

TG/DTG/DTA evaluation of flame retarded cotton fabrics and comparison to cone calorimeter data

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ABSTRACT

Unbleached cotton fabrics (UCF) with 12.5% polypropylene scrim treated with two phosphate–urea based fire-retardant (FR) formulations were evaluated for FR properties using thermogravimetry/differential thermogravimetry/differential thermal analysis (TG/DTG/DTA) method. In addition to testing the two FR-treated unbleached cotton fabrics (CF-FR1 and CF-FR2), bleached cotton fabric (BCF) treated with the two FR formulations (BCF-FR1 and BCF-FR2) was evaluated. Both formulations were washable with add-on of FR chemicals at 18.7% (FR1) or 17.4% (FR2) for UCF and 22.5% (FR1) or 24.9% (FR2) for BCF. The decreasing order of sums at maximal rates of samples degradation in air environment according to DTG method was: BCF (21.40%/min) > UCF (12.91%/min) > BCF-FR2 (12.83%/min) > BCF-FR1 (11.68%/min) > CF-FR2 (10.20%/min) > CF-FR1 (9.73%/min). It indicates that both formulations cause the decrease of thermooxidation of the products at slower rates than the starting material. Several endo- and exothermic peaks observed by DTA in inert and oxidative environment gives additional information about the degradation process. The order of decreasing thermal responses of the studied samples based on sums of DTA peak values of endothermic and exothermic peaks in air environment is: UCF (0.597 °C/mg) > BCF (0.120 °C/mg) > CF-FR1 (0.089 °C/mg) > BCF-FR1 (0.077 °C/mg) > CF-FR2 (0.062 °C/mg) > BCF-FR2 (0.053 °C/mg). This is in agreement with the cone calorimeter results according to which the flammability properties are improving with the decreasing heat release rates or ignition time prolongation in order: UCF > CF-FR1 > CF-FR2. The advantage of TG/DTG/DTA method is slower linear heating rate, which allows the more detailed evaluation of the light and flammable cotton fabric.

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1. Introduction

At the present time, it is more and more evident that flammable materials like cotton fabrics need to be tested by several independent methods to properly evaluate effectiveness of the flame retardancy formulations (Alongi, Ciobanu, & Malucelli, 2012; Gao, Wu, & Wu, 2009; Hagen, Hereid, Delichatsios, Zhang, & Bakitzis, 2009; Mostashari & Baie, 2010). Cone calorimeter testing is less sensitive when flammable and low density materials are tested in small quantities and high heat fluxes. In such cases, the heat release rate values are not giving the complete picture. Thermogravimetry is a predominant and important analytical method to evaluate the thermal processes of cotton. Also the use of limited oxygen index (LOI) method brings a difference picture due to atmosphere manipulation, but could be misleading at high FR add-on. It is also informative to know the temperature of the studied sample during the course of degradation, which is dependent upon the architecture of the burn-

ing chamber. This can be better achieved on thermobalance than with cone calorimeter. The effectiveness of flame retardant could then be evaluated on the basis of volatiles formed with increasing temperature, which is related to amount of residue produced. Also the effect of thermooxidation during the process could be observed with TG/DTG/DTA when comparing the degradation process in inert and oxidative environment (Šimkovic, Antal, Balog, Košík, & Plaček, 1985). A more effective flame retarded material exhibits smaller differences during the course of degradation for inert in comparison to oxidative conditions (Chang et al., 2011; Tian et al., 1999). The problem of residue behavior is especially important when large amounts of flame retardants are added (Chang et al., 2007; Parikh et al., 2003). When evaluating the residue behavior of fabric samples with initial mass of 2 g, the cone calorimeter might provide unreliable data due to the low sensitivity of a balance with a 0–500 g range (White, Nam, & Parikh, 2012). For such applications TG/DTG/DTA should be used for comparison.

The future of flame retardants is in a more environmental approach, and especially for cotton, less plastic containing and more polysaccharide containing composites need to be studied (Horrocks, 2011; Šimkovic, 2008). Examples of the use of

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the cone calorimeter for light composite sample evaluation are known (Šimkovic, Martvoňová, Maníková, & Grexa, 2005; Šimkovic, Martvoňová, Maníková, & Grexa, 2007; Šimkovic, White, & Fuller, 2012; Šimkovic, 2012). It indicates that at lower heating levels the shape of heat release rate (HRR) curve is broadening due to slower increasing of temperature in the heating chamber. Under these circumstances the heating conditions of cone calorimeter and TG/DTG/DTA are closer and might be complementary for the study of flame retarding conditions. In this way it could be learned if the samples could be tested properly with cone calorimeter due to several times smaller thicknesses used than for UCF sample. This series of TG/DTG/DTA tests is a follow-up to an evaluation of the FR fabrics in a cone calorimeter (White, Nam, & Parikh, 2012).

The goal of the present work was to use a TG/DTG/DTA system to understand the behavior and to evaluate the effectiveness of two phosphate–urea flame retardants in improving the fire performance of UCF and compare the results with cone calorimeter tests on the same materials. Additionally BCF samples modified with the same formulations were evaluated with TG/DTG/DTA. These formulations were developed for mattresses applications and tested on different settings (Nam, Parikh, & Condon, 2010; Parikh et al., 2003; Uppal, Mercemik, Sunghayun, Parikh, & Condon, 2010). Similar concepts were studied by other authors with the help of thermogravimetry (Liodakis, Fetsis, & Agiovlastis, 2009). This method was already used before (Šimkovic, Antal, Balog, Košík & Plaček, 1985) when DTG and DTA are used for evaluation of cellulose thermooxidation. Problems related to textile or decreasing the sample size when tested by cone calorimeter are also known (Limdholm, Brink, & Hupa, 2012; Schartel & Hull, 2007).

2. Experimental

2.1. Materials

Gray cotton blend NW fabric: Gray cotton needle punched nonwoven (NW) fabric (UCF; 87.5% cotton with 12.5% polymer scrim of polypropylene; N, 0.14; C, 46.69; H, 7.23; S, 0; P, 0.33), 1.3–2.5 mm thick, 150 g/m² was donated by the Warm Company, Lynnwood, WA.

Bleached cotton NW fabric: Bleached cotton spunlaced nonwoven fabric (BCF; 220–240 g/m; N, 0; C, 43.60; H, 6.67; S, 0; P, 0.19), 0.25 mm thick was developed at SRRC from bleached cotton fiber and bleached cotton comber oil fiber at 1:1 (w/w) ratio.

2.2. FR formulations

The non-durable FR formulations were the phosphate-nitrogen based formulations SRRC-1 and SRRC-2 as described previously (White, Nam, & Parikh, 2012). FR formulations were applied at the Southern Regional Research Center (SRRC) to the fabrics with two immersions and two nips on a Mathis padder. The thoroughly wetted and saturated fabrics were passed through the first padding at a nip pressure of 90 PSI at a speed of 2 m/min to obtain a low wet pick-up of about 60%. Fabrics were again saturated in the formulation, and passed through a nip pressure of 10 PSI at 2 m/min to obtain a wet pick-up of about 95–100%. The samples were placed on a pin frame and dried thoroughly in the Mathis forced-air dryer at 135 °C for 2 min 45 s. The technique of double immersion and double padding was used to obtain good penetration of FR chemicals into the fiber and uniform saturation of FR chemicals in gray fabrics. The add-on of FR chemicals was 18.7% for SRRC-1 (CF-FR1) and 17.4% for SRRC-2 (CF-FR2). The elemental composition of CF-FR1 specimen was: N, 3.08; C, 42.99; H, 7.13; S, 0; P, 2.51, while for CF-FR2 it was: N, 3.84; C, 43.18; H, 7.13; S, 0; P, 3.60. On BCF sample treated with SRRC-1 (BCF-FR1) it was: N, 3.27; C, 38.73; H, 6.18; S,



Fig. 1. UCF sample in sample holder before testing on cone calorimeter.

0; P, 2.31, while for BCF modified with SRRC-2 (BCF-FR2) it was: N, 3.89; C, 38.29; H, 6.54; S, 0; P, 2.31. The add-on of FR chemicals for sample treated with SRRC-1 formulation (BCF-FR1) was 22.5%, while for BCF-FR2 sample it was 24.9%.

2.3. Analytical methods

The elemental composition of specimens was performed on Macro Analyzer (Elementar Analysensystem GmbH, Donaustr. 7, 63452 Hanau, Germany) at detection ranges for C, 0–150 mg (or 100%); H, 0–15 mg (or 100%); N, 0–100 mg (or 100%); S, 0–18 mg (or 100%). Phosphorus was determined colorimetrically by calibration on phosphate.

Thermogravimetric measurements were run on SDT 2960 (TGA-DTA module; TA Instruments) thermobalance at 90 cm³/min nitrogen or air flow and 10 K/min heating rate from 20 to 1000 °C using 6–7 mg sample amounts in platinum pan.

The conditions used for cone calorimeter testing demonstrated in Figs. 1–4 were discussed previously in the earlier publication (White, Nam, & Parikh, 2012).

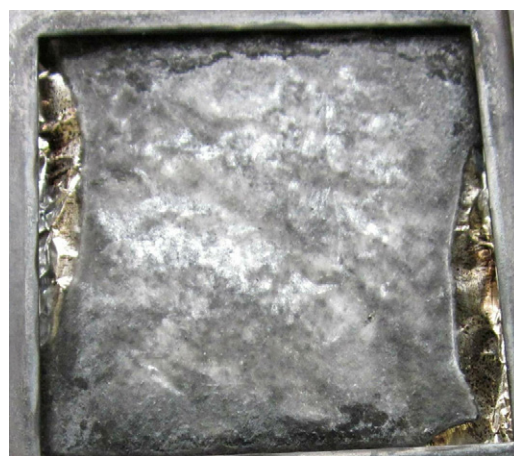


Fig. 2. UCF sample after cone calorimeter testing at 10 kW/m² flux.

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