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### Soybean Fibre: A Novel Fibre in the Textile Industry

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#### 1. Introduction

Although natural protein fibres such as wool and silk have good physical properties and have been used extensively in the textile industry, they are relatively expensive to use and process. In silk, a large quantity of mulberry leaves is required for the production of a very small quantity of silk resulting in an increased cost of production. In addition, apart from the economic aspects, animal fibres are physically limited in several aspects. Firstly, both wool and silk fibre vary in diameter and their performance profile is limited. Secondly, morphologically, the presence of scales on wool surface results in felting shrinkage and difficulties in dyeing. In contrast, regenerated protein fibres, such as soybean fibre, do not have a theoretical limit in fineness to which fibres may be drawn. In addition, soybean is a competitive production material for fibres in the textile industry since it is abundant and cost-effective. Although the price of petroleum increased 21 times over the last fifty years, the price of soybean protein increased only 6.5 times (Agricultural Statistics Board, 1990; Monthly Energy Review, 1991).

Another advantage of soybean protein is its higher protein content (40%), compared to peanuts (25%) and corn (10%) and the environmentally friendly production claimed by the manufacturers (Huakang Ltd., 2005). Additionally, the possibility that a plant protein can be modified by molecular genetic techniques, provide the opportunity to improve the properties of the fibre in specific applications.

#### 2. Soybean fibre: definition and morphology

Soybean fibre is a man-made regenerated protein fibre from soybean protein blended with PVA (Zhang et al., 2003). Soybean fibres have a cream colour and their diameter is around 20  $\mu$ m (Vynias, 2006). Scanning Electron Microscopy (SEM) analysis of soybean fibre indicated longitudinal striations on the surface parallel to the axis, varying in length and depth, Figure 1 (Vynias, 2006). SEM micrographs of the cross-section of soybean fibre, Figure 2, showed a kidney bean-like cross-sectional shape. Recent research on the cross-section of soybean correlates well with the previous finding indicating kidney form shape (Jiang et al. 2004). Studies on cross-section (Tsai and Su, 1991). It was suggested that non-circular cross-sections occur due to high coagulation rate in wet spinning.



Fig. 1. SEM Micrograph of Untreated Soybean Fibre (Vynias, 2006)





#### 3. Brief history of soybean fibre

The first attempts to manufacture textile fibres from soybean protein were carried out in Japan (Kajita and Inoue, 1940a; Kajita and Inoue, 1940b) and U.S.A. (Boyer et al. 1945). The wet-spinning process included the extraction of oil to achieve an oil-free meal, extraction of proteins from the meal with alkali, dispersion of the alkaline proteins, fibre formation by passage through a spinneret into an acid coagulating bath and post-spinning treatments. However, the fibres were never commercially produced due to the lack of functional characteristics. After the end of the World War II, petroleum became the major source of synthetic textile fibres due to its low cost and case of processing, marginalising the commercial outlook of man-made protein fibres. However, due to environmental concerns, during the last decade there has been a renewed interest in soybean fibre and a production line has already been established in China (Huakang Ltd., 2005).

#### 3.1 Soybean: A globular protein

Soy proteins consist of several individual proteins and protein aggregates with a wide range of molecular sizes. However, the most important proteins in soybean are globulins. Globulins can dissolve above or below their isoelectric point (pI) and are insoluble near their pIs. Soy proteins demonstrate maximum solubility at pH 1.5-2.5 and above pH 6.3 whereas minimum solubility is obtained between pH 3.75 and 5.25 (Pearson, 1984). Soybean proteins are mainly composed of two storage proteins glycinin and  $\beta$ -conglycinin with their isoelectric point being between pH 4 and 5 are responsible for the insolubility of soybean proteins in that pH range (Nielsen, 1985). B-conglycinin is a heterogeneous group of glycoproteins composed of varying combinations of three subunits namely a',  $\alpha$  and  $\beta$  with their Molecular Weights (MW) being 58,000, 57,000 and 42,000, respectively. The subunits contain hydrophobic regions and link to form compactly folded trimers (Kinsella et al., 1985).

Glycinin is a large oligomeric protein with a MW of approximately 350,000 Daltons. Electron miscoscopy and X-ray scattering techniques have shown that the quaternary structure of glycinin is a pair of identical face to face hexamers (Kinsella et al., 1985). In addition, Pearson (1984) proposed that these hexamers consist of two subunits, acidic or basic in relation to their isoelectric point. It was suggested that acidic subunits had a pI of 4.6-5.4 with a MW of approximately 40,000, whereas basic subunits had a pI of 8.0-8.5 and MW of approximately 20,000 (Nielsen, 1985). Utsumi and Kinsella (1985) suggested that the possible molecular forces involved in the formation of glycinin and  $\beta$ -conglycinin in aqueous solution are hydrogen bonding, hydrophobic associations, ionic interactions and disulphide linkages.

#### 3.2 Amino acid analysis of soybean fibre

The amino acid analysis of soybean fibre is presented in Table 1, with the predominant amino acid being glutamic acid. Acidic amino acids (aspartic and glutamic acid) account approximately for 30% of the total amount of amino acids present in the fibre (Vynias, 2006). In contrast, the sulphur containing groups are present in low amounts, with the cystine content being low (0.1%).

#### 3.3 Manufacturing process of soybean fibre

Soybean protein is a globular protein in its native stage and is not suitable for spinning. Therefore it has to undergo denaturation and degradation in order to convert the protein solution into a spinnable dope. The manufacturing process of soybean fibre is presented in Figure 3.

#### 3.4 Denaturation

Denaturation of soybean protein can be achieved with either:

- i. alkalis;
- ii. heat;
- iii. enzymes<sup>1</sup>.

<sup>&</sup>lt;sup>1</sup> athough denaturation of soybean with enzymes is documented in the literature it has not been applied in the spinning of soybean fibre and therefore not presented in this chapter.



Fig. 3. Manufacturing Process of Soybean Fibre (Zhang et al., 2003)

Amino Acid	Abbreviation	Soybean	Nature of the Side Chain
Glycine	Gly	8.8	Hydrocarbon
Alanine	Ala	7.5	
Phenylalanine	Phe	4.4	
Valine	Val	6.3	
Leucine	Leu	9.8	
Isoleucine	Ile	4.8	
Serine	Ser	6.4	Polar
Threonine	Thr	4.3	
Tyrosine	Tyr		
Aspartic acid	Asp	12.8	Acidic
Glutamic acid	Glu	18.2	
Histidine	His	5.5	Basic
Arginine	Arg	0.8	
Lysine	Lys	3.9	
Methionine	Met	0.8	Sulphur-containing
Cystine	CysH	0.1	
Tryptophan	Trp		Heterocyclic
Proline	Pro	5.6	

Table 1. Amino Acid Analysis of Soybean Fibre (DWI, 2003)

#### i. Alkali Denaturation

Boyer et al. (1945) obtained a patent for the manufacture of soybean fibre. In this patent, it was suggested the use of xanthate in spinning of soybean fibre. According to the proposed formula ethyl alcohol reacted with sodium hydroxide forming sodium ethylate (ethoxide) in excess of sodium hydroxide. Further, carbon disulphide was incorporated into the sodium ethylate solution resulting in the formation of sodium ethyl xanthate, a compound with yellow- orange colour that was allowed to age from ten to thirty days. A spinning solution consisting of soy protein, sodium ethyl xanthate, sodium hydroxide and water was prepared with the protein content being approximately 18% of the solution. Xanthate was suggested to react with amino groups forming carbaminodithio groups (-NHCSSH) that prevent the gelation of the protein and improve its elasticity (Zhang et al. 1999). However, Motonaga et al. (1965) have also used sodium sulphite to break disulphide bonds in soybean, Figure 4.

 $R - S - S - R + SO_3^{2-} - R - S - SO_3^{2-} + RS^{-1}$ 

Fig. 4. Reaction Mechanism of Sodium Sulphite Breaking of Disulphide Bonds

Zhang et al. (1999) also utilised sodium sulphite to cleave disulphide bonds in order to denature the protein in the spinning of fibres from soybean protein and poly(vinyl alcohol) (PVA). However, little change was observed in the viscosity of the spinning solution. In contrast, the inclusion of urea was reported to decrease significantly the viscosity. The beneficial effect of urea on denatured soybean to prevent gel formation of the solution has been reported by Ishino and Okamoto (1975) and Ishino and Kudo (1980).

#### ii. Thermal Denaturation

It is known that heating denatures soy proteins and produces gels (Wolf and Tamura, 1969). When soybean protein is heated two irreversible processes take place:

- i. The interchange and degradation of disulphide bonds and sulphydryl groups may lead to irreversible crosslinking;
- ii. heating may result in fission of the protein polypeptide.

Gel formation is not beneficial in fibre spinning with a number of studies reported in the literature on the thermal denaturation of soybean (German et al., 1982; Petruccelli and Anon, 1995; Sorgentini et al., 1995). In addition, the denaturation temperatures of  $\beta$ -conglycinin and glycinin have been reported to be in the 72-80°C and 85-95°C range, respectively (Arrese et al. 1991; Berli et al., 1999).

Zhang et al. (2003) reported benefits in terms of viscosity stability when thermal denaturation was used compared to alkali denaturation in the spinning of PVA/soybean blend fibres. The viscosity of the spinning solution was thermally stable at 70°C for approximately 3 hours. In addition, it was noted that the alkali-denatured solution was not suitable for the fibre spinning of soybean protein/PVA blends with only thermal denaturation providing benefits.

#### 3.5 Coagulation

The solution consisting of soybean and PVA is filtered and forced through the spinneret where orientation of the molecular chains occurs. The chain molecules are then arranged into a structure consisting of crystalline regions and amorphous regions. In the crystalline regions the molecules are closely packed and responsible for the strength and rigidity of the material, whereas in the amorphous regions are less closely packed and less ordered, creating flexibility and accessibility (Cook, 1984).

The orientation is highly maintained in the two consecutive coagulation baths of sodium sulphate and ammonium sulphate in water with 1M sulphuric acid (Zhang et al., 2003). The temperatures of the initial and last coagulation baths are maintained at 50 and 70°C, respectively, based on the spinning conditions of PVA (Sakurada, 1985). The cross-section of fibres produced is dependent on the coagulation rate and the blend of soybean/PVA used (Zhang et al., 2003). The next processing step followed is crosslinking in order to enhance the mechanical properties.

#### 3.6 Crosslinking

In the production of soybean fibres, after winding the coagulated fibre is passed into a crosslinking bath that contained formaldehyde (Boyer et al., 1945). Formaldehyde was one of the first crosslinking agents used with proteins. It is known that formaldehyde can react with amino, amido guanidyl, hydroxyl, phenolic and indole groups and reduce disulphide bonds (Carter, 1971). In addition, it was reported that formaldehyde, at room temperature, forms methylene bridges between amino groups or primary amide/guanidyl groups in a wide range of pH (Fraenkelconrat and Olcott, 1948).

Although formaldehyde is cost effective and can crosslink protein fibres, it is an irritant, mutagenic in certain bacterial and animal species and has been classified as a probable human carcinogen (Petersen, 1987; U.S. Environmental Protection Agency, 1987; Monticello et al., 1989). Consequently, due to possible health risks crosslinking of soybean fibre following spinning has been carried out with reagents that do not contain formaldehyde and are still cost effective.

Acetylation is reported to improve the properties of regenerated protein fibres. Atwood (1940) showed that the water resistance of casein fibre was enhanced by acetylation. In addition, Evans et al. (1947) investigated the effect of acetylation on various properties of zein fibres and suggested good resistance to boiling in alkaline solutions following the modification. Acylation of soy proteins has been reported to change the conformation of glycinins and increase surface hydrophobicity (Barman et al., 1977). Huang (1994), reported an improvement in the wet-spun tensile properties of soybean fibres treated with acetic anhydride suggesting the reaction of ε-amino groups of protein by acetic anhydride, Figure 5.



Fig. 5. Acylation of ε-Amino Groups of Proteins by Acetic Anhydride (Huang, 1994)

Enhanced fibre properties, in terms of tensile strength, were produced when glutaraldehyde was used as a crosslinking agent following spinning (Huang et al., 1995). Difunctional aldehydes can be used as cross-linking agents for protein molecules and modify the amino group of protein, forming a stable Schiff base. The chemical reaction of proteins and difunctional aldehydes such as glutaraldehyde is presented in Figure 6.



Fig. 6. Crosslinking Reaction of Glutaraldehyde with Proteins (Huang, 1994)

In agreement with the findings of Huang et al. (1995), Zhang et al. (2003) showed that the mechanical properties of PVA/soybean blend fibre can also be enhanced with cross-linking with glutaraldehyde.

#### 3.7 Washing-drying-drawing

In the final stages of production the fibres are washed, dried and subsequently drawn in order to enhance tensile properties.

#### 3.8 Physical properties of soybean fibre

Moncrieff (1975) summarised the properties of various regenerated protein fibres, Table 2. In all fibres, lack of wet strength can be observed with the tenacity of soybean fibres decreasing from 0.8 g/den to 0.25 g/den when wet. Generally, regenerated protein fibres have a tendency to be weak.

Properties	Casein	Groundnut	Zein	Soybean	Collagen
Tenacity (cN/tex)	8.0-9.7	6.2-8.0	10.6	7.0	nd
Elongation, % (dry)	60-70	40-60	25-35	50.0	nd
Specific gravity	1.3	1.31	1.25	nd	1.32
Moisture regain, %	14.0	15.0	10.0	11	nd
Effect of	Softens	No softening or	Non-	nd	nd
temperature	on heating	melting on heating	thermoplastic		

where nd: not determined

Table 2. Properties of Regenerated Protein Fibres (Harris, 1954)

Cook (1984) suggested that the molecules in regenerated protein are not aligned with precision and regularity in order to form crystalline regions in the fibre. In addition, it was noted that molecules could not bond and align in order to achieve the tensile strength characteristics of fibres with crystalline structures.

#### 3.9 Efforts to enhance the tensile properties of soybean fibres

A serious defect of soybean fibres is their poor tensile strength, especially in the wet state (Cook, 1984; Saltzberg, 1985). Vynias (2006) demonstrated that soybean fibre consists of high amounts of glutamic and aspartic acid. Both amino acids are highly polar amino acids that can attract water. Huang (1994) suggested that hydrogen bonding between water and protein is competing with the hydrogen bonding between molecules. In an attempt to explain the low wet tensile properties of soybean fibre, it was proposed that when soybean fibre is in an aqueous environment hydrogen bonding between molecules is replaced by hydrogen bonding between proteins and water. X-ray diffraction studies have shown little or no parallel orientation of soybean fibres (Harris, 1954).

A number of studies have been carried out to improve the tensile properties of soybean fibres. Kajita and Inoue (1940a) incorporated lecithin into the spinning solution in order to increase fibre strength. Sugar and tartaric acid were also utilised to reduce the brittleness of the fibres. Huppert (1943; 1944) treated fibres with nitrous acid whereas Croston et al. (1945) strain hardened fibres by stretching them up to 300%. The introduction of petroleum-based fibres during the 40's and 50's altered the commercial aspect of soybean fibres. However, lately, due to economic and environmental issues, soybean fibres are considered a competitive material in the textile industry. Zhang et al. (1999) used polyvinyl alcohol (PVA) in order to improve the drawability of soybean fibres. PVA is a synthetic polymer with high tensile strength and modulus (Sakurada, 1985), and has been used as reinforcement in keratin fibres (Katoh et al., 2004). Zhang et al. (2003) investigated the processing of blended

PVA/soybean fibre and demonstrated that the incorporation of PVA into the spinning process enhanced mechanical properties, Figure 7.



Fig. 7. Tensile stress-strain curves for PVA/soybean blend fibers at various compositions: (a) PVA; (b) PS9010; (c) PS4060; (d) PS2080; (e) pure soybean protein fiber [Zhang et al. 2003, *permission required*].

#### 3.10 Aesthetic properties

The promotional marketing features of soybean fibre typically emphasised by the manufacturer (Huakang Ltd., 2005) are:

- i. natural lustre and smooth surface;
- ii. good physical and dyeing properties;
- iii. breathability and comfort; and
- iv. fine appearance with excellent drape.

#### 3.11 Differences between soybean, silk and wool fibre

Since the amino acid composition of soybean fibre is significantly different from wool or silk, it was thought that a comparison of soybean wool and silk fibre would be informative and interesting. The amino acid compositions of soybean wool and silk fibre indicate that differences do not only occur in the macroscopic view (soybean-globular, wool/silk-fibrous) but also in the molecular scale. Acidic amino acids (glutamic and aspartic acid) are in much higher amounts than in wool, with the predominant amino acid being glutamic acid. However, sulphur-containing amino acids such as cystine are lower than in wool, indicating less cross-linkages through disulphide groups (Stevens, 1990).

#### 4. Instrumentation and characterization of fabrics

#### 4.1 Surface characterization of fabrics

Scanning Electron Microscopy (SEM)

The morphology of untreated and modified soybean fibers was examined by SEM and was carried out on a Hitachi S-300N instrument. An SEM instrument is presented in Figure 8.



Fig. 8. A Hitachi 3700N SEM instrument (www.hht-eu.com)

#### 4.2 X-ray photoelectron spectroscopy (XPS)

XPS measurements were carried out with a Kratos Axis Ultra XPS instrument, Figure 9. The samples were analyzed with a monochromatic AlKa radiation source (1486.6eV) operating at base pressure of 3 ×10-9 torr. Wide survey spectra were recorded at a pass energy of 100 eV to determine the surface chemical compositions. High-resolution spectra were obtained at a pass energy of 20 eV to determine the chemical state of phosphorus. All surface analyses were obtained in triplicate in order to ensure reproducibility and confirm uniformity of treatment.



Fig. 9. Kratos Axis Ultra XPS instrument (http://xps.web.its.man.ac.uk)

#### 4.3 FT-IR Spectroscopy (Ferraro and Basile, 1978)

An FT-IR spectrometer basically consists of two parts: 1) an optical system which uses an interferometer and 2) a dedicated computer. The computer controls optical components,

collects and stores data, performs computations on data, and displays spectra. The real advantages of an FT-IR spectrometer result from the use of an interferometer rather than a grating or prism.

Compared to the conventional techniques FT-IR spectroscopy offers the following advantages (Frank, 1984): a) considerable saving of time; b) a better signal to noise ratio; c) high wave number precision; d) averaging of spectra produces better quality final spectrum. The schematic illustration of the Nicolet Magna-IR 750 spectrometer used in this study is shown in Figure 10.

The radiation from the infrared source is collimated and passed to the beam splitter of an interferometer. A part of the divided beam goes to the moving mirror whereas the other goes to the fixed mirror. The return beams recombine at the beam splitter resulting in interference. The reconstructed beam is then directed through the sample and focused onto the detector. The transmitted frequencies reach the detector in the form of an interferogram, which after Fourier transform, is converted to a normal spectrum.



1 Mid-infrared source; 2 Mirror; 3 Aperture; 4 Parabolic mirror; 5 Beamsplitter; 6 Interferometer; 7 Sample accessory; 8 Sample compartment; 9 Detector mirror; 10 Liquid nitrogen cooled detector; 11 Power supply; 12 HeNe Laser; 13 Slide mirror; 14 Beam port to microscope attachment.

Fig. 10. Diagram of Nicolet (750) FT-IR Spectrometer

**4.4 Attenuated total (internal) reflectance (Ferraro and Basile, 1978; Hesse et al. 1997)** ATR Spectroscopy is a non-destructive and sensitive surface analysis technique. The substrate to be analysed is pressed into intimate optical contact with a prism (crystal) that is transparent over the range of IR wavelengths to be studied. The IR radiation enters the prism and is incident on the surfaces of the prism at angles greater than the critical angle, with multiple internal reflections occurring. By varying the angle of incidence, it is possible to vary the number of internal reflections within the ATR element. In practice, up to 100 internal reflections may be employed. The substrate surface is pressed against the ATR prism and at each reflection the electric vector of the IR radiation samples the surface in contact with the prism. To obtain internal reflectance, the angle of incidence must exceed the 'critical' angle. This angle  $\theta_c$  is a function of the real parts of the refractive indices of both the

sample and ATR prism, Equation 1, where  $n_2$  is the refractive index of the sample and  $n_1$  is the refractive index of the prism. The evanescent wave decays exponentially with distance from the surface of the prism into the sample over a distance on the order of a few microns. Thallium bromide/iodide (KRS-5), zinc selenide or germanium, are typical crystal materials.

$$\theta_{\rm c} = \sin^{-1} \left( n_2 / n_1 \right) \tag{1}$$

#### 4.5 TGA measurements

TG measurements were performed on a TA Q100 instrument with a refrigerated cooling system (RCS), Figure 11. The thermal analyser was operated at a heating rate of  $10^{\circ}$ C/min and an environment of 100% N<sub>2</sub> gas flow with the gas rate being 50 mL min<sup>-1</sup>. The start temperature of measurements was 35°C and 0.1 mg of sample was utilized. The residual amount of char remaining after heating was determined after heating to 700°C. The maximum degradation rate point (MDRP) for each sample was determined as the temperature at, which each differential DTG showed a peak.



Fig. 11. Thermogravimetric analyzer (TA Q100 instruments)

#### 4.6 Limited Oxygen Index (L.O.I.)

The L.O.I. of soybean fabrics was measured according to BS 4599-2:1999 on a Stanton Redcroft FTA flammability test unit. L.O.I. values were calculated based on the following equation.

L.O.I.(%) = 
$$\frac{[O_2]}{[O_2] + [N_2]}$$
 (2)

#### 4.7 Vertical flammability test

The burning behavior of treated soybean fabrics was also assessed with the BS 5438 vertical flammability test method. Fabrics of  $180 \times 650$  mm length and width were mounted on a

suitable clamp and placed in a standard cabinet with a 2 mm/sec airflow. The bottom edge of the fabric was exposed to a standard flame for 1 sec and after the removal of the flame, the flame spread speed (mm/sec) was determined by the average of 10 measurements for both warp and weft directions.

#### 4.8 Yellowness Index

The Yellowness Index, YID, of the soybean fabrics was determined with a Datacolor International Spectrophotometer under illuminant D65/10<sup>o</sup> observer conditions.

#### 4.9 Tensile strength of fabrics

The soybean fabrics were conditioned for 24 h at 20°C and 65% R.H. before testing on an Instron 5564 testing system. Tensile strength of the untreated and Pyrovatex CP modified fabrics were determined as the average of 10 measurements in the warp direction using an Instron instrument, Figure 12.

#### 4.10 Evaluation of crease recovery

The crease recovery was evaluated by measuring the dry crease recovery angle. All samples were conditioned at  $20 \pm 2$ °C and  $65 \pm 5\%$  R.H. for at least 24 hours prior to testing. The crease recovery angle was determined in accordance with the BS EN 22313:1992. A press was utilised to apply a load of 10 Newton on an area of 15mm ×15mm of the folded specimen. The specimen were loaded for 5 minutes and transferred to the holder using a pair of tweezers. Reading of the crease recovery angle was obtained 5 minutes after the removal of the load, with the mean value calculated for the warp and the weft direction for both sides of the samples.



Fig. 12. Instron 5560 series instrument (http://instron.itrademarket.com)

#### 5. Wet processing of soybean fabrics

#### 5.1 Flame retardancy

An important factor of the quality performance of fabrics is the level of flame retardancy of fibres required to improved safety for the consumer. An inherent deficiency of soybean fibre

is its poor creasing performance. The flame retardancy of soybean fabrics can be imparted by utilising phosphorus and non-phosphorus reagents.

#### 5.2 Classification of flame retardants

The non-phosphorus containing flame retardants can be categorized into three groups:

- a. compounds with low decomposition temperature that produce a foam that creates a barrier between the flame and substrate such as boric acid and its sodium salts;
- b. compounds that sublime or release non-flammable vapours such as carbonates, halides, hydrated salts and ammonium salts
- c. compounds that cause dehydration and char promotion, such as sulphamic acid or ammonium sulphate.

The last group is of significant importance, since sulphamic acid and sulphamates have been known for many years as flame retardants for protein fibres and in particular wool (Aarons 1960).

#### 5.3 Mechanisms of flame retardancy

The development of flame-retardant finishing systems have been followed by intensive investigation into the mechanisms of flame retardancy with several theories being proposed due to the complexity and probable multiplicity of these mechanisms. A number of physical and chemical mechanisms have been developed in order to provide an insight into the flame retardancy of textiles fibres with the following being the most important (Benisek, 1978)

physical mechanisms

i.

ii.

- glasslike coating
- evolution of non-combustible gases
- adsorption of heat
- chemical mechanisms
  - modification of thermal decomposition
- reducing the production of combustible gases
- increasing char formation

According to the *first physical mechanism*, the flame retardant is decomposed by heat to form a glass-like coating that performs as a barrier between the fibre, the flame and the atmospheric oxygen. Consequently, the solid layer entraps the highly flammable tars of decomposition leading to a reduction in the combustion reactions. Inorganic compounds such as borax, boric acid and diammnoium hydrogen phosphate behave in this manner.

The *second physical mechanism* involves selected reagents that decompose at elevated temperatures yielding inert or low oxidisable gases. Flame retardancy is imparted by dilution of the flammable gases during combustion or by blanketing the material with an inert atmosphere. As a result, the oxidising atmosphere is either reduced or eliminated the fuel-to-air ratio is altered with higher temperatures required for ignition. Inorganic carbonates and halides, ammonium salts and hydrated salts belong to this category. Flame-retardants of the *third physical mechanism* can maintain the temperature of the fabric below the minimum combustion temperature. In addition they have to dissipate large amounts of energy from the flame front at a rate equal or greater than at which it is supplied.

*Chemical mechanisms* have been widely used to explain the role of flame-retardants. It was proposed that the flame retardant agent altered the degradation of the substrate, increased char formation and/or reduced the flammable gas production. In addition, the effectiveness

of volatile halogen derivatives resulted in the recognition of a radical-trap mechanism. Halogens such as chlorine or bromine trap radicals that are formed during oxidation such as  $H^+$ , OH- and HO-2, Figure 13.

 $RBr + H^{-}$ 

 $\rightarrow$  HBr + R<sup>·</sup>

Fig. 13. Entrapment of radicals during oxidation by halogens

#### 5.4 Sulphamic acid as flame retardant reagent for soybean fabrics

Sandoz (1955) patented a process whereby protein fibres could be treated with sulphamic acid providing a high degree of resistance to acid dyes. The mechanism of reaction was investigated and it was found that sulphation occurs on the free amino and hydroxyl groups of serine and threonine, whereas the sulphation with sulphuric acid occurs merely on the hydroxyl groups (Elliott et al., 1958).

In addition, wool treatment with sulphamic acid increased substantivity for basic dyes with improved wash and light fastness (Cameron and Pailthorpe, 1987). Lewin et al. (1975) have reported that wool sulphation with sulphamic acid can impart benefits in terms of flame resistance. Examination of Table 3 indicates the soybean fabric has a low L.O.I. value, suggesting it has relatively poor flammability properties in comparison to other proteinaceous fibers such as wool which typically has an L.O.I. value of 25–26% (Vynias, 2006).

SA treatment	Curing time	L.O.I	Yellowness	Strength loss
level, (% w/v)	(min)	(% ± SD)	Index	(%)
untreated		$18.8 \pm 0.1$	41.9	
10	0.5	$23.2 \pm 0.1$	44.5	nda
10	1	$22.9 \pm 0.3$	46.6	nda
10	2	$22.5 \pm 0.2$	47.3	nda
10	5	$22.0 \pm 0.1$	48.9	nda
20	0.5	$24.8\pm0.2$	46.9	12.5 (21.6) <sup>b</sup>
20	1	$24.7\pm0.1$	46.9	13.6 (22.7) <sup>b</sup>
20	2	$24.6 \pm 0.3$	48.9	16.5 (26.1) <sup>b</sup>
20	5	$23.1 \pm 0.1$	52.6	19.3 (28.9) <sup>b</sup>
30	0.5	$22.9 \pm 0.1$	47.1	ndc
30		$22.6 \pm 0.1$	48.2	ndc
30	2	$21.9 \pm 0.2$	49.4	ndc
30	5	$21.7 \pm 0.1$	53.8	ndc

a150°C, cure temperature, no urea included in pad formulation

<sup>b</sup>Strength loss of fabric cured at 170°C

° Not determined

Table 3. Effect of Sulphamic Acid (SA) Application Concentration on the L.O.I., Yellowness Index, and Strength of Treated Soybean Fabric

A 20% w/v sulfamic acid treatment level appears, however, appears to offer the most effective application level in terms of increasing the L.O.I. value, but that increased yellowing and strength loss is also associated with increasing the sulfamic acid application levels and the curing temperatures and time. The nature of this associated fiber degradation

must be related to the combination of elevated temperature and acidity progressively damaging the fiber and hence reducing the beneficial effect of the sulfamic acid treatments. Cameron and Pailthorpe (1987) similarly reported that the reaction of sulfamic acid with wool also resulted in an increase in fiber yellowness. Therefore, the need for short thermal exposure is apparent and SEM examination of the 30s treated fibers indicated little topographical change occurs in this period. Flame resistant finishing of wool with sulfamic acid in the presence of urea has been reported to increase weight gains, improve flame retardant performance, and enhance dissolution of the sulfamic acid in water.

Vynias and Carr (2006), similar to wool, found that application of 20% (w/v) urea with the sulphamic acid also increased the fibre modification/weight gain, increased the L.O.I. values, and lowered the increase in fabric yellowness. All these effects were achieved by probably increasing the uniformity of reaction throughout the fibre by increased accessibility. Elliot (1958) investigated the mechanism of the reaction and found that sulphation occurs at the free amino and hydroxyl groups in the wool. Vynias and Carr (2006) found with Fourier Transform Infrared (FTIR) analysis that the sulphamic acid treated soybean fibres also indicated that the hydroxyls were sulphated with an increase in the peak intensity observed at 1000 cm<sup>-1</sup>, which has previously been assigned to sulfate esters (Carr and Lewis., 1993). While the incorporation of urea into the formulation increased the L.O.I. values, the effect of increasing the cure treatment time was still to reduce the L.O.I. value (Table 4). This behavior was also reflected in the BS 5438: 1989 vertical flammability test where the although the fiber modification initially reduced the flame spread speed, with increasing treatment time the associated degradation of the fibre resulted in the flame spread performance again deteriorating. Obviously in any treatment for soybean fibres the acceptable balance between serviceability and fibre damage has to be established commercially.

Urea Concentration	Curing time	Weight gain	Yellowness Index	L.O.I.	Flame spread speed
(% w/v)	(min)	(%)	-	(%±SD)	(mm/sec)
-	-	-	41.9	$18.8 \pm 0.1$	-
0	0.5	7.9	46.9	$24.8 \pm 0.2$	3.0
0	1	8.4	46.9	$24.7 \pm 0.1$	5.0
0	2	9.1	48.9	$24.6 \pm 0.3$	7.0
0	5	9.3	52.6	$23.1 \pm 0.1$	10.0
20	0.5	10.9	45.8	$25.6 \pm 0.1$	2.0
20		11.6	46.7	$25.5 \pm 0.2$	5.0
20	2	12.5	46.8	$25.3 \pm 0.1$	6.0
20	5	13.2	48.9	$24.1 \pm 0.3$	10.0

Table 4. Effect of Urea Concentration on the Weight Gain, L.O.I., Yellowness Indices, and Flame Spread Speed of Soybean Fabric Treated with 20% w/v Sulphamic Acid

The TG curves of sulphamic acid/urea modified soybean showed a similar behaviour to those of sulphamic acid modified soybean, however there is a shift of the onset point to a higher temperature of approximately 190°C for all samples examined. In addition, the inclusion of urea, at comparable curing times, enhanced the char formation, Figure 15. It was observed that the addition of urea in the finishing bath enhanced the flame retardancy of all sulphamic acid treated soybean fabrics. However, low durability to laundering was

observed upon washing. A cationic agent (Matexil FC-ER) that was applied subsequent to the finishing treatment was found to be beneficial in terms of durability to laundering. Even though, in our work, sulphamic acid treatment of soybean fabric imparted improved flameretardant properties, the requirement in textile industry for a cost effective finishing treatment without aftertreatment is still evident.



Fig. 14. TG curves of untreated and sulfamic acid treated soybean fabric. (-, untreated fabric, -, sulfamic acid modified fabric, -  $\bullet$ -, urea/sulfamic acid modified fabric) (Vynias and Carr, 2006)

#### 5.5 Pyrovatex CP as flame retardant reagent for soybean fabrics

Phosphorus-containing reagents may provide an alternative process to this prospect. It is well known that phosphorylation with phosphoric acids and derivatives results in hydrolytically unstable ester bonds in flame-retardant finishing. Therefore, to overcome this negative aspect, Jones and Noone (1962) prepared more stable flame retardants based on phosphoric acid esters. Although Tesoro et al. demonstrated that phosphonoacetamide derivatives of cellulose impart acceptable flame retardancy, it has been shown that the most commercially successful phosphonates are the N-methylol diakyl phosphonopropionamides. Pyrovatex CP (Ciba) has been successfully used as a flameretardant reagent for textile fibers, such as cotton (Hebeish et al., 1994/Price et al., 1997), nylon (Yang et al., 1992), and lyocell (Hall et al., 1998). It is a phosphoruscontainingflameretardant (N-methylol diakyl phosphonopropionamide) with the structure shown in Figure 15. Kapura (1994) found using NMR and HPLC analysis that Pyrovatex CP does not consist of an individual compound but of several species based on dimethoxyphosphorylpropionamide.

Fig. 15. Structure of Pyrovatex CP

The effect of Pyrovatex CP on the flame retardancy of soybean has been recently studied (Vynias, 2010). The results indicated, Table 5, a slight increase in the L.O.I. values with increasing concentrations of Pyrovatex CP. Similar to soybean, little benefit was observed in terms of flame retardancy for cotton fabrics treated with Pyrovatex CP alone (Nakanishi et al., 1999) suggesting that additives were required. Tesoro et al. (1969) have demonstrated the synergistic effect of nitrogen on phosphorus compounds, resulting in enhanced flame retardancy, with the application of a melamine formaldehyde compound in a phosphonate flame-retardant finishing. Therefore, an etherified methylolated melamine reagent (Lyofix MLF), was incorporated into the pad formulation with the expectation to enhance flame retardancy (Vynias, 2010).

Pyrovatex CP	Lyofix MLF	L.O.I	Flame spread speed	Tensile strength	Yellowness
(% w/v)	(%  w/v)	(% ± SD)	(mm/sec)	$(kN/m \pm SD)$	Index
untreated	-	$18.8 \pm 0.1$	17	$17.6 \pm 0.1$	42.1
0	3	$19.3 \pm 0.5$		$13.6 \pm 0.8$	44.3
0	6	$19.4 \pm 0.3$	16	$15.0 \pm 0.6$	45.2
10	0	$20.5 \pm 0.4$	10	$14.5 \pm 1.1$	43.2
10	3	$23.4 \pm 0.5$	6	$16.2 \pm 0.7$	46.2
10	6	$23.7 \pm 0.3$	5	$16.8 \pm 1.1$	47.4
20	0	$21.1 \pm 0.3$	9	$14.4 \pm 0.5$	43.5
20	3	$24.3 \pm 0.4$	5	$14.9 \pm 0.5$	48.9
20	6	$24.6 \pm 0.2$	3	$16.1 \pm 0.6$	50.2
30	0	$21.0 \pm 0.1$	9	$13.9 \pm 0.7$	43.7
30	3	$23.1 \pm 0.3$	9	$15.1 \pm 0.8$	50.3
30	6	$23.5 \pm 0.1$	8	$16.3 \pm 0.5$	52.1

Table 5. Effect of Pyrovatex CP and Lyofix MLF on the L.O.I, Flame Spread Speed, Tensile Strength, and Yellowness Indices of Treated Soybean Fabrics

The inclusion of Lyofix MLF at 3% w/v application level enhanced flame retardancy compared to the Pyrovatex CP treatment alone, Table 5. This finding is not unexpected as melamine resins have been reported to be the most effective reagents for P-N synergism for cotton (Wu et al., 2003 /Wu et al., 2006). However, at higher concentration of Lyofix MLF (6% w/v), only a marginal improvement in the L.O.I. value was observed with a negligible beneficial effect on the flame-retardant properties. The vertical flammability test data support these findings where, although, the fibre modification with Pyrovatex CP initially reduced the flame spread speed, with increasing concentrations of Lyofix MLF an enhanced flame spread performance was achieved, presented in Table 5. However, at higher concentrations of Lyofix MLF an increase in yellowness was observed for all 10–30 % w/v concentrations of Pyrovatex CP examined, suggesting discolouration of the fabric. The surface of the fibre plays an important role in heat transfer within the material. Therefore, it was decided to investigate the effect of the chemical treatment on soybean surface utilizing SEM. SEM analysis of fibre indicated longitudinal striations on the surface parallel to the axis, varying in length, and depth, Figure 16(a).

In our trials, prolonged curing time (5 min) altered the structural characteristics of the fibre, Figure 16(b). Soybean became flatter with cracks appearing at the edges of the fibre, an indication of damage.



Fig. 16. SEM micrographs of (a) untreated soybean (b) cured at  $150^{\circ}$ C for 5min, (c) 20% w/v Pyrovatex CP, (d) 20% w/v Pyrovatex CP and 6% w/v Lyofix MLF.

Increasing concentrations of Lyofix MLF in the finishing bath improved the tensile properties of the treated soybean due to the rigidity caused by the melamine resin. Nevertheless, in all trials the tensile strength of the modified fabrics was still lower compared to that of untreated soybean.

#### 5.6 XPS analysis

XPS was used to monitor the deposition of Pyrovatex CP at the fibre surface and to identify changes on the atomic composition following the inclusion of the melamine resin. Untreated soybean, Table 6, does not contain phosphorus. However, examination of the P(2p) spectrum of Pyrovatex CP modified soybean indicated the presence of phosphorus species located at 135eV, which may be attributed to the P<sup>+5</sup> form of the phosphonopropionamide derivative, (Beamson and Briggs, 1992), Figure 17. Increasing the concentration of Pyrovatex CP resulted in an increase in the surface phosphorus, Table 6. Nevertheless, the incorporation of Lyofix MLF into the flame-retardant system, altered the surface atomic composition. An apparent increase in the N/P ratio was observed at all concentration levels of Pyrovatex CP examined. Raising the concentration of Lyofix MLF led to an increase in the surface nitrogen with the intensity of the N(1s) spectrum becoming higher with increasing concentration of Lyofix MLF, Figure 18.

Flame-retardant finishing with Pyrovatex CP/Lyofix MLF system was found to be more beneficial with regard to flame retardancy compared to Pyrovatex CP alone. However, in textiles, it is of great importance that the finishing treatment is durable to laundering. Therefore, the effect of washing on the flame retardancy performance was investigated. Successive washing cycles reduced the L.O.I. values for all treatments studied. However, after three washing cycles, the L.O.I. values for Pyrovatex CP were significantly reduced to the same value to that of untreated fabric, Table 7.

		$\langle \rangle$					$\langle -$	
Pyrovatex CP	Lyofix MLF	c	0	N	s	P	C/N	N/P
(% w/v)	(% w/v)							
unteated	-	74.7	19.8	3.2	0.3	-	23.3	-
20	0	71.6	22.9	5.1	0.2	0.5	14.0	10.2
x1	0	71.9	23.5	4.0	0.2	0.4	18.0	10.0
x3	0	72.4	23.7	3.3	0.2	0.4	21.9	8.3
20	3	68.8	23.7	7.2	0.2	0.5	9.6	14.4
x1	3	68.6	24.7	6.3	0.2	0.4	10.9	15.8
x3	3	69.6	24.9	4.9	0.2	0.3	14.2	16.3
20	6	68.7	22.9	7.9	0.3	0.5	8.7	15.8
x1	6	68.3	23.5	7.6	0.2	0.4	9.0	19.0
x3	6	68.6	23.9	6.9	0.2	0.3	9.9	23.0

Table 6. Effect of Pyrovatex CP and Lyofix MLF on the C/N and N/P Surface Atomic Ratios of Treated Soybean Fabrics



Fig. 17. P(2p) XP spectrum of 20% w/v Pyrovatex CP treated soybean, cured at 150°C for 30 sec.



Fig. 18. N(1s) XP spectrum of (a) untreated soybean, (b) 20% w/v Pyrovatex CP modified soybean, (c) 20% w/v Pyrovatex CP, 3% w/v Lyofix MLF, (d) 20% w/v Pyrovatex CP, 6% w/v Lyofix MLF.

, ,	Pyrovatex CP	Lyofix MLF	L.O.I	Flame spread speed	Yellowness
	(% w/v)	(% w/v)	(% ± SD)	(mm/sec)	Index
	untreated		$18.8 \pm 0.1$	17	41.9
	20	0	$21.1 \pm 0.3$	9	43.5
	x1	0	$19.1 \pm 0.2$	11	43.3
	x3	0	$18.8 \pm 0.3$	16	43.1
	20	3	$24.3\pm0.4$	5	48.9
	x1	3	$23.7 \pm 0.1$	7	48.7
	x3	3	$23.0 \pm 0.3$	10	48.5
$\sim$	20	6	$24.6 \pm 0.2$	3	50.2
	x1	6	$24.1 \pm 0.3$	5	50.0
	x3	6	$23.8 \pm 0.1$	5	49.8

Table 7. Effect of Laundering on the L.O.I., Flame Spread Speed and Yellowness Indices of Treated Soybean Fabrics

The flame spread speed increased, from 9 mm/sec to 16 mm/sec, resulting in a fabric that had low protection against the flame. In contrast to Pyrovatex CP treatment, the Pyrovatex CP/Lyofix MLF treated sample provided higher L.O.I. values after three washing cycles, demonstrating wash-durable flame-retardant properties. It is likely that the bonding of Lyofix MLF with Pyrovatex CP and soybean fiber is more resistant to hydrolysis during laundering than that between Pyrovatex CP and soybean. The highest flame retardancy performance was obtained with 6% Lyofix MLF with the loss in L.O.I. values reaching 3.3%

after three washing cycles, Table 7. In addition, the flame spread was approximately 70% lower than that of untreated soybean.

The effect of laundering on the phosphorus and nitrogen content of treated cotton is well documented (Horrocks et al. 1992/Wu et al. 2007). In addition, it is known that Pyrovatex CP is bound to cotton by its methylol group with the bonding being highly resistant to hydrolysis (Wu and Yang, 2006). However, to our knowledge, the effect of laundering on the fiber surface of flame-retardant fabrics has not been investigated. Therefore, the enhanced durability to laundering imparted in the Pyrovatex CP/ Lyofix MLF soybean fabric was investigated by the XPS technique to probe changes at the fiber surface following hydrolysis due to laundering. In the light of XPS analysis it was revealed that for the Pyrovatex CP modified soybean fabric, washing after one and three cycles reduced the N/P ratio at the fiber surface, Table 6.

In contrast, after three washing cycles, the N/P ratio for Pyrovatex CP/Lyofix MLF treatment increased with increasing concentrations of Lyofix MLF. Although surface phosphorus content (% atomic) was reduced in all treatments, it is evident that nitrogen was retained on the surface. Therefore, the benefits imparted by Pyrovatex CP/ Lyofix MLF system compared to the Pyrovatex CP system alone can be attributed to nitrogen "binding" on the surface, complexing with phosphorus and delivering associated enhanced flame retardancy.

#### 5.7 Crease recovery

Finishing techniques using physical or chemical approaches have been extensively used in textile materials with regard to improve functional properties. One of the most important functional properties is crease resistance with the consumer demanding garments manufactured to high specifications and crease-free appearance. Although synthetic fabrics demonstrate excellent crease resistance, in the case of natural fibres an inherent problem is the ease of creasing and poor crease shedding. Therefore a number of chemical treatments have been developed to overcome this drawback. In this section the mechanisms of crease resistance and a brief review of formaldehyde and non-formaldehyde based finishing treatments are discussed.

#### 5.8 Mechanisms of crease recovery

Crease-resist agents react with the hydroxyl groups of cellulose in the presence of heat and catalysts to form covalent crosslinks between adjacent cellulose chains (Welch, 2000). Therefore a three dimensional networked is created at the interior of the fibre that imparts increased resilience to the fabric. Any bending or deformation imparted to the fabric during garment use or laundering causes return of the fabric to the flat or creased configuration it had when the crosslinks were originally introduced. Marsh (1962), Figure 19, used a model in order to explain the fibre recovery from the deformations in the collapse state to impart dry crease recovery and in the swollen state to impart wet crease recovery.

Crosslinks between cellulosic chains in the diagram are represented by solid lines, while hydrogen bonds between chains are represented by dotted lines. The hydrogen bonding network remains intact in the collapsed state due to the crosslinking, delivering dry crease recovery. However, in the wet state, hydrogen bonding is disordered with covalent crosslinks between polymer chains imparting crease resistance.



Fig. 19. Diagram representing the dry crease recovery of the collapsed fibre (Marsh, 1962)

#### 5.9 Formaldehyde-free crease resistant finishing treatments

Marsh (1962) showed that the crease resistance of cellulosic fabrics could be enhanced with thermosetting resins such as formaldehyde and urea-formaldehyde. Formaldehyde is considered as the most efficient crosllinking agent for cellulose and protein fibres (Welch, 2000). However, despite its low cost, it is an irritant and mutagenic in certain bacterial and animal species and is classified as possible human carcinogen (Petersen, 1987; U.S. Environmental Agency, 1987; Monticello et al. 1989).

Therefore, the need to develop formaledehyde-free durable press finishing agents has become evident. A formaldehyde-free crosllinking agent in order to impart crease recovery should fulfil the following criteria (Welch, 2000):

- Stable in hard water
- Colourless and non-volatile during crosslinking
- Suitable for high-speed continuous fabric processing
- Less irritating, odorous, toxic or mutagenic than formaldehyde
- Available and low cost

Although a number of different reagents have been used as crosslinking agents to cotton to impart crease recovery such as glyoxal (Welch and Peters, 1987) and glutaraldehyde (Frick and Harper, 1982), N-methylol agents are the most common DP finishing agents applied to cotton with 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU) being the major formaldehyde-based DP reagent (Welch, 2000).

Petersen (1983; 1987) examined the mechanism of reaction of N-methylol and Nallkoxymethyl compounds with cellulose under acidic conditions and the relationship between the chemical constitution of these compounds and their reactivity towards cellulose. A number of substances could be classified with respect to their reactivity with cellulose and their hydrolytic stability following reaction with the fibre. Compounds such as methylol and alkoxymethyl derivatives of urea and of ethylene and propylene ureas are highly reactive but are moderately stable when reacted with the fibre. In contrast, low reactivity was demonstrated for 4,5-dihydroxy- and 4,5-dialkoxyethylene ureas and 4,6 dihydroxy- and 4,6-dialkoxy-5,5-dimethylpropylene ureas containing -NH<sub>2</sub>OH and -N-CH<sub>2</sub>OR groups. Nevertheless, the former substances are covalently bound to cellulose and show excellent resistance to hydrolysis.

#### 5.10 Crease-resistant finishing of soybean with polycarboxylic acids

One alternative development aimed at overcoming the formaldehyde release problem has been based on polycarboxylic acids. Compounds that possess two or more carboxyl groups in each molecule are referred to as polycarboxylic acids. The former reagents can form crosslinks in cotton through esterification of hydroxyl groups of adjacent cellulosic chains. However, it has been demonstrated that tricarboxylic acids are more beneficial with regard to crease recovery than dicarboxylic acids. Among the new crosslinking agents being investigated polycarboxylic acids appear to be the most promising agents (Welch, 1992; Welch 1994). Rowland et al. (1967) were the first to provide evidence that carboxylic acids could impart crease recovery to cotton coupled with strength retention.

In addition, Welch (1988) demonstrated that 1,2,3,4-butanetetracarboxylic acid (BTCA) was the most effective crosslinking agent for cotton cellulose, imparting high crease recovery values and durability to laundering. However, the relatively high cost of BTCA and the high application temperatures it required has prevented its use in the textile industry on a commercial scale.

#### 5.11 Effect of polycarboxylic acids on the crease recovery of soybean fabrics

Fixapret CP is a low concentration 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU) resin that has been widely applied to cellulosic fibres in order to improve end-use performance (Heywood, 1995). DMDHEU resins are based on the reaction of glyoxal, urea and formaldehyde and reacts with cellulose by etherifying the hydroxyl groups in the amorphous phase. The concentration of Fixapret CP had a beneficial effect on crease recovery resulting in higher values of crease recovery angle, Table 8. However, at concentrations (>100g/l) Fixapret CP had an adverse effect on crease resistance coupled to a marginal increase in yellowness. Nevertheless, the crease recovery obtained was higher compared to the untreated soybean. After the treatment with Fixapret CP it was revealed that curing times (>2min) offered little benefits in terms of crease resistance. In addition, the fabric became stiffer with a discolouration of the substrate being evident.

The effectiveness of maleic acid as a cellulose crosslinking agent has been examined by Welch and Peters (1997). Maleic acid contains two carboxyl groups per molecule and provides in the presence of phosphoric acid and sodium hypophosphite moderate to fair durable press properties for cotton fabrics (Welch, 2000). In soybean fabric, crease resist finishing of soybean fabrics with maleic acid did not impart any benefits in crease resistance regardless of the concentration used or the presence of the persulphate free radical initiator. The crease performance of fabrics was worse than that of the unmodified soybean.

Maleic acid (MA) and Itaconic acid (IA) are cheap dicarboxylic monomers that can undergo an esterification reaction with cotton cellulose. At elevated temperatures the molecules lose water prior to the formation of anhydrides (Marvel and Shepherd, 1959; Traskmorrell et al., 1990). Choi (1992) investigated the effectiveness of MA/IA system as a non-formaldehyde durable press finishing for cotton. It was found that treatment of cotton with MA and IA can improve durable press rating and crease recovery angle. In addition, it was suggested that a copolymer system of maleic acid and itaconic acid can be more efficient than MA alone through the formation of 5 or 6 membered anhydride rings by the dehydration of carboxyl groups in the polymeric backbone. The persulphate initiates free radical polymerisation of the unsaturated acids in situ, so forming a polycarboxylic acid species.

The 1:1 mole ratio of MA and IA, Table 8, improved marginally the crease recovery of soybean fabrics. However, when citric acid was used as polycarboxylic acid, an improved

484

crease resistance was observed in the treated fabrics. The best crease recovery value was obtained with a 100g/l citric acid concentration for 30 seconds. The effect of increasing curing time was to reduce slightly crease resistance. It seems that longer exposure times damage the fibre, resulting in a stiffer and harsher fabric.

Reagent	Curing temperature	Time	Yellowness	Recovery
g/1	°C	min	Index	Angle $(^{\circ}\theta)^{+}$
untreated			41.9	172
Fixapret CP <sup>a</sup>				
100	150	0.5	44.0	205
100	150	1	44.6	194
100	150	2	45.2	190
Maleic acid <sup>b</sup>				
50	150	0.5	43.9	170
50	150	1	44.0	165
50	150	2	44.1	160
Maleic-				
Itaconic acid <sup>c</sup>				
1:1	150	0.5	43.5	183
1:1	150	1	44.0	180
1:1	150	2	44.4	175
Citric acid <sup>d</sup>				
100	150	0.5	44.7	227
100	150	1	46.0	217
100	150	2	48.1	210

<sup>a</sup>: 12 g/l MgCl<sub>2</sub> were incorporated into the finishing treatment

<sup>b</sup>:  $20 g/1 K_2 S_2 O_8$  were incorporated into the finishing bath

c: 1:1 refers to the ratio of reagents in the pad formulation with a 20 g/1  $K_2S_2O_8$  added to the treatment

d: 40g/l formic acid and 60g/l sodium hypophosphite were added in the formulation bath

+: the sum of the warp and weft of the treated fabrics

Table 8. Effect of different chemical treatments on the dry crease recovery and yellowness indices of soybean fabrics (Vynias, 2006)

#### 5.12 Durability to laundering of citric acid treated soybean fabric

Citric acid was found to be the most beneficial among the polycarboxylic acids examined. In this study, the durability of citric acid treatment on the crease resistance performance of soybean was examined with the results presented in Table 9. It was showed that washing resulted in a decrease in the crease recovery angles. After three washing cycles the crease recovery angle for citric acid modified soybean was reduced from 227° to 195°, respectively. However, the crease recovery angle was still higher than the untreated. Nevertheless, further washing (x5 cycles) led to a crease resistance performance similar to that of the untreated.

Sample	Recovery Angle ( $^{\circ}\theta$ )	Yellowness Index
untreated	172	41.9
CA*	227	44.5
CA, x 1	210	44.4
CA, x 3	195	44.2
CA, x 5	175	44.0

\*all samples were treated with 100g/l citric acid, 40g/l formic acid at 150°C for 30 seconds x: number of washing cycles

Table 9. Effect of laundering on the recovery angle and yellowness index of citric acid modified soybean fabrics

#### 5.13 Fourier Transform Infrared (FT-IR) analysis of soybean fabric

FT-IR is a complementary technique able to probe the vibrational behaviour of molecules. FT-IR spectroscopy using Attenuated Total Reflectance (ATR) mode is now an established technique for the surface analysis of protein fibres (Sibilia, 1975). For example, in wool, the intermediate oxidation products of cystine can be quantitatively determined when FT-IR (ATR) technique is coupled with second derivative spectroscopic analysis (Schumacher-Hamedat, Fohles and Zahn, 1985; Bridge, Fell and Wardman, 1987; Carr and Lewis, 1993). With regard to crease resistance, FT-IR technique has been successfully used to study the ester crosslinking mechanism of cotton with polycarboxylic acids (Yang, 1991; Yang and Wang, 1996a; Yang and Wang, 1996b). Vynias (2006), used FT-IR to investigate qualitatively and quantitatively the extent and nature of the modification of soybean with citric acid and to examine the conformational changes occurring in soybean.

#### 5.14 FT-IR analysis of untreated soybean fabric

The FT-IR (ATR) absorbance spectrum of untreated soybean is shown in Figue 20, and the main band assignments are listed in Table 10.

Frequency (cm <sup>-1</sup> )	Assignment			
3267	N-H (s) secondary amides			
	O-H (s) hydrogen bonded (broad)			
2937	CH <sub>2</sub> (as)			
2905	CH <sub>3</sub> (ss)			
2855	$CH_2$ (ss)			
1636	Amide I C=O (s), C-N (s), C-C-N (d)			
1531	Amide II N-H (ib), C-N (s), C-C (s)			
1434	$CH_2$ and $CH_3$ , C-H (d)			
1407	$CH_3$ C-H (d)			
1236	Amide III N-H (ib), C-N (s)			

Table 10. Infrared Band Assignments for Soybean Fabric (Harrick, 1967; Hesse, Meier and Zeeh, 1997)



Fig. 20. FT-IR (ATR) Spectrum of Untreated Soybean Fabric

#### 5.15 FT-IR analysis of citric acid treated soybean fabric

Surface analysis of soybean to a depth of 2.0-2.5 µm in the ester/carboxyl region between 1740 and 1710 cm<sup>-1</sup> can be achieved with the ATR technique. Taking into consideration the "surface" modification of the citric acid treatment the ATR technique would certainly be useful in probing any changes on the surface due to the reagent. Using the second derivative, the most prominent change after citric acid modification was a well-defined peak appearing at 1720 cm<sup>-1</sup> in contrast to that of untreated soybean, Figure 21. This is attributed



Fig. 21. Second Derivative FT-IR Absorbance Spectra of Citric Acid Treated Soybean

to the carbonyls of the carboxylic acid and the ester overlapping at 1720 cm<sup>-1</sup> (Hesse et al., 1997). Yang (1991), made the same observation when he characterised ester crosslinkages in cotton with FT-IR Photoacoustic Spectroscopy. The ester/carbonyl signal at 1720 cm<sup>-1</sup> increased with increasing citric acid concentration.

#### 5.16 FT-IR analysis of washed citric acid treated soybean fabric

The wash durability of citric acid modification on the soybean fabric was studied with the FT-IR (ATR) technique. After the first washing cycle, a decrease in the carboxyl carbonyl band was observed (0.128 to 0.120) indicating the relatively poor stability of the finish under perborate-based washing conditions. Further washing to three cycles reduced the carboxyl carbonyl signal to 0.095, Figure 22. However, the signal obtained was still higher compared to the untreated. Nevertheless, after five cycles, the surface carboxyl groups attributed to citric acid were significantly reduced to a value similar to the untreated. Therefore, it can be suggested that, for the citric acid modified soybean fabrics wash durability can be imparted up to three washing cycles. This suggestion correlates well with the crease resistance performance of washed fabrics evaluated by the crease recovery angle presented previously.



a: untreated; b: 20g/l citric acid; c: 50 g/l citric acid; d: 100 g/l citric acid; e: 150 g/l citric acid

Fig. 22. Effect of Laundering on the Second Derivative Band Intensity at Ester/Carbonyl Signal (1070cm<sup>-1</sup>) of Citric Acid Treated Soybean Fabrics

#### 6. Future work

The performance properties of the soybean fibre still need to be improved in order to achieve wider commercial usage. As the fibre is based on protein and PVA there are

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488

opportunities for fibre modification to improve the performance, however there are associated limitations due to the chemical reactivity and sensitivity of the soybean proteins and protein aggregates.

An area of future research would therefore be to further characterize the distribution of the components within the bulk of the fibre and at the surface, and their chemical accessibility. Though this detailed information selective treatments could be developed to improve basic performance and maintain properties during wet and dry processing. Directly related to this improvement could be a better understanding of the nature of the fibre fracturing process during bleaching and its associated impact on handle and wear. In addition, it would be important to examine the flat abrasion behaviour and the fibre fibrillation performance.

In future studies the effect of softeners, elastomeric silicones and alternative polymeric crosslinkers should be examined with a view to improving crease resistance, while still maintaining fabric strength and handle. Both sulphamic acid/urea and Pyrovatex CP treatments imparted improved flame retardancy to the soybean fabric. However, the processing still needs to be improved, particularly in terms of fabric handle and wash durability. The role of alternative fixing agents, which could improve fastness as well as flame retardancy should be further examined. Similarly the range of flame retardant agents should be extended, including non-durable finishes. Underpinning all of these developments, a further investigation of the decomposition mechanism of the treated soybean could be carried out in order to potentially engineer more durable and cost-effective flame retardant finishes.

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494



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Soybean is an agricultural crop of tremendous economic importance. Soybean and food items derived from it form dietary components of numerous people, especially those living in the Orient. The health benefits of soybean have attracted the attention of nutritionists as well as common people.

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