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# Intumescent Flame retardant coating for Cotton fabric and Evaluation of their kinetic parameters

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**Abstract:**

Organic cellulosic materials are highly flammable and therefore these materials need to be modified to prevent from fire. This is usually carried out by means of incorporation of flame retardant material into the polymer/ cellulosic based materials. However, an alternative and a new way to provide flame-retardant properties to such material may be carried out through application of flame-retardant coatings. Furthermore, this coating provides flame resistance without affecting the bulk properties or processing of the polymer substrate and can be applied easily to any substrates. In this study, attempts were made to develop a new approach based on an innovative coating. The coating composition includes compound based on phosphorus and nitrogen and char forming agents which trigger several fireproofing mode of actions in condensed and gas phases. This article investigates the preventive modification of cotton fabric to exhibit flame retardancy by application of intumescent coating through Pad-Dry-Cure technique. This study also explores the flammability and kinetic parameters of cellulosic material and coated fabric samples. Activation energy of thermal degradation of samples was determined using Coats-Redfern method. Result of the study has shown that samples coated with intumescent coating formulation were best as far as physical properties are concerned. The Limiting Oxygen Index (LOI) values of sample found increased from 18 to 26.4 % on coating. Activation energy of coated sample is found to be lower than uncoated fabric.

**Key words**: Cellulosic fibre, intumescent, coating, flame retardant, activation energy, LOI.

## Introduction:

Cellulosic polymeric material like fabric, papers, wood etc. undergoes degradation on ignition, forming highly burnable volatile products mainly leavoglucosan with spread of fire causing injuries and losses in fire accidents [1]. Cellulose thermally decomposes below 300 °C under dehydration, depolymerisation and oxidation with release of CO, $CO_2$ and

carbonaceous residue [2, 3]. At higher temperature (> 300°C), tar consisting leavoglucosan as a major flammable constituent is formed [4, 5]. Function of flame retardant is to increase char at the cost of flammable volatiles. Many flame retardants available in the market such as chlorine-type flame retardant, bromine-type flame retardant, phosphorus-halogen type flame retardants and also inorganic flame retardant to make cellulosic polymer as flame proof [6]. But halogen based flame retardant releases highly toxic and corrosive fumes during combustion. There has also been major interest in replacing halogenated flame retardants because of environmental and toxicity issues [7, 8]. In this study, an attempt has been made to develop a new approach based on intumescent coating. Intumescent flame retardant system requires acid source, a swelling agent and a char forming agent [9-13]. The coating composition includes compound based on phosphorus and nitrogen and char forming agents which activate many fireproofing mode of actions in condensed and gas phases.

**Experimental:**

**Materials**

For intumescent flame retardant system, ammonium polyphosphate (APP) as an acid source, melamine as a swelling agent and Pentaerythritol (PER) as a carbon source were obtained from Clariant Co., Germany. Cotton Fabric (area density, 220.5 g/m$^2$ ) was used for back coating (purchased from market) and acrylic resin, Zytrol-7800 as binder (Zydex Industries,Gujrat, India).

**Preparation of intumescent formulation**

Intumescent formulation was prepared containing intumescent components (APP, pentaerythritol and melamine) in ratio 3:1:1. The acrylic resin was used for coating the intumescent formulation on simple Cotton Fabric. The intumescent formulation was prepared by mixing evenly in pastel and mortar.

**Intumescent formulation application and curing process**

The intumescent formulations solution was prepared with desired proportions of M:L (material to liquor) ratio that incorporated into sample by using pad- dry- cure technique. The solution was placed in the trough of padding mangle for giving treatment. After dipping the samples into the solution for approximately 2 minutes, the sample was passed through the rolls at 2.5 kPa pressure.

Two dip two nip were given to get desired chemical level of weight add on. The cotton fabric sample was taken for this study was basis weight of 220.5 g/m$^2$. Initial weight of sample taken for this proposes was 8.32 gram. The coating amount was adjusted using bars of

different numbers and coat weight was maintained to 302.3 g/m$^2$.Then the coated sample was dried in oven and cured at 105- 115 $^0$C for 2 minutes.

**Thermal Analysis**

Thermal degradation of samples was carried out by thermogravimetry (TG) (TA instruments SDT Q600). Samples in platinum crucibles were analyzed from ambient temperature to 600 °C (heating rate, 15 °C/min). Nitrogen was used as carrier gas (flow rate, 100 ml/min).

**Limiting Oxygen Index (LOI)**

LOI values that measure performance of flame retardancy were measured using a Stanford Redcroft FTA flammability unit BS-2782 instrument. Samples were tested according to standard method ASTM D2863, ISO-4589.

**Kinetics Study**

TG data were analyzed for kinetic study using Coats-Redfern method [14, 15]. It is assumed that only a single reaction occurs while a sample undergoes a certain temperature rise at a steady heating rate, β. Thus, Coats-Redfern equation when n≠1 is given below:

$$ln[\frac{1-(1-\alpha)^{1-n}}{(1-n)T^2}] = ln\frac{AR}{\beta E} + ln[1 - \frac{2RT}{E}] - \frac{E}{RT}$$

…….Equation (i)

Where n=order of reaction, E is Activation Energy, A is Pre-exponential Factor, R is gas constant,

β is heating rate i.e, 15 $^0$C/Min,

α is degree of conversion= $(W_0-W_T)/(W_0-W_f)$ where $W_0$ is initial weight of sample, $W_T$ is residual weight of sample at temperature, T $^0$C, $W_f$ is the final weight of sample.

when order of reaction, n=1 then Coats-Redfern equation become as below

$$ln[\frac{-\ln(1-\alpha)}{T^2}] = ln\frac{AR}{\beta E} + ln[1 - \frac{2RT}{E}] - \frac{E}{RT}$$ …….Equation (ii)

**Result and Discussion:**

**Thermal analysis**

The untreated sample shows one stage of thermal degradation in range of 285-390 $^0$C with weight loss of 75.2% and with DTG peak at 361 °C as given in Table 1 and shown in Fig 1-2. The sample degraded almost completely up to 390 °C leaving no char yield at 600 °C. The thermal degradation of sample was due to pyrolysis decomposition reactions.

**First stage of thermal degradation:**

First stage of thermal degradation of intumescent coated paper in temperature range 231-272 °C shows 31.5 % (Fig 1) weight loss. The first stage thermal degradation of sample is mainly due to pyrolysis reactions corresponds to release of phosphoric acid from intumescent. This released phosphoric acid starts phosphorylate the cellulose as well as the pentaerythritol at about 200 °C. In the meantime the melamine (the third component of intumescent formulation as swelling agent) sublimes at about 245 °C and start releasing $NH_3$ at about 255 °C and continues up to 385 °C and weight is less in inert atmosphere. This way, a cover of swelled material starts building on the polymer substrate as a thermal barrier. The dispersion of combustible volatiles from the burning substrate to combustion phase is also prevented. The DTG peak at 241 °C (Fig 2) for coated sample also predicts the changes in decomposition stage of cotton fabric sample.

**Second stage of thermal degradation:**

Second stage of thermal degradation of intumescent coated sample in temperature range 265-600 °C shows 31 % weight loss as in Fig 2. This may be due to pyrolytic decomposition, deoxygenation, dehydrogenation, and aromatization of char. At the same time complex formation takes place of intumescent component with the substrate and solidification through cross-linking reaction of residual char occurs in this stage. No DTG peak is shown in this stage because of gradual weight loss.

**Kinetics Study of Thermal Degradation**

The kinetic parameters of thermal degradation of untreated and its coated samples were determined using first order Coats-Redfern method as in equation (ii) on data obtained from TG and is given in Table 1 using Coats Redfern Plot (Fig 3). Activation energies of coated sample and untreated sample was calculated in range of degree of conversion (1-α=0.9-0.49) and (1-α=0.9-0.1) respectively, which falls in first stage of thermal degradation of maximum mass loss. Activation energy of coated sample (137.9 kJ $mol^{-1}$) is found lower than that of untreated paper (163.5 kJ $mol^{-1}$). Decrease in activation energy is due to catalyzing effect of phosphoric acid and metals, which show that dehydration path during thermal degradation of cotton is chosen resulting in more char formation at the expense of tar.

**LOI and Char Yield**

Char yield at 600°C and LOI values of untreated paper and its coated sample were obtained. Higher the value of LOI and char yield, better is the flame resistance of the material. LOI

value for pure untreated sample (18%) was found to increase for intumescent coated sample (26.4%). No char yield was obtained for pure untreated paper sample at 600°C. Char yield for intumescent coated sample increased from zero to 26.3%.

**Conclusions**

TG curves of coated fabric shows two stages of thermal degradation which are mainly due to dehydration, pyrolytic decomposition and aromatization of char, respectively. For coated sample, pyrolytic degradation gives a less amount of tar consisting flammable volatile products and correspondingly higher char yield. Decrease in activation energy is due to catalyzing effect of released phosphoric acid, and support that dehydration path during thermal degradation of polymeric substrate is preferred in which more char is formed at the expense of tar. LOI value for pure untreated fabric (18%) increased for coated fabric (26.4%) again supports the flame retardant behaviour of coated sample.

Hence the thermogravimetric analysis, LOI and char yield results has demonstrated that coatings decompose, absorb heat, swell and form the protective char layers at the different temperature ranges, and, therefore, these cooperated reactions provide a good fire protection for the polymeric substrate in a fire.

**References:**


1. Ben Evarts , Lightening Fires and lightening strikes, NFIRS report to NFPA, December 2010
2. M.N Parbhakar, A.U.R.Shah, J-II Song, Composites Research., 28(2) (2019) 29.
3. W. E. Franklin, *J. Fire Retardant Chem*., **9** (1982) 263.
4. C. J. Biermann and R. Narayan, *J. Polym. Sci: Part C, Polym. Letters*, **25** (1987) 89-92.
5. P. Zhu, S. Sui, B. Wang, K. Sun and G. Sun, *J. Anal. Appl. Pyrol*., **71** (2004) 645-655.
6. R. Y. Yeh, Y. Yang and Y. S. Perng, *Taiwan J For Sci.*, **26(1)** (2011) 87-97.
7. S. M. lomakin, G. E. Zaikov and M. I. Artsis, *Int J Polm Mater.*, **32** (1996) 173-202.
8. G. Camino, in *Fire Retardant Polymeric Materials*, edited by G Nelson (ACS, USA) 1995, 461-492 (chap 10).
9. H. T. Deo, N. K. Patel and B. K. Patel, *J Engg Fibres & Fabrics*, **3** (4) (2008), 23-38.
10. D. Wesolek, R. Gasiorwski, S. Rajeswski, J. Walentowska and R. Wojeik, *Polymer*, 8(12) (2016), 419.

11. M. C. Yew, N. H. Ramli Sulong, M. K. Yew, M. A. Amalina and M. R. Johan, Materials Res. Innovations, **18**(2014) SUPPL 6, 384-388.
12. S. Rabe, Y. Chuenban and B. S. Chartel, *Materials*, 10 (5) (2017), 455.
13. M.S. Islam, Theo, G.M. V. Ven, *Bioresources*, 16(2) (2021), 4354.
14. A. W. Coats and J. P. Redfern, *Nature*, London, **201** (1964) 68.
15. J. B. Dahiya, K. Kumar, M. M. Hogedorn and H. Bockhorn, *Polym. Int*., **57** (2008) 722-729.

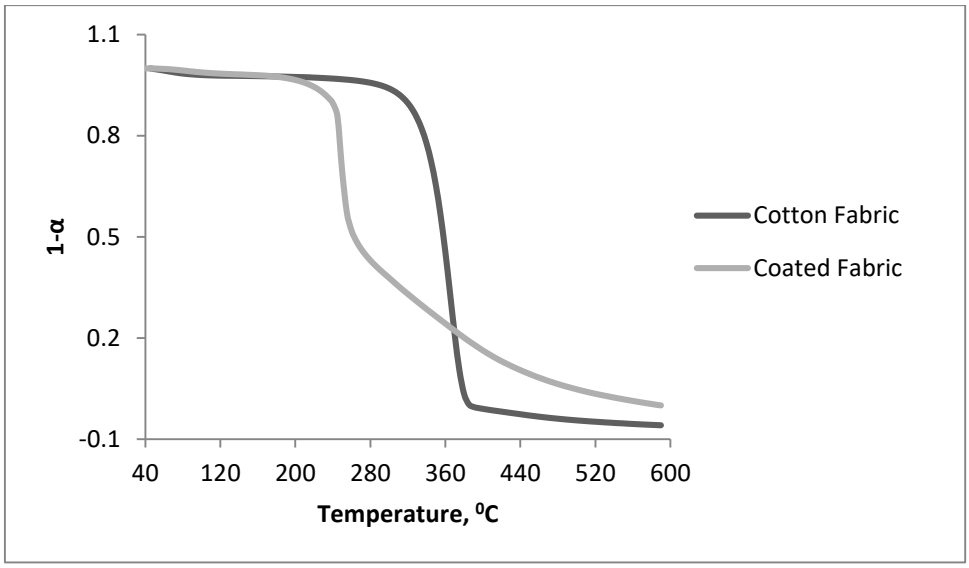


**Fig 1: TG curves in Nitrogen at heating rate of 15°C/min of Cotton Fabric and Coated Fabric**

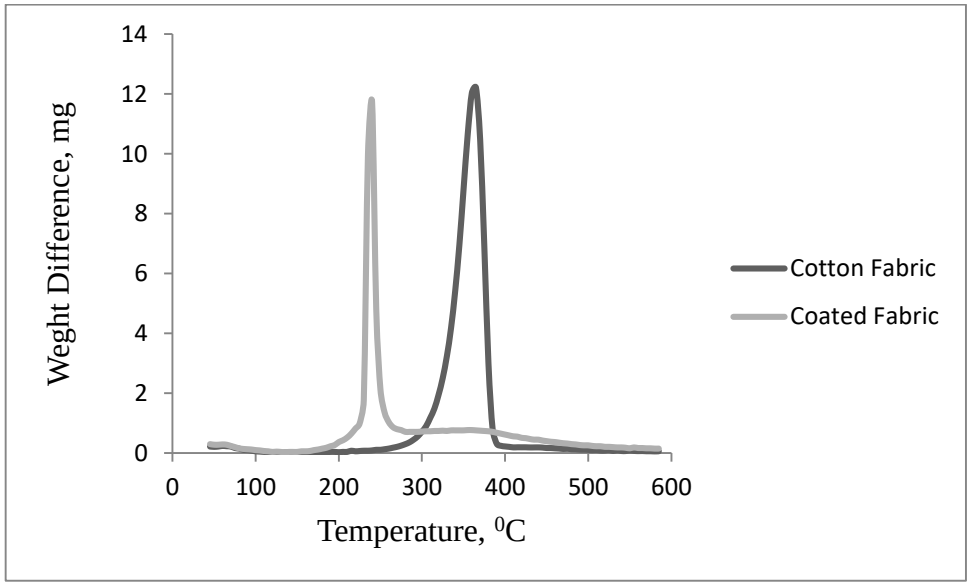


**Fig 2: DTG curves in Nitrogen at heating rate of 15°C/min of cotton fabric and Coated fabric**

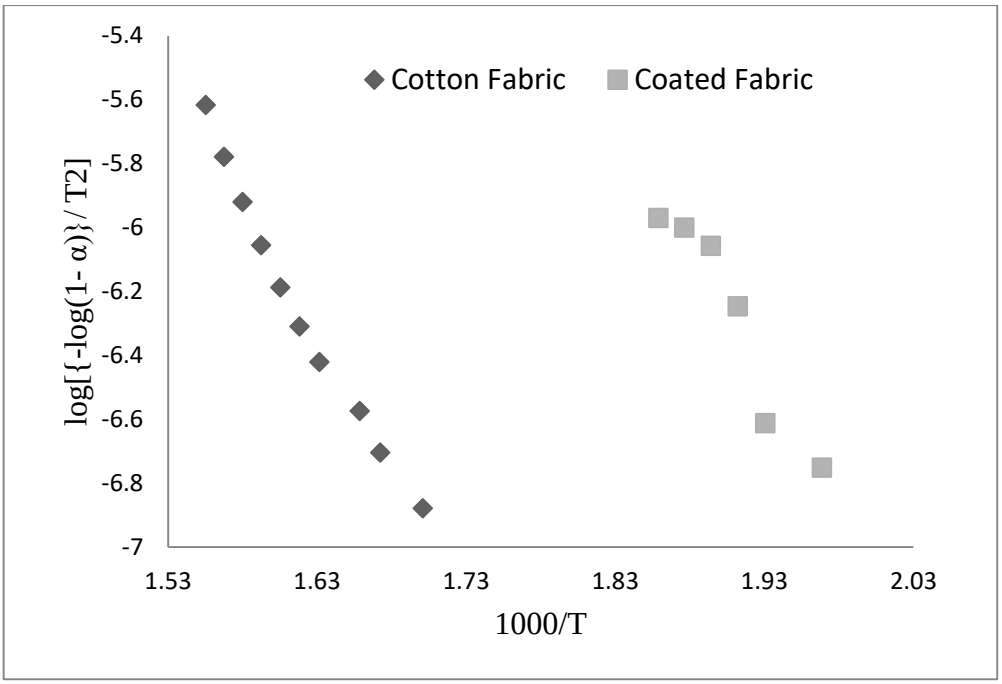


**Fig 3: Coats-Redfern plot for first stage of thermal degradation of cotton fabric and coated fabric**

**Table 1: DTG peaks, LOI and Char Yield and Kinetic Parameters of untreated paper and its coated sample**

| Sample | TG Stages | Temperature Range ($^0$C) | Weight Loss (%) | DTG Peak ($^0$C) | 1-α range | Activation Energy, (kJmol$^{-1}$) | ln A (min$^{-1}$) | Char Yield (%) at 600 $^0$C | LOI (%) |
|---|---|---|---|---|---|---|---|---|---|
| **Untreated Paper** | Single | 285-390 | 75.2 | 361 | 0.9-0.1 | 163.5 | 22.73 | Nil | 18 |
| **Coated Paper** | First Stage | 200-280 | 31.5 | 241 | 0.9-0.49 | 137.9 | 22.20 | 26.3 | 26.4 |
| | Second Stage | 280-600 | 31.0 | - | - | - | - | - | - |