The Scientific and Technological Research Council of Turkey, under grants of 2214 International Doctoral Research Fellowship Programme. The authors would like to thank Ata Textile for fabric supply and Prof. Dr. B. Kandola and Chen Zhou for the technical support in performing combustion tests in Bolton

University, Institute for Materials Research and Innovation Laboratories.

References

[1]. Horrocks, A.R. and D. Price, *Fire retardant materials*. 2001: Woodhead Publishing.

[2]. Fukatsu, K., *Thermal degradation behaviour of aromatic polyamide fiber blended with cotton fiber.* Polymer degradation and stability, 2002. **75**(3): p. 479-484.

[3]. Nazare, S., B. Kandola, and A.R. Horrocks, *Use of cone calorimetry to quantify the burning hazard of apparel fabrics.* Fire and Materials, 2002. **26**(4-5): p. 191-199.

[4]. Ames, S. and S. Rogers. *Large and small scale fire calorimetry assessment of upholstered furniture*. in *Proceedings of Interflam*. 1990. Interscience Communications Ltd London.

[5]. Schartel, B., et al., *Fire retardancy of polypropylene/flax blends.* Polymer, 2003. **44**(20): p. 6241-6250.

[6]. Yang, J.-C., et al., *Flame retardation of cellulose-rich fabrics via a simplified layer-by-layer assembly*. Carbohydrate polymers, 2016. **151**: p. 434-440.

[7]. Li, Z.-F., et al., *Fire retardant and thermal degradation properties of cotton fabrics based on APTES and sodium phytate through layer-by-layer assembly.* Journal of Analytical and Applied Pyrolysis, 2017. **123**: p. 216-223.

[8]. Huggett, C., *Estimation of rate of heat release by means of oxygen consumption measurements.* Fire and Materials, 1980. **4**(2): p. 61-65.

[9]. Parker, W.J., *Calculations of the heat release rate by oxygen consumption for various applications.* Journal of Fire Sciences, 1984. **2**(5): p. 380-395.

[10]. Ji, J., L. Yang, and W. Fan, *Experimental study on effects of burning behaviours of materials caused by external heat radiation.* Journal of Combustion Science and Technology, 2003. **9**(2): p. 139-143.

[11]. Atakan, R., et al., *Development of a flame retardant chemical for finishing of cotton, polyester, and CO/PET blends.* Journal of Industrial Textiles, 2018: p. 1528083718772303.

[12]. Raziye Atakan, G.O. *Mechanical Assessments of Fire-off on CO/PET Fabrics*. IOP Conf. Ser.: Mater. Sci. Eng. 460 012052, 2018.

[13]. Horrocks, A.R., M. Tune, and L. Cegielka, *The burning behaviour of textiles and its assessment by oxygen-index methods.* Textile Progress, 1988. **18**(1-3): p. 1-186.

[14]. Salmeia, K.A., S. Gaan, and G. Malucelli, *Recent advances for flame retardancy of textiles based on phosphorus chemistry.* Polymers, 2016. **8**(9): p. 319.

[15]. 4589, B.E.I., *Plastics-Determination of burning behaviour by oxygen index*, in *Part 2: Ambient-temperature test*. 1999: Brussels.

[16]. BS ISO, E., *Reaction-to-fire tests — Heat release, smoke production and mass loss rate* in *Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)*. 2015: Switzerland.

[17]. Babrauskas, V., *The cone calorimeter*, in *SFPE handbook of fire protection engineering*. 2016, Springer. p. 952-980.

[18]. Schartel, B., M. Bartholmai, and U. Knoll, *Some comments on the use of cone calorimeter data.* Polymer degradation and stability, 2005. **88**(3): p. 540-547.