

A.R.Horrocks, *Inherently Flame Resistant Fibres in: Update on Flame Retardant Textiles: State of the Art, Environmental Issues and Innovative Solutions*, J. Alongi, A.R Horrocks, F. Carosio and G. Malucelli (Eds), pp. 178-206, Smithers Rapra, Shawbury, UK (2013)

5. Inherently Flame Resistant Fibres

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5.1 Introduction

Inherently flame retardant fibres are those which have no need of any external finish or treatment in order to impart some desired level of flame retardancy. There are principally three types of such fibre:

- i. Fibres which are based on conventional or the more common man-made or synthetic fibre structures to which flame retardancy has been conferred during modification of the manufacturing process.
- ii. Fibres that have a structure that is inherently flame retardant without any additional treatment during processing, and
- iii. Inorganic and ceramic fibres.

This chapter will review examples of these fibres that are currently commercially available, most of which were developed over 20-50 years ago and which have stood the test of time in spite of possible toxicological and commercial concerns that have affected their ability to maintain an acceptable fraction of the total market for such fibres in a variety of applications that have differing demands in level of flame retardancy coupled with their associated physical and general textile fibre properties. The development of these fibres as well as many others which have not seen successful commercial development have been reviewed in the past by a number of authors [1-5].

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5.2 Inherently Flame retardant Fibres based on Modified Versions of Conventional Synthetic and Regenerated Fibres

Table 5.1 summarises those major fibres falling into this group which include both synthetic and regenerated fibres.

Conventional synthetic fibres may be rendered inherently flame retardant during production by either incorporation of a flame retardant additive in the polymer melt or solution prior to extrusion or by copolymeric modification before, during or immediately after processing into filaments or staple fibres. However, problems of compatibility, especially at the high temperatures used to extrude melt-extruded fibres like polyamide, polyester and polypropylene and in reactive polymer solutions such as viscose dope and acrylic solutions have ensured that only a few such fibres are commercially available. A major problem in developing successful inherently flame retardant fibres based on conventional fibre chemistries is that any modification, if present at a concentration much above 10wt% (whether as additive or comonomer) may seriously reduce tensile properties as well as the other desirable textile properties of dyeability, lustre and appearance and handle, to mention but a few. Table 5.2 [2, 6, 7, 8] lists the flame retardant fibre properties of LOI and selected tensile properties of selected FR fibres listed in Table 5.1 compared with their typical non-flame retardant analogues. Thus the increases in flame retardant properties measured as increases in LOI are not matched by significant changes in tensile properties which means that fibre mechanical properties generally will be comparable to the respective unmodified analogues. Other properties such as dyeability, etc., are also little affected by the respective flame retardant modifications present.

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FR regenerated cellulose fibres: At the present time only two significant contenders, both based on the traditional viscose process, are commercially available – Lenzing FR[®] from Lenzing AG which comprises an organophosphorus and nitrogen/sulphur-containing species, Clariant 5060 [2, 2-oxybis) 5,5-dimethyl - 1, 2, 3 - dioxaphosphorinane - 2, 2 - disulphide) at 10-15 wt% loading and Visil AP (Sateri) which contains about 30 wt% polysilicic acid. The former was developed by Sandoz and Lenzing over 30 years ago [9] and remains today the most significant flame retardant viscose variant. The Visil fibre was originally produced by Kemira, Finland [10] and later manufactured by Sateri. The presence of the Clariant 5060 additive confers condensed phase, char-promoting activity in a similar manner seen for the phosphorus- and nitrogen-containing finishes applied to cotton described in Chapter 4. The polysilicic acid present in Visil viscose fibre is particularly interesting in that not only is it largely phosphorus-free but on heating, both a carbonaceous and siliceous char is formed. The aluminium salt aftertreatment (probably aluminium phosphate, hence the “AP” in Visil AP) raises the LOI from an otherwise value of 26-27 to about 30%, increases sensitivity to alkalis in the pH range 7-9 and also increases wash durability to acceptable commercial levels. The presence of the silica in the residue ensures that thermally-exposed fabrics revert to a ceramic char, thus affording high levels of protection to temperatures as high as 1000°C. Both flame retardant viscose types have acceptable tensile properties (see Table 5.2) as well as general textile properties.

Weil and Levchik [4] cite similar silicic acid-containing viscose fibres such as Daiwado’s Corona[®] and others made in China. The whole area of flame retardant regenerated cellulose fibres has recently been reviewed by Burrow [11].

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Polyester: While a number have been developed to semi-commercial scale, one group of flame retardant poly(ethylene terephthalate) fibres which continues to be successful is the well-established Trevira CS[®] range of products originally developed by Hoechst [12], which contains the phosphinic acid comonomer shown in Table 5.3. Phenyl-P substituted variants of this structure are believed to be produced in Korea and China [5]. Other flame retardant systems, both based on phosphorus-containing additives, are also shown although only the Toyobo GH (and variants) introduced in the 1970s have been commercially available, although their availability today is not known. The additive present is sulphonyl bis phenol phenylphosphinate oligomer present at about 7-9wt% [13]. The Rhodia Antiblaze 1045 additive is the former Mobil Chemical Antiblaze 19 compound which is available in dimeric form as a melt additive and in monomeric form as a polyester textile finish (Antiblaze CU) as described in Chapter 4. All three of these FR polyester variants do not promote char but function mainly by reducing the flaming propensity of molten drips normally associated with unmodified polyester. As yet, no char-promoting flame retardants exist for any of the conventional synthetic fibres and this must constitute the real challenge for the next generation of acceptable inherently FR synthetic fibres.

Polyamide: Inspection of Table 5.1 shows the presence of only one flame retardant polyamide 6.6 (Nexylon FR, EMS-GRILTECH) of unknown composition, although some properties are given in Table 5.2 and which was announced in September 2012 [8]. This paucity of FR polyamides reflects their high melt reactivities and hence poor potential flame retardant additive compatibilities. This problem has been further discussed by Weil and Levchik [5]. Flame retardants that are commercially acceptable for bulk and engineering aliphatic nylons such as PA6 and PA6.6 such as the poly(bromostyrene)/antimony and

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melamine cyanurate-based systems are unsuitable for fibres because of the high concentrations (>10 wt.-%) required and the associated reduction in fibre tensile properties [5]. However, the recent interest in metal salts (notably aluminium) dialkyl phosphinates by Clariant suggest that acceptable FR fibres might be possible since they manufacture a special low particle diameter ($D_{50} \sim 2\text{-}3\mu\text{m}$) fibre grades Exolit OP930/935. This phosphinate may be used alone or combined with melamine polyphosphate although in bulk polymers total levels of 15wt% or so are required for acceptable levels of flame retardancy. To date it is not known whether commercially successful PA6 and PA6.6 fibres based on this agent are available.

Acrylics and modacrylics: Flame retardant acrylics are usually so highly modified in terms of comonomer content that they are termed modacrylics. This latter group has been commercially available for fifty years or so [2] but at present few manufacturers continue to produce them. The principally preferred comonomer is vinylidene chloride and to enhance the flame retardant activity of the chlorine present, antimony III oxide (ATO) is included, although this may reduce the lustre of the fibres and resulting fabrics. Furthermore, the presence of both chlorine and ATO is causing environmental sustainability questions to be raised. The lack of real success of modacrylics in the furnishing sector is largely because of the superiority of back-coatings (see Chapter 4) applied to normal acrylic fabrics which create high levels of flame retardancy more cost-effectively and without affecting fabric aesthetics. There are currently no acrylic fibres containing simple additive systems.

Polypropylene: This fibre poses a particular challenge because of its low melting point ($\sim 165^\circ\text{C}$), its tendency to undergo random scission to highly flammable smaller hydrocarbons and complete absence of any tendency to form char [14]. However, because of the relative ease of producing PP fibres, manufacturers are more likely to produce their own flame

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retardant versions without use of specific brands and based on additive formulations which are traditionally based on bromo-organic species in the presence of a synergist. Examples of the former include tris(tribromopentyl) phosphate (FR-370, ICL) and pentabromoacrylate (FR-1025, ICL) both of which may be used without the need for an antimony III oxide synergist. Weil and Levchik's review [4] cites alternative synergists to include free radical generators such as 2,3-dimethyl-2,3-diphenylbutane (Perkadox® 30, Akzo) or the hindered amine radical stabiliser Flamstab® NOR 116 which is an N-alkoxy-2,2,6,6-tetramethyl-4-substituted morpholine as also reviewed by Zhang and Horrocks [14]. This latter enables lower than expected levels of bromo-organic species to be used [15].

Blends of conventional inherently FR fibres: Often an inherently flame retardant fibre is blended with a flammable or less flame retardant fibre to create a yarn and hence textile having intermediate flammability. As Tesoro and Rivlin showed over 40 years ago [16] and as reviewed by ourselves over 25 years ago [7], the final blend or composite (if the yarn contains doubled component yarns of each fibre type) rarely has a final flammability that is linearly dependent on blend composition. For example, the blends flame retardant viscose/PVC and PVC/polyester gave LOI values much less than expected from a weighted average of their individual component values. Also blends of aromatic high performance fibres such as polyaramid (see Section 5.3) such as aramid/cotton, aramid/polyester and aramid /wool show similar behaviour. In some of these examples, e.g. aramid/polyester and aramid /wool and flame retardant viscose/PVC, the lowest blend LOI value is lower than that of the flammable component alone usually when this was present in the 25-50% mass range as a result of the so-called scaffolding effect first seen for polyester/cotton blends [7, 17]. Positive deviations from the calculated linear trends were reported for aramid/modacrylic, FR

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viscose/aramid, FR viscose/wool and FR viscose/cotton blends [16]. Carroll-Porczyński [18] and Ishibashi et al [19] carried out similar investigations on a large series of 50/50 blends of flame retardant, chlorine-containing fibres such as PVC, modacrylic and polychal (copolymer of poly(vinyl chloride) and poly(vinyl alcohol) blended with most conventional natural and man-made fibres to show that the expected transfer of flame retardant activity from the chlorine presence in the former to the latter fibres is not always predictable. Thus blends containing PVC or modacrylic showed little if any expected transfer of activity until the chlorofibre content exceed 50% with FR viscose/modacrylic blends showing a modest positive effects in a 50/50 composition. More recently, we studied the more normal negative “S-shaped” trend as shown in Figure 5.1 for blends of modacrylic/Visil viscose when char length determined by BS 5438:1989, Test 2 (face ignition) was plotted against blend composition [20]. This trend actually shows a positive flame retarding effect since char lengths are minimal in the 50/50 blend region and so corroborates the earlier work cited above [7, 16-19] in that the inherently flame retardant modacrylic component with expected vapour phase activity is transferred to the greater char-forming Visil component only when present in the 30-70% range with a minimum char length at about 50%. Figure 5.1 also shows results following ignition application to the face and back of the single jersey fabrics and indicate that while they are little influenced by the mode of ignition, there is an effect of yarn structure. At lowest Visil contents the rotor-spun blends show lower char lengths and hence superior flame retardance; the converse is seen at the highest Visil blends. This is also certainly the case in blends of cotton and PVC. The Protex M (Waxman Fibres and Kanecaron) range of fabrics feature blends of the modacrylic Kanecaron Protex fibre with cotton in almost equivalent fractions as a 55% modacrylic/45% cotton composition. The

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fabrics are suitable for furnishings, bedding, protective clothing, including welding and similar hazard protection. Similar inherently flame retardant/wool blends also find commercial acceptance in a number of similar applications.

Blends may also be designed to replace part of a more expensive, high performance flame retardant fibre in a fabric as exemplified by a number of FR viscose/aramid blends developed over the years. While polyaramid and other high performance fibres are discussed in Section 5.3 below, 50/50 polyaramid/ FR Lenzing (Lenzing AG) [21] blends are used for protective clothing items. Furthermore, blending conventional flame retardant fibres like Lenzing FR[®] with high tenacity polyaramids increases wear and tensile characteristics. Other blend examples exist and will be discussed in the next section.

5.3 High Heat and Flame Resistant Fibres and Textiles Based on Aromatic and Cross-linked Structures

In the UK, Europe and the USA, still the majority of flame and heat resistant fibres and textiles are chemically after-treated; this probably constitutes a figure of about 80% by weight. Inherently flame and heat resistant fibres and textiles, including the inherently FR viscose and synthetic fibres above comprise the remaining percentage. Table 5.4 lists the main members of the group of high heat and flame resistant fibres currently available with transition temperatures, maximum service-use temperatures and LOI values [22]. It is evident that in the case of those having highly aromatic structures which have associated high second order transition temperatures, T_g , of 275°C or greater and ill-defined melting temperatures

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even higher, these fibres may be used at service temperatures of at least 150°C or higher since they show neither significant thermoplastic properties nor tendencies to thermally degrade below 350°C or so. Consequently, and unlike the inherently FR fibres based on conventional fibre chemistries described in the previous section, these fibres may be used in textile structures having both heat and fire resistance. Included in this table are the melamine-formaldehyde fibres, which although not aromatic in structure, are highly cross-linked and extremely char-forming in character. They also may resist temperatures of 150°C or higher during service life because they start to cross-link and then form char at temperatures above this level. All fibres in Table 5.4 have high char-forming potentials which are responsible for their low flammabilities and, as established by van Krevelen [23], their high LOI values, usually $\geq 30\%$.

Bourbigot et al. have produced one of the few recent comparisons of the fire performance of a number of these high temperature and fire resistant polymer-based fibres based on cone calorimetric data [24]. Figure 5.2 shows a comparison of the rate of heat release (RHR) versus time curves following the exposure to fabric samples at 75 kW/m² flux and the peak values in each are a measure of the maximum energy released during a simulated combustion. If each of the data sets in Figure 5.2 is expressed as RHR per second then the parameter known as fire growth index (FIGRA) results and this may be plotted against time to give a better measure of relative fire propagating behaviour. Figure 5.3 presents these results from which it is seen that the increasing fire growth property is in the order:

PBO < Kynol ~ PPTA < Technora < Oxidised acrylic

The decreasing order in terms of LOI as a fire measure is:

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PBO > Oxidised acrylic > < PPTA ~ Kynol > Technora

Both these measures used to order these fibre types indicate that PBO is the least flammable and hence most protective fibre while the respective orders for the other fibres differ. However, given that cone calorimetry is recognised to be a reasonable simulation of a fire, then the FIGRA order is probably closer to defining the relative fire protective behaviour of these fibres.

Each of these major fibre groups has been described elsewhere [22, 25] and so their major features only are presented below. The main groupings of these inherently heat and fire resistant fibres may be divided into the following, namely, the thermosets, the aramids and arimids, the polybenzazoles, the semicarbons and inorganics. Not only are the generic chemistries similar within each grouping, but their properties and potential application suitabilities are similar.

Thermoset polymeric fibres: Typified by the melamine-formaldehyde fibre Basofil® (BASF) and the phenol-formaldehyde (or novoloid) fibre Kynol® (Kynol GmbH), they have the common feature that when heated, they continue to polymerise, cross-link and thermally degrade to coherent char replicas. Derived chars have especially high flame and heat resistance as a consequence of their high carbon contents although their relatively low strengths prevent their being processed easily into yarns and so they are more often incorporated into nonwoven fabric structures. In addition, their inherent colour (pink for Basofil® and gold for Kynol®) ensures that they are usually used as a barrier fabric and not in face fabrics, although the melamine-formaldehyde structure in Basofil® does allow the fibre to be dyed with small molecular disperse dyes. Respective thermal properties are listed

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in Table 5.4, which indicates very similar high levels of heat and flame resistance. Typical end-use applications of both Basofil ® and Kynol® in thermal protection include fire blocking and heat insulating barriers and heat and flame protective apparel. Fibres may be blended typically with meta- and para-aramid fibres to improve tensile properties including strength and abrasion resistance in both nonwoven felts and fleeces for fire blocking aircraft seat fabrics and firefighter's clothing, for example. Such fabrics may be aluminised to improve heat reflection and hence fire performance.

The aramid and aramid family: This group is perhaps the most well-known and exploited of all the inherently heat and flame resistant fibres developed since 1960 and all members of this group are typified by having thermal resistances in excess of 300°C for short-term exposures and high levels of inherent flame resistance (see Table 5.4).

The most commonly used thermally resistant aramids are based on a meta-chain structure as typified by the original Nomex® (Du Pont) fibre and more recent commercially available fibres, e.g. Conex® (Teijin), Apyeil® (Unitika) and Fenilon® (Russia), in addition to modifications having modified tensile properties (e.g. Inconex, Teijin) and antistatic properties (Apyeil- α , Unitika). Nowadays these fibres have improved dyeing properties and are available in full colour ranges. They have the advantage acceptable "nylon-like" tensile and physical properties, they have minimal thermoplastic characteristics with second order transition temperatures, T_g , of about 275°C and an ill-defined melting point accompanied by thermal degradation starting at 375°C (see Table 5.4). They are ideal for use in protective clothing. Improvements in thermal performance in terms of increased char strength have been achieved by blending with small amounts of para-aramid fibres (e.g. Nomex III contains 5%

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Kevlar) and so is more suitable for applications where direct heat exposure is possible, e.g. firefighters' station uniforms, coveralls, jackets, trousers, gloves, flight suits or tank crew coveralls. Other variants include antistatic and moisture management properties (Nomex Comfort) and one specifically designed for firefighters' clothing (Nomex Outershell).

The para-aramids are typified by Kevlar[®] (DuPont) and Twaron[®] (Teijin) and are based on poly (para-phenylene terephthalamide) or PPTA. While having enhanced tensile strengths and moduli as a consequence of the extreme symmetry of their polymer chains and hence order or crystallinity, they also have improved thermal performance with a second order transition temperature of about 340°C and decomposing above 590°C (see Table 5.4). However, since thermal degradation is similar to that occurring in the meta-aramids, the LOI values are also similar at 30-31%. Their higher cost, poorer textile processing properties and higher modulus ensure that use in applications such as protective textiles are limited to 100% contents only when performance demands are exceptional and so more often than not they are used as minor blend components (e.g. 5% in Nomex III[®]).

A copolymeric derivative of the para-aramid fibres was introduced by Teijin in 1985 under the tradename Technora[®]. This fibre is based on the 1:1 copoly(terephthalamide) of 3,4'-diaminodiphenyl ether and p-phenylenediamine [22] and is claimed to have a much higher chemical resistance than PPTA as well as increased abrasion and steam resistance, which are useful properties in many protective applications. Technora[®] has a decomposition temperature of about 500°C and other properties are comparable with PPTA although its LOI value is slightly lower at 25%.

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Of a number of reported aramid fibres, only the example P84[®] introduced by Lenzing AG during the mid-1980 period and now produced by Inspec Fibres (USA) has been commercially exploited. As Table 5.4 indicates, these fibres have superior thermal properties to aramid and so find use in applications including protective outerwear, underwear and gloves, either as 100% or blended with lower cost fibres like flame retardant viscose. For instance, a 50/50 P84/Lenzing FR[®] (Lenzing AG) blend is available for knitted underwear with high moisture absorbency. Spun-dyeing of P84 fibres enables their natural gold colour to be replaced by those often demanded by customers who may require more appropriate and bright safety colours.

The final member of this grouping is the poly (aramid-arimid) fibre, Kermel[®], which was produced initially by Rhone-Poulenc of France in 1971 and is now produced by Rhodia Performance Fibres. Its overall properties are very similar to those of the meta-aramids and in 1993, a so-called third generation Kermel was announced claiming to have superior colouration properties. Typical of this group is its poor UV stability, and so must be protected from intense radiation sources. It competes in protective clothing markets where again it is used as 100% or as blends with other fibres, including FR viscose and wool. Composite yarns with high modulus aromatic fibres like the poly (para-aramids) have yielded the modification Kermel HTA[®], a yarn with a para-aramid core (35%) and a Kermel fibre wrapping (65%) to give improved abrasion resistance.

Polybenzazole group – polybenzimidazole (PBI) and polybenzoxazole (PBO) fibres: These fibre-forming polymers are so-called ‘ladder polymers’ and are essentially wholly aromatic polymer chains. The two common examples available commercially are the polybenzimidazole, PBI[®] (Celanese) with the full chemical name poly (2,2’-(m-phenylene)-

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5,5'-bibenzimidazole) and the polybenzoxazole, Zylon[®] (Toyobo) with the full chemical name poly(para-phenylene benzobisoxazole) and generic acronym, PBO. Their similarity in polymer chain structures and high degree of chain rigidities gives both of these fibres their superior thermal properties, as shown in Table 5.4, with thermal degradation temperatures well in excess of 400°C and superior LOI values well over 40%.

PBI has been introduced to the commercial markets only during the last 20 years or so in spite of its development during the early 1960s. The current PBI fibre is a sulphonated version of the above polymer and this improves shrinkage resistance at high temperature. Like many highly aromatic polymers it has an inherent colour – bronze – and cannot be dyed. The fibre is more often than not used as a blend and one well known blend is PBI Gold[®] in which a yarn spun with both PBI and Kevlar in a 40/60 blend. This gives rise to gold-coloured fabrics with fire protective properties claimed to be superior even to those made from Nomex III[®]. This blend is now well-established in firefighters' clothing both in the USA and UK for both outershells as well as underwear, hoods, socks and gloves. Other uses include industrial workwear, aluminised proximity clothing, military protective clothing and fire barrier/blocker applications and, because PBI is several times as expensive as the meta-aramids, this superior performance comes at a price.

Zylon[®] or PBO is a more recently developed fibre than PBI[®] and has outstanding tensile properties as well as thermal and fire properties superior to any of the polymer-based fibres mentioned in this chapter (see Table 5.4). While there are at least two variants of fibre, Zylon-AS and Zylon-HM of which the latter has the higher modulus, both have the same thermal and burning parameter values. Principal examples of thermally protective textiles include heat protective clothing and aircraft fragment/heat barriers where its price, similar to

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that of PBI, restricts its use to applications where strength, modulus and fire resistance are at a premium.

Semicarbon: The semicarbon fibres include any in which the structure is essentially carbon while retaining acceptable textile properties, unlike true carbon fibres [26]. Within the group, the oxidised acrylics represent the sole commercial group and are produced following controlled, high temperature oxidation of acrylic fibres during the first stages of carbon fibre production. Since the early 1980s, a number of commercial versions were announced including Celiox (Celanese), Grafil O (Courtaulds), Pyron (Stackpole), Sigrafil O (Sigr Elektrographit, now SGL) and Panox (SGL UK Ltd., formerly R K Textiles), many of which are now obsolete. Current examples include Panox[®] (SGL Carbon Group), Pyromex[®] (Toho Rayon) and Lastan[®] (Asahi). Their low tenacity creates the problem of ease of processability for these weak fibres, although they can be spun into yarns by the woollen system. Thus they are produced as a continuous tow that is stretch-broken by conventional means for eventual conversion into coarse woollen-type yarns. The limiting oxygen index is typically about 55% and so fabrics are extremely thermally resistant giving off negligible smoke and toxic gases when subjected to the even the most intense of flames. Unfortunately, the fibres are black and so are rarely used alone except in military and police coverall clothing where the colour is a bonus. Therefore, oxidised acrylic fibres are usually blended with other fibres, typically wool and aramid in order to dilute the colour and introduce other desirable textile properties. Because of their extreme fire resistance and lower cost than PBI and PBO, they find applications as blends in anti-riot suits, tank suits, FR underwear, fire blockers for aircraft seats and heat resistant felts (insulation), hoods and gloves. When aluminised, they are very effective in fire entry/fire proximity suits.

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Blends of high performance organic fibres: Blends of these fibres have been commercially exploited for a number of years with aramid-FR viscose being perhaps one of the first attempts to reduce the cost of the final fabric while maintaining a high level of fire protection. As also mentioned in the foregoing sections, blends of meta-aramids with para-aramid or PPTA (e.g. Nomex III[®], Du Pont; Kermel HTA[®], Rhodia) and PBI and para-aramid (PBI Gold[®], Celanese) have been developed to give a balance between the fire properties of both with the higher modulus and strength of the para-aramid. Very few, if any blends have been produced to generate synergies of fire resistance and other properties while, perhaps, improving other desirable features, including cost. Bourbigot et al [27] have recently reported a number of possibly synergistic blends made by mixing yarn by yarn of wool with PPTA gave improved the flame retardancy and generally improved thermal stability of the whole fabric. The suggested mechanism was that the molten char of wool coats adjacent para-aramid fibres, hindering the diffusion of oxygen to them and so negating their sensitivity to oxygen from the air and consequent thermal oxidative degradation. Subsequent research [28] has indicated that intimate blends of wool/PPTA show synergy when only 30% or greater PPTA is present as opposed to 70% or more in the previously blended yarn results. Furthermore, synergy was noted in wool/Technora[®] blends which can show reduced peaks of heat release rates with respect to 100% Technora[®]; some wool/PBO blends show similar encouraging results suggesting that the possibility of enhanced fire performance at reduced cost and improved aesthetics is feasible commercially using these interesting blends.

5.4 Inorganic and Ceramic Fibres

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Inorganic and ceramic fibres have no tendency to burn and so may be used in applications where high temperature and chemical reactivity of the environment combine to determine respective durabilities. These fibres include the various forms of glass, silica and alumina available, although more exotic ones like stainless steel, boron nitride and silicon carbide are available for specialist end-uses. Such extreme thermal protection as shown in Table 5.5 is a desirable feature in applications such as furnace linings or hot component insulation in car exhaust catalysts or around combustion chambers in jet engines where working temperatures and occasional flash temperatures are in excess of 500°C and even 1000°C in extreme circumstances. However, while fire resistance is an intrinsic feature of these inorganic fibres and textiles, their poor aesthetics limits their use to these extreme technical applications, although glass or ceramic-cored, organic fibre-wrapped yarns may be used to improve this feature.

The most well-established are the family of glass fibres which has been recently exhaustively reviewed [29] and continues to service many protective needs where heat and fire resistance are essential. The prime use has usually focussed on their reinforcing quality and so the fibres, because of an inherent brittleness and poor textile general character are used as reinforcing elements in flexible textile as well as rigid composite structures. Where glass fibre assemblies are used in non-reinforcing applications, they are usually contained to prevent fibre damage and loss of product coherence. Their use as filter media for high temperature gas and liquid filtration, as battery separators and as fire and acoustic insulation in aircraft and other transport systems are examples here. To respond to these different application needs, a wide range of glass compositions is available to suit many textile applications and fibres made from various compositions have softening points in the range

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650-970°C. When heated above 850°C, devitrification and partial formation of polycrystalline material occurs as the former glass fibres become more similar in character to ceramic materials. This devitrified form melts at 1225-1360°C, which is high enough to contain most fires for several hours. Recent examples of their use in high performance applications include their use in flexible roofings for sports and similar constructions – the recently developed and installed roof of the Olympic Stadium in Berlin with a surface area of 42,000 m² is a recent example in which the upper roof is made up of a highly tear-resistant fiberglass fabric of coated with PTFE (polytetrafluoroethylene), to give a lifespan of at least thirty years, excellent fire resistance and a self-cleaning surface [30].

Ceramic fibres on the other hand, have even poorer textile properties, are often more expensive and are mostly used as refractory fibres as insulating and fire barrier materials for applications requiring resistance to temperatures of at least 1000°C for prolonged periods. They tend to have polycrystalline structures, hence their exceptional high temperature characteristics. These often very specialist fibres, reviewed recently [31, 32], are not often produced in appropriate fibrous dimensions for normal textile processing and are more usually available as nonwoven or wet laid webs. This group may be classified into four major groups, namely, alumina-based, silica-based, alumina-silica and silicon carbide fibres.

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Alumina-based fibres reflect those of monocrystalline α -alumina fibres which while resisting temperatures up to 1400°C in oxidising atmospheres with little or no creep below 1600°C cannot be produced as fine, staple or continuous filaments. However, polycrystalline fibres such as Saffil[®] (Saffil Ltd., UK), are available as a lofty, nonwoven, wet laid web or “blanket” with a density of about 100 kg/m² and may be used in refractory and fire barrier applications at temperatures as high as 1600°C (see Table 5.5).

On the other hand, the silica-based fibres such as Quartzel[®] (Saint-Gobain, France), while having slightly inferior fire and heat performance, are available as continuous filament yarns, filament-based nonwovens and wet laid papers. Thus they may be knitted and woven to yield fabrics with applications in furnace *insulation*, combustion chamber insulation in aircraft, ablative composites for military and other markets and hot corrosive gas and liquid filtration. Falling between these two extremes are the alumina-silica fibres exemplified by the Nextel[®] range of products (3M, USA), which are determined by the alumina:silica ratio and rarely contain less than 60% alumina. Even small amounts of silica of the order of 3.0wt.-% permit the sintering of the transitional forms of alumina. This delays nucleation and growth of α -alumina up to 1300° C. Thus varying the amount of silica leads to a various forms of alumina-silica fibres with a range of high temperature behaviours. For example, Nextel 610[®] comprises 99% (being similar to Saffil[®]), Nextel 720 comprises 85% and Nextel 312 comprises 62% Al₂O₃ to give a maximum user temperature range of 1260-1370°C. These again are available in yarn, fabric and nonwoven forms for similar applications. Variations to the properties of these mixed oxide fibres, principally fibre physical characteristics like flexibility or strength retention at high temperature with time, may be made by introducing

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other oxides like boric oxide, B_2O_3 (Nextel 312 and 440) or zirconia, ZrO_2 (Nextel 650), although these do not enhance the maximum thermal working conditions.

Silicon carbide or SiC fibres are obtained by pyrolysing precursor fibres spun from an organosilicon polymer above $1200^\circ C$. As Table 5.5 shows these have some of the highest service use temperatures of all current ceramic fibres reflecting their extremely high melting points. Polycarbosilane or a derivative thereof is a typical precursor polymer and such polymers consist of six atom rings of Si and C. One commercial example of silicon carbide is Nicalon[®], (Nippon Carbon), which is available as cf tow for use in high temperature rigid composites with either polymer-based resin or metal matrices such as tungsten.

There are a range of other inorganic fibres such as basalt fibres (e.g. Basaltex[®], Marureel, Belgium) from a naturally-occurring complex silica/alumina/other oxide basalt rock similar to glass in composition, used as an asbestos-replacement and available in filament and nonwoven forms with claimed superiority to glass fibres in terms of temperature performance (see Table 5.5). Fibres such as Basaltex[®] are bronze in colour and available as continuous filaments with nominal diameters in the range 9 to 24 μm . Derived woven fabrics are used as fire barriers, thermal insulation and composite reinforcements.

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Table 5.1 Inherently flame retardant conventional fibres in common use.

Fibre	Flame retardant structural components	Mode of introduction
Regenerated:		
Viscose	Organophosphorus and nitrogen/sulphur-containing species e.g. Clariant 5060 [2, 2-oxybis) 5,5-dimethyl - 1, 2, 3 - dioxaphosphorinane - 2, 2 - disulphide), 10-15 wt%] in Lenzing FR® (Lenzing AG); 30 wt% polysilicic acid and complexes e.g. Visil AP (Sateri)	A A
Inherent Synthetic:		
Polyester	Organophosphorus species: Phosphinic acidic comonomer e.g. Trevira CS®, (Trevira GmbH); phosphorus-containing additive, Avora CS (KoSa); sulphonyl bis phenol phenylphosphinate oligomer, Heim/Toyobo GH (Toyobo, Japan); phosphorus-containing additive, Fidion FR® (Montefibre), Brilén FR (Brilén, Spain)	C/A
Polyamide	Unknown modification, Nexylon FR (polyamide 6.6) (EMS-GRILTECH)	
Acrylic (as modacrylic)	Halogenated comonomer (35-50% w/w) plus antimony compounds e.g. Velicren FR® (Montefibre); Kanecaron® (Kaneka Corp.); Sevel®FRSC (Fushun Huifu Fire Resistant Fibre Co., Japan)	C

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Polypropylene	Halo-organic compounds usually as brominated derivatives, e.g. tris(tribromopentyl) phosphate (FR-370, ICL); hindered amine stabiliser, eg. Ciba Flamstab® NOR 116, plus bromo-organic species	A
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Polyhaloalkenes	Poly(vinyl chloride) or chlorofibre, e.g. Clevyl (Rhovyl SA); Rhovyl (Rhovyl SA)	H
	Polyvinylidene chloride, e.g. Saran™ (Asahi Kasei)	C

Key

- A : additive introduced during fibre production
- C : copolymeric modifications
- H : homopolymer

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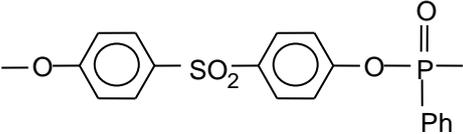
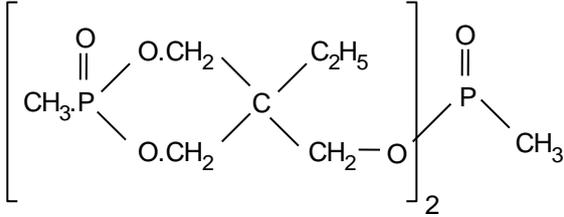
Table 5.2: Properties of inherently FR conventional (medium tenacity) staple fibres [2, 6, 7, 8]

Fibre	LOI, %	Tenacity, cN/dtex	Breaking strain, %
Viscose	18-19	2.1-2.5	15-20
Lenzing FR*	28	1.6-2.4	14-22
Visil AP	28-30	1.4-1.6	20-25
Polyester	20	3.5-4.5	30-50
Trevira CS	28-30	NA	-
Toyobo GH/Heim	30	NA	-
Nylon 6.6	21-22	3.5-4.0	30-40
Nylon 6.6 FR (Nexylon FR)	28	4.5-5.0	>50
Acrylic	18	2.0-3.6	20-55
Modacrylic (generic)	29-32	1.8-2.5	20-40
Polypropylene	18	3.0-7.0	15-25
FR polypropylene**	Est. >23	Est. ~3.0	NA
Polyethylene	18-19	2-3	~40
Poly(vinyl chloride)	37	1.6-2.0	~40
Poly(vinylidene chloride)	60	2.0	15-30

Notes: * taken from Lenzing web-site: <http://www.lenzing.com/en/fibers/lenzing-fr/specifications.html>; ** No commercial branded form currently is produced but use of additive systems should give the estimated values.

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Table 5.3: Flame retardant modifications for polyester fibres

Generic type	Nature	Structure
Phosphinic acid derivative (Trevira CS)	Comonomer	$\begin{array}{c} \text{O} \\ \\ \text{HO} - \text{P} - \text{Y} - \text{COOH} \\ \\ \text{X} \end{array}$ <p>X = H or alkyl Y = alkylene</p>
Bisphenol-S oligomer (Toyobo GH)	Additive	
Cyclic phosphonate (Amgard/Antiblaze 1045)	Dimeric additive	

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Table 5.4 Maximum service lifetimes for heat resistant fibres in thermally protective textiles [12]

Fibre genus	Second order temperature, °C	Melting temperature, °C	Onset of decomposition, °C	Maximum, continuous use temperature, °C	LOI, vol %
Melamine - formaldehyde	NA	NA	370	190	32
Novoloid	NA	NA	>150	150/air; 250/inert	30-34
m-Aramid	275	375-430 (decomp)	425	150-200	28-31
p-Aramid	340	560 (decomp)	>590	180-300	29-31
Copolymeric p-aramid	-	-	500	200-250	25
Arimid (P84)	315	-	450	260	36-38
Aramid-	<315	-	380	NA	32

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arimid					
Semicarbon	NA	NA	NA	~200/air	55
PBI	>400	NA	450/air;1000/inert	~300 (est)	>41
PBO	-	-	650;>700/inert	200-250(est)	68

Notes: NA=not applicable; (decomp)= with decomposition; (est) = value estimated value; (env) = depends on chemical inertness of the environment

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Table 5.5: Maximum service lifetimes for inorganic heat resistant fibres

Fibre genus	Second order temperature, °C	Melting temperature, °C	Onset of decomposition, °C	Maximum, continuous use temperature, °C	LOI, vol %
Glass	650-970	NA	850	>300	NA
Silica-based (eg Quartzel®)	-	>1700	-	1200	NA
Alumina-based (eg Saffil®)	-	>2000	NA	1600	NA
Alumina-silica-based (eg Nextel®)	-	>1800	NA	1260-1370	NA
Silicon carbide (eg Nicalon, Nippon Carbon)		2650-2950	NA	<1800	NA
Basalt (eg Basaltex)	-	1300-1700	NA	650-850	NA

Notes: NA=not applicable; (decomp)= with decomposition; (est) = value estimated value; (env) = depends on chemical inertness of the environment

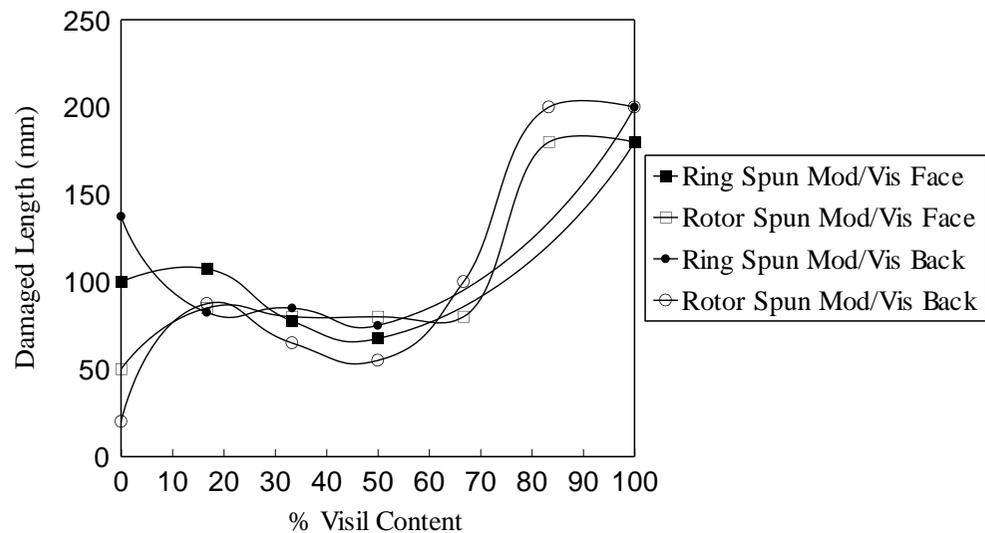


Figure 5.1: Effect of blend composition on the maximum damaged or char length of knitted modacrylic/Visil viscose samples tested on both back and face (BS 5438:1989, Test 2, face ignition)

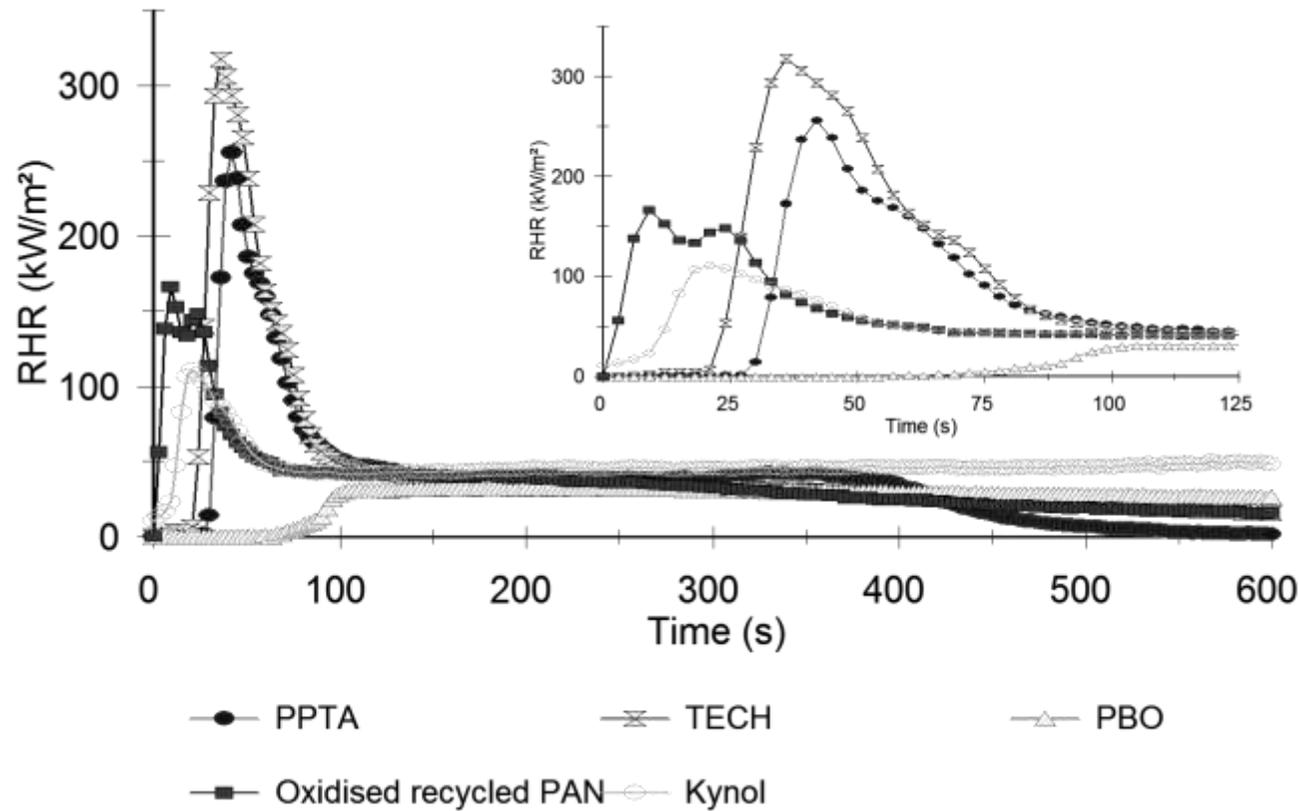


Figure 5.2: Rate of heat release (RHR) curves (external heat flux = 75 kW/m²) of selected knitted high performance fibres in spun yarn: Key: PPTA is poly(p-phenylene terephthalamide) (Kevlar), TECH is Technora, PBO is poly(benzoxazole) (Zylon), oxidised recycled PAN is a semicarbon and Kynol is a novoloid. (reproduced with permission from John Wiley and Son) [14]

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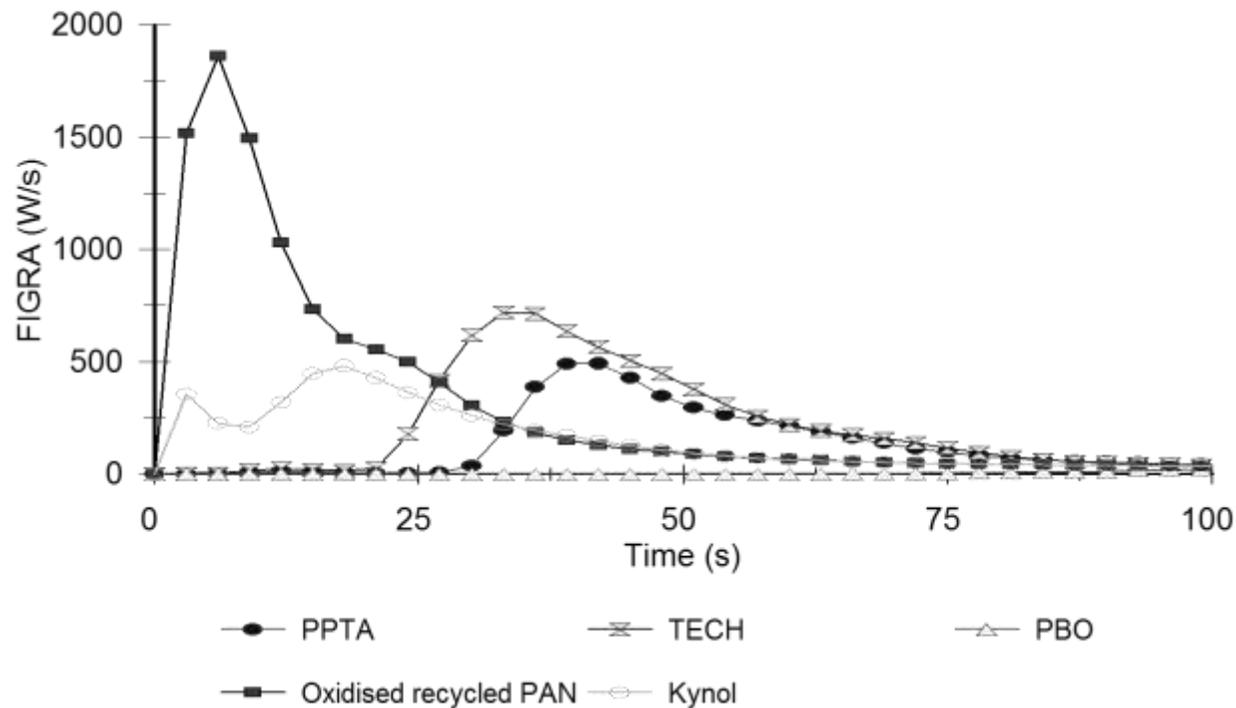


Figure 5.3: Error! Reference source not found. Key as in Figure 5.2 (reproduced with permission from John Wiley and Son) [14]