Development an new Fire retardant system based on an innovative synthetic textiles bounded with a intumescent fire retardant backcoating

Magniez Carole [1], Poutch Franck [3], Dubois André [1], Delobel René [2]

1. I.F.T.H Institut Français Textile Habillement
2. Laboratoire de Chimie et physicochimie des solides Ecole Nationale Supérieure de Chimie de Lille
3. C.R.E.P.I.M centre de recherche pour l'étude des procédés pour ignifugation des matériaux

<u>ABSTRACT :</u> The aim of the work is to develop innovative synthetic textiles bounded with a fire retardant backcoating at laboratory scale and to develop a methodology to study the fire behaviour of this new materials. The evaluation of fire behaviour is obtained by performing measurements on a cone calorimeter with heat flux of 30 kw/m². A method which allows to estimate combustion synergism coefficients between fire retardant resin and textile fibres is presented in this paper. The experimental results point out the effect of surface treatment by intumescent fire retardant resin on textile material during the combustion.

INTRODUCTION

In Europe, all national classes concerning the safety of construction products in case of fire will be replaced by harmonised system of test. The new regulation, including new tests methods to qualify products will become compulsory effective from January 2007. European directives are being introduced in order to minimise fire risk. Fire retardant Textile products for construction and building elements are concerned and will be prepared to meet the strict regulations required by European legislation.

In early 1996, the recommendations of 5 Th draft status report OCED stated that research activities for fire retardant system should be developed on formulations without halogen. Indeed, the toxicological and ecotoxicological properties of fire retardant materials involve the toxicity of combustion products. A major cause of fire fatalities is associated with the emission of toxic gas.

The present range of available flame retardant systems for textile backcoating is based on halogen derivatives. Moreover, flames retarding conventional fibres have an expansive cost**.** That why, cost reduction, recycling, environmental concerns, new regulations have put pressure on the research of innovative fire retardant systems for textile.

The use of intumescent systems could be an answer to flame retardant challenges in the textile industry **(1,2).** An intumescent system produces a so-called expanded char structure. This structure decreases the heat transfers and in consequence the degradation of the material. Many systems based on intumescent have been investigated in the field of paints (**3)**. However, they are not suitable for textile products because these systems contain high levels of fire retardant additives incompatible with the textile softness.

So, an alternative plan consists in applying a fire retardant backcoating formulation to textile fibres. Moreover the evolution of the technology of the machinery for coating presents clearly the will of research efforts in chemical diversification and process optimisation **(4,5)** . The objectives of the works are to develop an innovative product : synthetic textile fibres bounded by backcoating resin treated by fire retardant system based on an intumescent concept.

These paper reports the fire behaviour of different textile fibres bounded with a fire retardant resin. The cone calorimeter is an essential apparatus to study the fire behaviour. The evaluation of fire performances by cone calorimeter is based on scientific measures (rate of heat release) even though the predictive tools for the characterisation of fire behaviour for textiles is mostly based on an empirical approach **(6,7,8)**. It's why, this paper examines the fire synergism coefficient in order to estimate the thermal protective property of intumescent coating using results recording by the cone calorimeter and a new methodology.

EXPERIMENTAL

Polyamide, polyester and polypropylene non wovens were used in this work as typical examples of furnishing fabrics. Different textile resin binders in water emulsion form were used in association with flame retardant totally free of halogens. The additives were incorporated at 15 % (wt/wt) in the resin.
Each coating paste was spread out, placed in an oven to dry at 100°C for 15 minutes and cured at 130 °C for 5 minutes to allow resin cross linking to occur. This technique permits to form a film or to bind textile fibres. In fact, the wet treated pastes were deposited on non woven textile, and the coated fabrics were dried in the same conditions. The coating add-on (40 % (wt/wt)) was determined by measuring the area and the weight of the rectangular sample $100\times100\times5$ mm^3 (figure 1).

Figure 1. photography of textile fibres bounded

Samples were exposed to a cone calorimeter according to ASTM 1354 under a heat flux of 30 kw/m² in horizontal position. This flux was chosen because the weight of sample is weak and it corresponds to the hazard fire start. The heat release is one of major parameter characterising the hazard of material. The values of rate of heat release (R.H.R) were determined by oxygen consumption **(9).**
A spark igniter was used to start gas combustion. Three sample of each coated material were investigated. All samples have the same weight and same surface.

The curves of rate of heat release difference between experimental and calculated RHR curves allow to point out the efficiency of carbon char. These curves are computed as follows :

$$\mathbf{Cos\ (t) = RHR_{exp}\ (t) - RHR_{th}\ (t)}$$

The $\mathbf{RHR_{Th}}$ **(t)** are computed as follows :

$$\mathbf{RHR_{th}\ (t) = (x/100)*R_f\ (t) + (y/100)*R_l\ (t)}$$

- $\mathbf{R_f}$ **(t)** rate of heat release of textile fibres in accordance with time
- $\mathbf{R_l}$ **(t)** rate of heat release of coating in accordance with time
- **x** proportion of textile fibres and **y** proportion of coating
- $\mathbf{RHR_{exp}}$ **(t)** rate of heat release of coating in accordance with time

Thermogravimetric analyses were carried out at 10°C / minute under air flow using a Mettler TGA 50 thermobalance. The weight of sample were around 30 mg.

RESULTS AND DISCUSSION

Fire behaviour and thermal degradation of backcoating

As can be seen from table 1 and figure 2, the using fire retardant system based on intumescence phenomenon results in decreasing the rate of heat release of resin binder.

Table 1 : cone calorimeter data

Reference	RHR kw/m²	Ignition time
Resin non treated	816	59 second
Resin treated	572	45 second

The study of the thermal degradation and the determination of thermal degradation synergism coefficients (figure 3) based on the curves of weight difference between experimental and calculated TG curves (8) has shown that the development of intumescence carbon char formation starts at 350°C. The intumescence formulation develops a carbon char who plays the role of shield under heat flux (figure 4). Scanning electron micrographs of char indicate that the thermal shield is composed by little cells (figure 5). So, the material issue of the degradation of treated resin is composed of gaseous products in a cellular material. The carbon char has clearly three functions **(10,11,12)** :

- To trap combustion gas in cells, it involves a decreasing a heat release,
- To limit thermal transfers in the material: thermal insulation,
- To limit diffusion of oxygen to the material.

The degradation of carbon char begins at 600 °C.

Figure 2 : rate of heat release

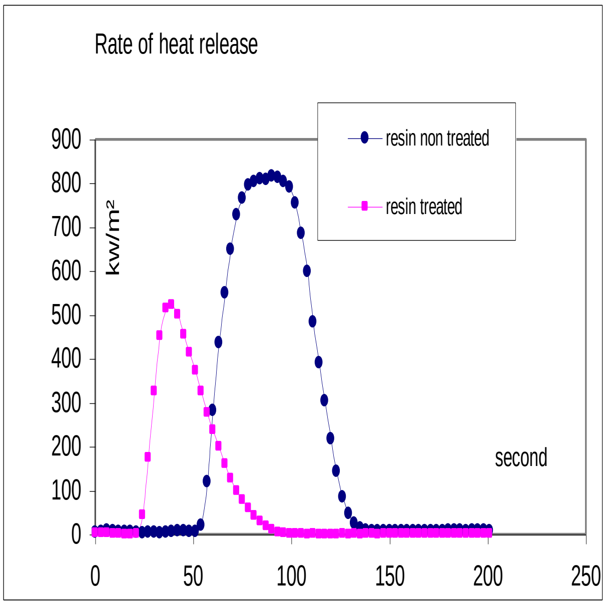


Figure 3 : thermal degradation synergism coefficient

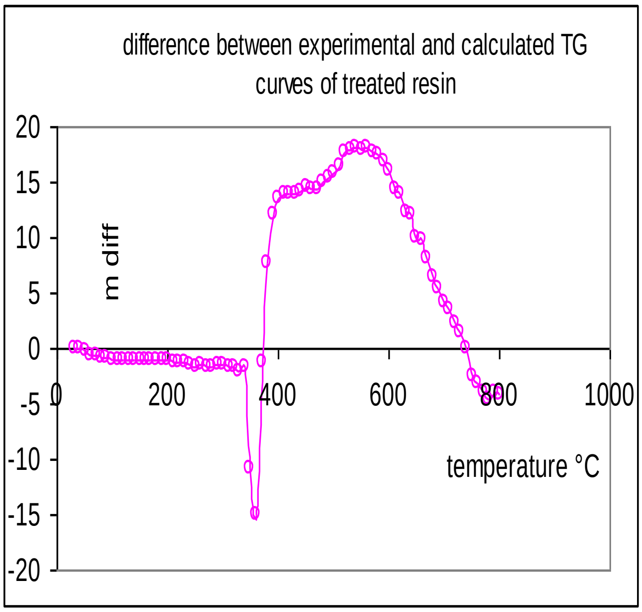
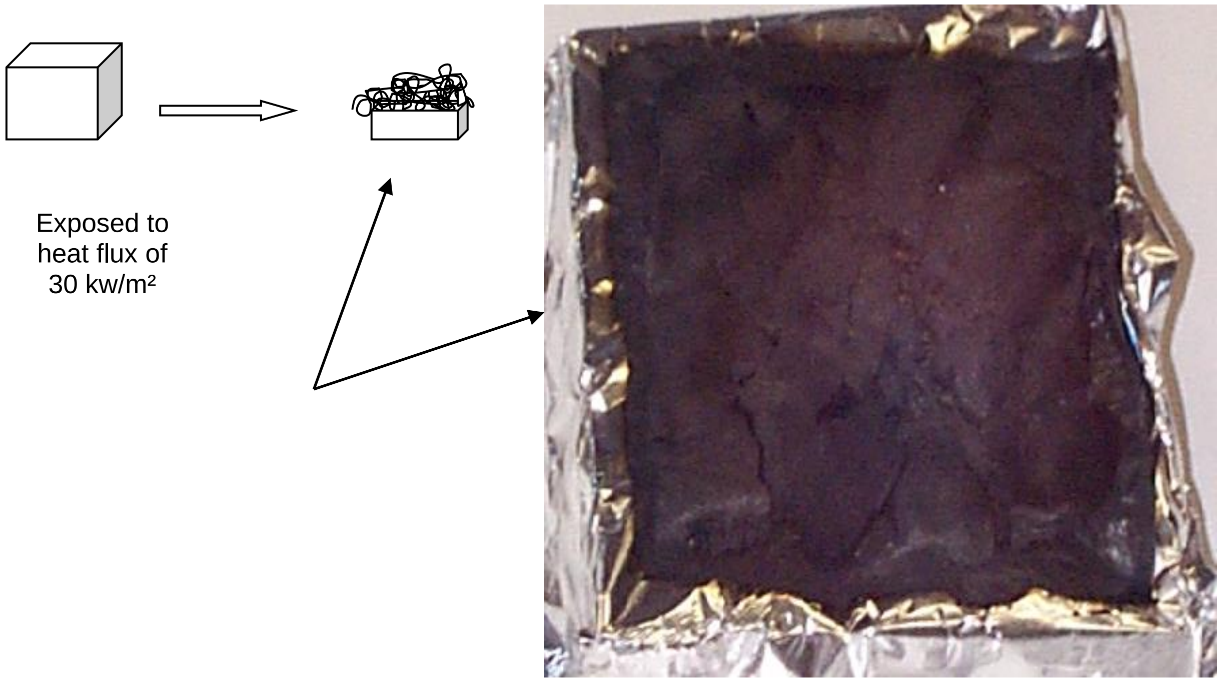


Figure 4 : photography of carbon char and schema of bubble model ***(13)***



Fire behaviour and thermal degradation of textiles fibres bounded by treated resin

The goal of deposited treated back coating is to make further degradation of textile fibres more difficult. Indeed, when an adherent and insulating layer of char is built up on the surface of textile fibres, carbon char insulate the materials from the flame **(14).**

Table 2 shows that for samples based on polypropylene or polyester non woven, the presence of treated resin decreases the peak of heat release. The ignition time is decreased. The fast formation of foaming carbon char involves a trap of combustion gas as early as the beginning of textile fibres degradation. Moreover, an early decomposition of intumescent system involves an increase of fire proofing properties of the material **(15).**

Figure 5 : scanning electron micrographs of carbon char issue of treated backcoating exposed to heat flux of 30 kw/m² for 5 minutes

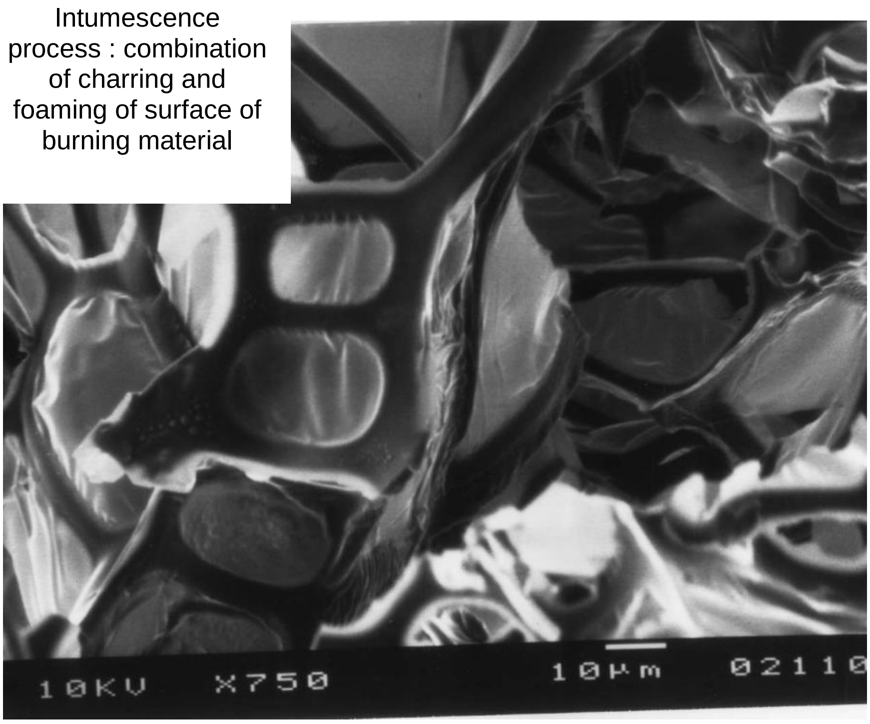
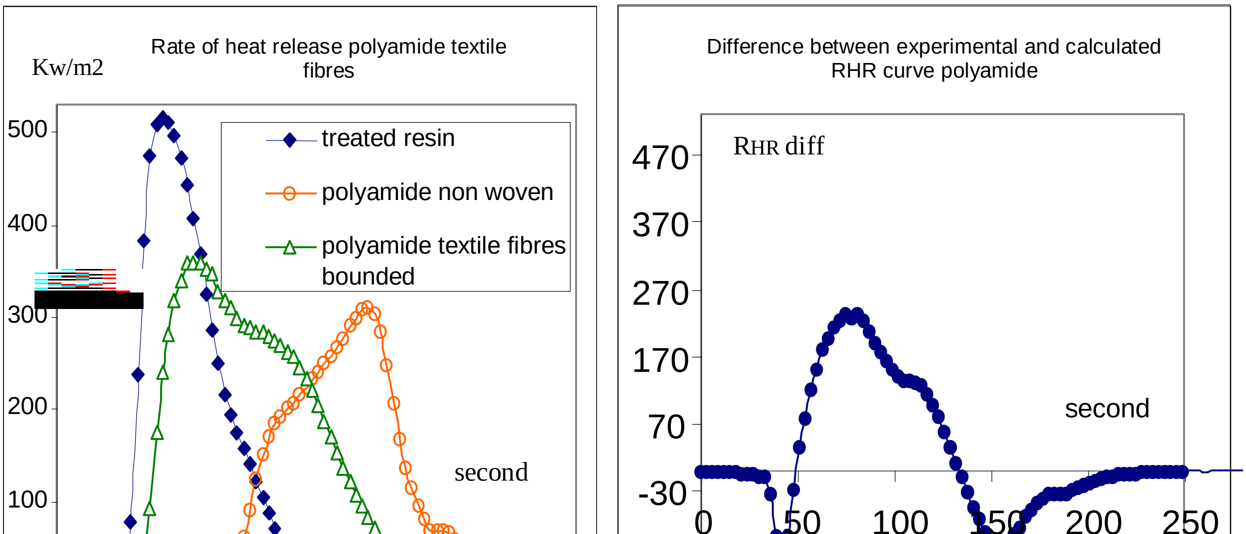


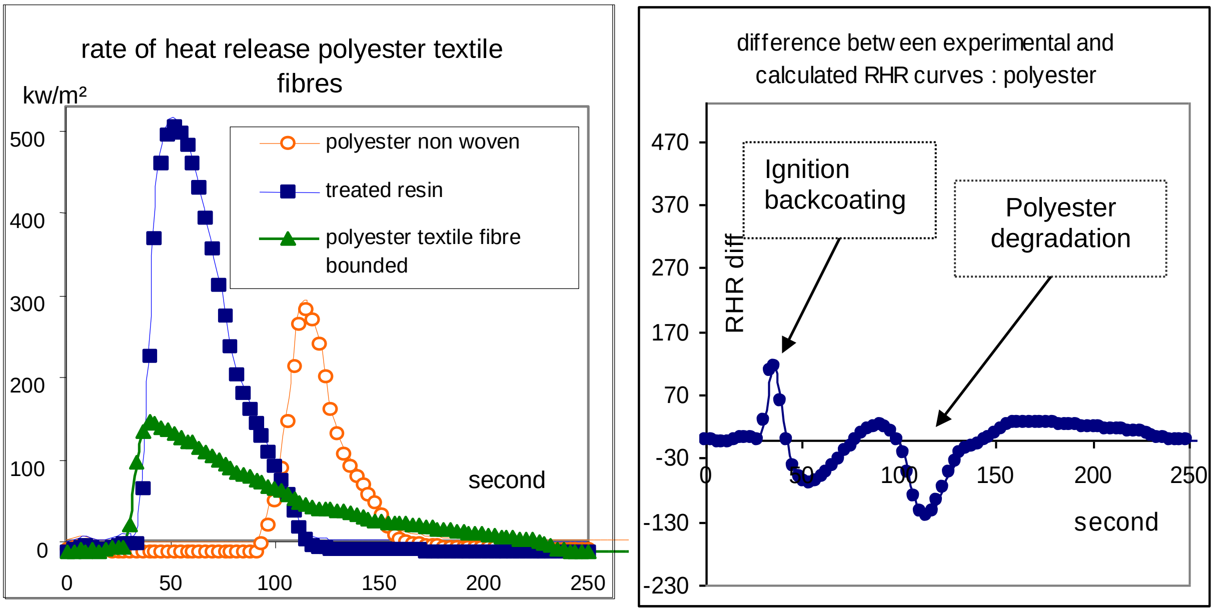
Table 2 : cone calorimeter data, heat flux of 30 kw/m²

Reference materials	Peak of heat release kw/m²	Total Heat release KJ	Ignition time second
Polyamide non woven	331	189	98
Polyester non woven	289	85	95
Polypropylene non woven	230	182	30
Polyamide bounded by treated resin	377	275	48
Polyester bounded by treated resin	153	172	37
Polypropylene bounded by treated resin	165	276	35

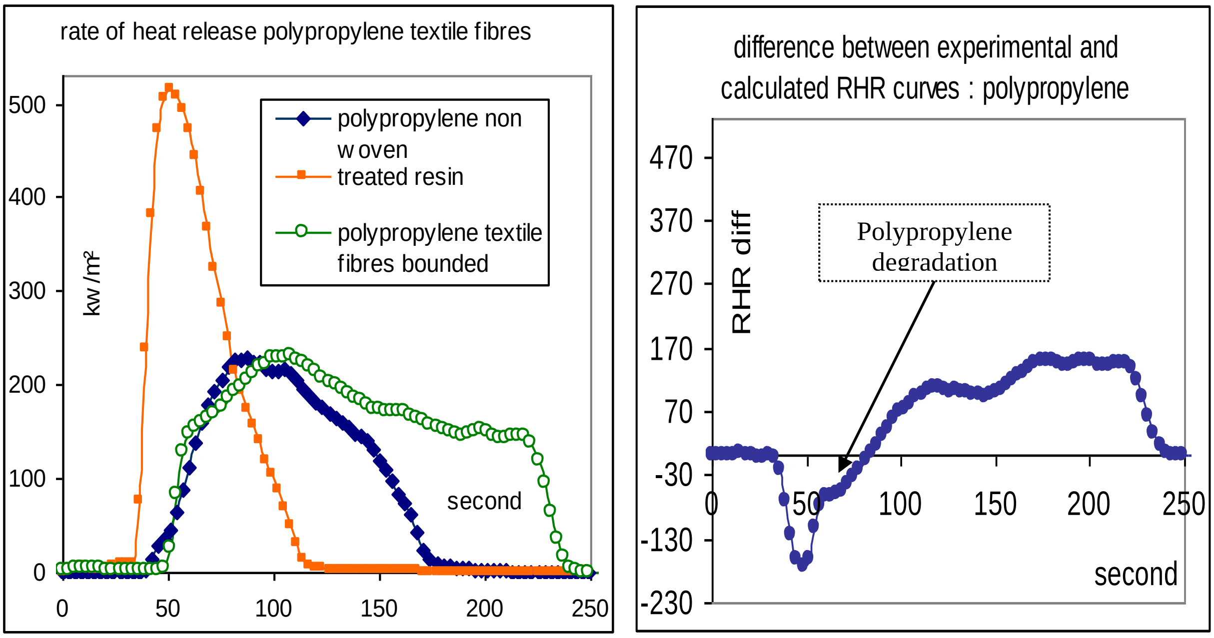
Figure 6 : rate of heat release and fire synergism coefficients : polyamide textile fibres and polyamide textile fibres bounded by treated resin



<u>Figure 7 :</u> rate of heat release and fire synergism coefficients : polyester textile fibres and polyester textile fibres bounded by treated resin



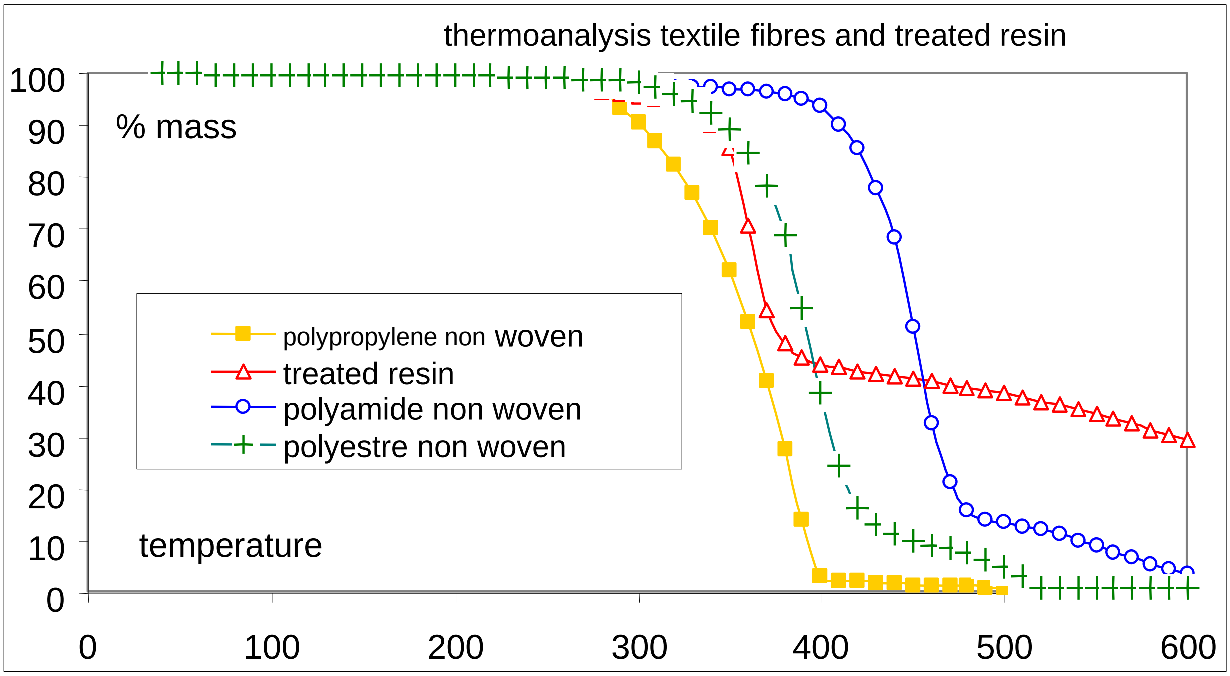
<u>Figure 8 :</u> rate of heat release and fire synergism coefficients : polypropylene textile fibres and polypropylene textile fibres bounded by treated resin



The curves of rate of heat release difference between experimental and calculated R.H.R curves (figures 6,7,8) point out that the degradation reactions of the association of textile fibres and binder resin, at the beginning of combustion (30-80 seconds as the case may be) are less exothermal ; except for the case of polyester textile fibres where the ignition of backcoating involves an exothermal peak at 35 second. The endothermal peak in curves of rate of heat

release difference constitutes proof that the carbon char issue of treated resin degradation plays the role of insulating shield. Moreover, it is interesting to note that for the polypropylene and polyester material bounded by the treated resin, the total heat evolved is the same. The carbon char limits just the peak of rate of heat release.
However, the power of insulating shield is not efficiency for polyamide textile fibres. If the curves of rate of heat release difference shows an important increase of heat release even though the textile fibres are not decomposed, the carbon char will not protect the textile support (figure 6).

Figure 10: thermoanalysis of synthetic textile fibres



The treated backcoating develops a carbon char, constituted by cells. The efficiency of power insulation of shield depends on the nature of the textile fibres. The carbon shield is particularly effective when the degradation of textile fibres are following on the degradation of resin treated by intumescent system, precursor of char (figure 10), If the timing of decomposition is respected, the char will trap the evolving gases and the rate of heat release will decrease.
For example , in case of polyamide fibres the char is destroyed before the degradation of textile support, so any gas could be trapped and the rate of heat release increase. That why, the studied resin is not suited to protect the polyamide non woven.

CONCLUSION

It is shown in this studies that intumescent system are excellent halogen free flame retardant for the binder resin dedicated to bind textile fibres. Coating the textile surface with resin treated by halogen free flame retardant is more beneficial from the point of view of toxic products of decomposition and of combustion than the use of classical halogen system based on chlorine and bromine. The treated resin and textiles fibres must have the same process of thermal degradation in order to make efficiency the carbon shield issue of the resin degradation and to reduce rate of heat release. The works will be now focus on research on intumescent concept for textile fibres and backcoating and on the decrease of quantity of charge. The method based on the curves of rate of heat release difference is adequate to estimate and classify the association of textile fibres and binder resin. The exploitation of this methodology permits:

-To understand the fire behaviour of the textile product s
-To orient the research on binder resin and intumescent concept
-To elaborate a predictive toolkit for the optimisation fire proofing textiles.

(1) Horrocks Innovation in fire retardant fillers for carpet backing journal coated fabrics vol 22 p 143 (1992)
(2) Horrocks, Kandola novel intumescents application to textiles journal coated fabrics
vol 27 p 17 (1997)
(3) Desfilhes Faut-il redécouvrir les systèmes intumescents, in elastiques modernes et élastomères according to conference René Delobel les plastiques et la sécurité au feu 8 june 1994
(4) Dubois enduction contrecollage in industrie textile n°1275 appril 1996
(5) Dubois , Rumeau enduction et contrecollage in industrie textile n°1317 february 2000
(6) Richard-Campisi, Le Tallec, Delobel, Houillon nouvelle methode pour déterminer la tenue au feu des matériaux en temps réel Techtextil symposium 1994

(7) Houillon, Richard logiciel d'évaluation de la tenue au feu des textiles utilisés dans le second œuvre Techtextil symposium 1994
(8) Richard, Brassart, Delobel prévision du comportement au feu industrie textile N°1252 march 1994
(9) Huggett, C,J. Fire and flammability,12 (1980)
(10) Le Bras, Bourbigot, Siat, Delobel, in Fire retardancy of polymers : the use of intumescence, new intumescent polymeric materials, p 266 the royal chemical society (publisher) cambridge (1998)
(11) Morice, Bourbigot, Leroy J.Fire Science 15, p 358, (1997)
(12) Bertelli, Camino, Marchetti, Costa, Locatelli in Fire retardancy of polymers Structural studies on chars from fire retardant intumescent systems (1998)
(13) Butler in polymeric foams science and technology ACS symposium series Chapter 15 (1997)
(14) Levchik,Wilkie in fire retardancy polymerics materials char formation p 171 (1999)
(15) Bourbigot, Le Bras, Delobel, J Fire sciences vol 13 (1995)