

AN INVESTIGATION INTO THE PROPERTIES OF COTTON FIBRES AS USED
IN NONWOVEN FABRICS

Thesis

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ABSTRACT

The purpose of this investigation was to determine the properties that characterise cotton fibres in the various stages in the CPNF process as well as to determine which fibre characteristics are required to entangle the fibres to produce a successful CPNF. The criteria adopted in this work for a successful CPNF include:

1. the tensile strength of the fabrics
2. a well-defined pattern
3. absorbency and wicking

The properties selected for investigation were

1. the cotton fibre surface (using SEM and DSC analyses)
2. the degree of degradation of the cotton fibre as a result of the CPNF process (using cellulose fluidity measurements)
3. the non-cellulosic content of the fibre (using IR, DSC, and Chemical analyses)
4. fibre friction
5. absorbency and wicking
6. tensile properties

Analysis of the results indicates clearly what the fundamental properties of the cotton fibre must be for a successful cotton CPNF to be manufactured. The conclusions also indicate the necessary properties a man-made fibre must have to produce a successful CPNF.

CHAPTER 1 - INTRODUCTION

Figure 1.1 - a coherent patterned nonwoven fabric



Nonwoven cotton fabrics have been made using a series of various pressure water jets which impinge on fibre webs from above (1). The process entangles the web and produces a pattern in the fabric. The fibre used is greige (unbleached) cotton, and the fabric is bleached and dried. The final product is a coherent patterned nonwoven fabric (CPNF) and is the object of this research. Figure 1.1 shows a typical CPNF and the fabric itself can be seen in Appendix 1.

Attempts to apply the process used to make this particular CPNF to synthetic fibres such as polyester (commonly used with a resin binder in nonwoven fabrics) and a bicomponent polyolefin fibre (usually thermal bonded) (2), have not been successful.

The properties of the cotton that give the CPNFs their unique characteristics are not fully understood. The study of these properties forms the basis of this thesis.

1.1 Definition of a nonwoven fabric

When nonwoven technology was still in its early stages (1962), the ASTM (American Society for Testing and Materials) definition of a nonwoven fabric was:

"Nonwoven fabrics are textile fabrics made of carded web or fibre web held together by adhesives" (3).

This definition recognises only one of the methods of producing nonwoven fabrics. As the technology expanded and new methods of producing these nonconventional textiles were invented, the associated terminology also grew (3). A later definition (1971) is that of Krcma's (3) :

"textile fabrics made of a fibrous layer which may be a carded web, a fibre web, a system of random laid or oriented fibres or threads, possibly combined with textile or non-textile materials such as conventional textile fabrics, plastic films, foam layers, metal foils etc. and forming with them a mechanically bound or chemically bonded textile product".

Examples of the mechanical means of bonding are felting, needle-punching and binding threads.

A more acceptable definition which encompasses CPNFs is that given by EDANA (European Disposables and Nonwovens Association) in 1985 (4)

"A nonwoven is a manufactured sheet, web or batt of directionally or randomly orientated fibres, bonded by friction, and/or cohesion and/or adhesion, excluding paper and products which are woven, knitted, tufted, stitch-bonded incorporating binding yarns or filaments, or felted by wet-milling whether additionally needled or not. The fibres may be of natural or man-made origin. They may be staple or continuous filaments or be formed in situ".

The criteria adopted in this work for a successful CPNF include

1. tensile strength of the fabric - the CPNF should be able to withstand reasonable handling during processing and also afterwards as a medical dressing for instance. This property of tensile strength is related to the success of fibre entanglement.
2. well-defined pattern - this is important from the aesthetics point of view and also because it also gives a measure of the success of fibre entanglement.
3. absorbency and wicking - The CPNF should be absorbent.

These criteria are based on anticipated end uses of the CPNF, such as in medical dressings, as well as on criteria used by institutions involved in the development of methods used in the evaluation of nonwoven fabrics (4, 5).

1.2 Scope of the present work

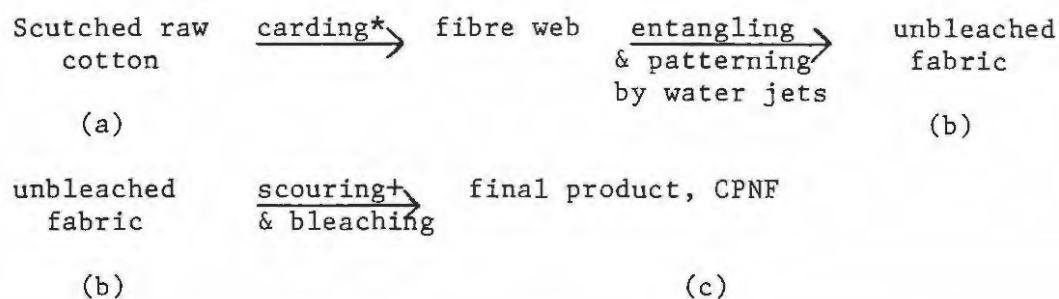
One of the aims of this work is to understand the mechanism that produces the coherent patterned fabric by investigating the effect on the cotton fibres of the processes involved. The three stages of the process give rise to basically three types of samples that can be investigated separately. The three sample types are:

- a. raw scutched* cotton fibres (greige cotton). This material has not been washed (see Appendix 2).
- b. the coherent patterned fabric produced after the water jets impinge upon the fibre. The temperature of the water is room temperature, ranging from 15°C to 23°C (see Appendix 3).
- c. the scoured and bleached fabric. The fabric is scoured and bleached in one stage using sodium hydroxide, sodium carbonate, hydrogen peroxide and a surfactant (see Appendix 1).

* Mechanically opened, cleaned and formed into a continuous lap

The stages in the CPNF process are shown in figure 2.2 below.

Figure 2.2 - Diagram showing the sample stages in the CPNF process



Much work has been done on the evaluation of cotton fibre characteristics and their relation to processing performance as well as fabric properties (6), but no work has been done on the coherent patterned nonwoven fabric. The properties which are of interest in this study are those that appear likely to undergo change during the CPNF process, as well as those that can be used to characterize the nonwoven fabric.

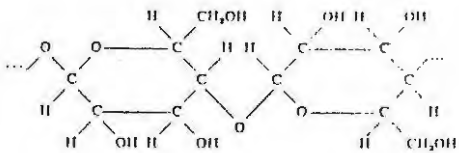
-
- * The disentanglement, cleaning and intermixing of fibres to produce a continuous web or sliver suitable for subsequent processing.

 - + The treatment of textile materials in aqueous or other solutions in order to remove natural fats, waxes, proteins and other constituents, as well as dirt, oil and other impurities.

The fibre properties selected for investigation were:

1. the cotton fibre surface
2. the degree of degradation of the cotton fibre as a result of the CPNF process
3. non-cellulosic content of the fibre, particularly the natural waxes (this includes both inorganic and organic material)
4. fibre friction
5. absorbency and wicking
6. tensile properties

The fibre properties of man-made fibres were investigated in order to get a better understanding of the success of the cotton CPNF (see Appendix 1). The man-made fibres investigated are shown in figure 3.3.

Figure 3.3 - Some man-made fibres investigated in this study		
Trade Name	Fibre Type	Chemical Unit & Formula
GRILENE (see App. 4)	Polyester	Polycondensate of Terephthalic acid and Ethylene glycol (7) (Polyethylene Terephthalate) $\left(-O \text{ CO} \begin{array}{c} \diagup \diagdown \\ \diagdown \diagup \end{array} \text{CO O CH}_2 \text{ CH}_2 - \right) (8)$
CHISSO EA (see App. 5)	Polyolefin, a side-by-side bicomponent fibre (9)	Polypropylene and Polyethylene (9) Polypropylene - $\text{CH}_2\text{-CH-}$ (8) CH_3 Polyethylene $\left(- \text{CH}_2 - \right) (8)$
ENKA BICO (see App. 6)	A mixed bicomponent fibre	Polyethylene and a polyester Polyethylene $\left(-\text{CH}_2- \right) (8)$ Polyethylene Terephthalate $\left(-O \text{ CO} \begin{array}{c} \diagup \diagdown \\ \diagdown \diagup \end{array} \text{CO O CH}_2 \text{ CH}_2 - \right) (8)$
STANDARD VISCOSE RAYON (see App. 7)	Regenerated Cellulose (10,11)	Cellulose (12)  Reproduced from (12)

1.2.1 The cotton fibre surface

After the carding process, there are two stages in the process of producing CPNF. First of all, the water jets impinge on the greige cotton producing the patterned fabric. Secondly, this fabric is scoured, bleached and dried to produce the final product.

The study of fibre surfaces by Scanning Electron Microscopy (SEM) has been found to be useful in the characterization of cotton (13,14,15). A detailed investigation of the cotton fibre surface at the various stages of processing has been conducted in this work using scanning electron microscopy (SEM).

The cotton fibre surface is also an important property that affects the behaviour of the fibre during general processing (6). SEM has been used to study the effect of washing processes on the cotton fibre surface (14). It was felt that similar work on the fibre before the various stages in the processing used on forming CPNF could help in understanding the reason why cotton fibres in CPNF stick together while other material fibres do not.

Differential Scanning Calorimetry (DSC) has been used successfully to distinguish between washed and unwashed cotton fibres (16,17). The washing process removes particulate matter and substances such as waxes from the cotton fibre surface. In so doing, the washing process changes the fibre surface. DSC has been used

to investigate changes in the cotton fibre surface at the various stages of processing. The chemical composition of the water-soluble extract of the cotton fibre at the various stages of the process was determined in conjunction with the DSC analysis.

1.2.2 The degree of degradation of the cotton fibre is a result of the CPNF process

Cotton is a cellulose fibre. The cellulose molecule is made up of a chain of glucose rings and can be represented as $(C_6H_{10}O_5)_n$, where n is typically in the order of 10^4 (12). The molecular chain is typically $5 \times 10^{-6} m$ in length (12).

The fluidity of a cellulose solution can be related to the degree of polymerization of cotton (18). This chemical test can thus be used as a means of measuring the extent of degradation of cotton as a result of chemical action, processing or other means. Fluidity is defined as the reciprocal of viscosity (19). The effect of dissolving long-chain molecules in a solvent is to increase the viscosity of the solvent (19). The shortening of the cellulose chain results in a decrease in the viscosity of the cellulose solution. In order to determine whether the CPNF process had any effect on the molecular chain length of cotton, the fluidity of the greige cotton fibres was

compared to the fluidity of the patterned entangled fabric as well as the scoured and bleached patterned fabric. A number of methods exist for the determination of cellulose fluidity. The method chosen to determine cellulose fluidity in this investigation is based on the dispersion of cellulose in cuprammonium hydroxide (19).

1.2.3 Non-cellulosic content of the cotton fibre

In its natural state, the cotton fibre has an outer layer of fats, pectin and waxes, which act as a surface lubricant (6,20). Studies have shown that cotton wax is not totally removed by normal scouring and bleaching processes (6,20). Thus, even in the final fabric, the non-cellulosic content is still a property that is worth considering, because it affects the wetting and absorbency properties of the fabric (6). The chemical treatment of the entangled web produced by the CPNF process has a combined scouring and bleaching effect on the cotton fibre. Taking this into account, the chemical extracts of the cotton fibre taken at various stages in the CPNF process were analysed. The extraction process involved washing the cotton fibre with organic solvents. Various organic solvents such as benzene, carbon tetrachloride, and dichloromethane have been used by other workers to remove oils and waxes from cotton fibre (20,21). In this work dichloromethane was used because it is not as toxic as benzene and carbon tetrachloride, (22).

1.2.4 The cotton fibre friction

Fibre friction can be considered from two different standpoints. There is fibre to fibre friction as well as fibre to metal friction (22). Both types of friction have been found to play an important role in the traditional textile processes such as carding and spinning (6,23). In the production of nonwovens, a low fibre to metal friction is desirable in order to card a stable web. At the same time, a high fibre to fibre friction provides a cohesion that facilitates the bonding process (23).

The removal of the natural lubricants from the fibre changes the state of the fibre surface and thus affects its frictional properties (20,24). Since the presence of natural wax on the cotton fibre surface reduces fibre friction, its removal should increase fibre friction. Some methods for the determination of fibre friction have been reported in literature, (25,26,27).

In this study the results of fibre to metal friction are reported. Furthermore, results for fabric to fabric friction are also given in the absence of suitable equipment to measure fibre to fibre friction. Details of the fabric to fabric friction method are given in chapter 6.

1.2.5 Absorbency and wicking

The term absorbency is used in this study to refer to the fabric properties which manifest themselves in the absorption of aqueous fluids by fibrous material (28,29). The complex nature of the sorption process is due to the complex nature of fibrous materials. The properties of the fibrous material are in turn determined by its chemical structure and physical properties. The geometric arrangements of the component fibres as well as their properties of matter of the individual fibres determine the physical properties such as absorbency and wicking of the fibrous material (30).

In this study, the fibrous material has a single component, the cotton fibres that make the patterned fabric. In nonwoven fabrics, the method used to produce the initial fibre web before it is formed into the cohesive nonwoven fabric is important (30). In the case of CPNF, the fabric is made from a carded web so the cotton fibres have a preferred orientation lengthwise along the roll of fabric, which is known as the machine direction. This is in the direction of motion of the moving surfaces which the fibres go through during carding. The wicking results given in Chapter 7 will illustrate the effect of the fibre orientation on the wicking properties.

The absorbency properties of the CPNF were studied as well as those of fabrics which were made from fibres that were specially treated (eg. solvent-extracted fibre, washed fibre). These absorbency results give a measure of the degree of bonding or interlocking of the component fibres (28,29).

In this work absorbency capacity measurements have been used to determine the amount of liquid absorbed from dryness. The wicking measurements have been used to determine the speed with which a given liquid is absorbed and distributed in the absorbent material.

1.2.6 Tensile Properties

Tensile properties, in this case the tensile strength and the tensile energy, are important physical properties of fabrics because they contribute to both the behaviour of the fibres during processing as well as to the properties of the final fabric or yarn (31).

These tensile properties are determined mechanically, by continuously measuring the force developed as the sample is elongated at a constant rate of extension. The tensile strength is the force at breaking point of the fabric sample (kg or Newton) per given width of the sample (31).

The tensile energy is the work done in extending the sample to breaking point and is determined from the area under the force-elongation curve (31).

The tensile strength is a measure of the degree of cohesion achieved by the method of manufacture of the nonwoven fabric (30). As with other physical properties of nonwoven fabrics, it is also dependent to a certain extent on the strength of the individual fibre components.

Natural cellulosic fibres such as cotton are strong and modestly extensible. The strength of cotton fibres is either unaffected by moisture or it may increase with increasing moisture (30). In this study, the tensile strength tests were performed on dry fabrics that had been conditioned at standard temperature and humidity, and also on wet fabrics.

In this study the tensile properties have been used to determine the degree of cohesion achieved by the patterning process in the cotton CPNF, as well to determine the degree of cohesion in CPNF made with other fibres.

1.2.7 Objectives

The objectives of this research project may be summarized thus:

1. To determine the properties that characterise cotton fibres in the various stages in the CPNF process.
2. To determine which fibre characteristics are required to entangle the fibres to produce a successful CPNF.

CHAPTER 2 - A SCANNING ELECTRON MICROSCOPE STUDY OF
COTTON AND OTHER FIBRES

A Scanning Electron Microscope (SEM) study of the cotton fibre was initially conducted with the aim of investigating the cotton fibre surface. The nature of the SEM lends itself well to the study of surface morphology because the image formed by scanning the object is an exact representation of its surface (32). Very high magnification can be achieved. The highest used in this study was 5000X.

The method used was a direct mounting technique which has been successfully used by many other workers (14,15,33). The more laborious and slow method of surface replication was not used (13).

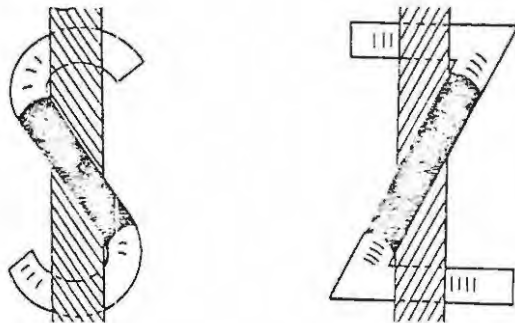
2.1 THE FIBRES

2.1.1 The Cotton Fibre

On drying, (see section 4.1.1) the cotton fibre collapses to form a flat, ribbonlike structure with spiral twists known as convolutions along its length (32, 34). These convolutions vary from a few to many and according to the literature have the following characteristics (32):

1. the direction of the spiral can be in the left-hand or in the right-hand direction. This can be readily seen in figure 2.10.

2. the direction can be reversed frequently along the length of the fibre and the number of reversals depend mainly upon the fibre wall thickness. The change of direction of twist along the fibre can be correlated with the internal structure of the fibre.
3. as opposed to the wrinkles and folds that are caused by the collapse during drying, the true convolutions have 180° twists which are either in an S twist direction or a Z twist form direction with respect to the fibre axis.



5. the number of convolutions varies from about 50 to 100 per inch of fibre length and is determined by such factors as cotton variety as well as conditions of growth and drying.

The convolution characteristics discussed above can be observed at fairly low magnifications (see figure 2.1).

In this study they were observable in both the fibre and the fabric micrographs taken at magnifications of 100X and below (see figures 2.1, 2.2, 2.5, 2.6, 2.7, & 2.8).

Electron microscopy is a well established technique in the study of fine details of cotton fibre surfaces (13,32,34). Electron micrographs of surface replicas of the dried mature cotton have shown that its outside wall, known as the primary wall has a "wrinkled, fluted or corrugated aspect" (20).

Most of the non-cellulosic content of the fibre is to be found in the primary wall (20,32). When these surface coating materials are removed by purification processes such as scouring or solvent extraction, the fibrillar structure of the primary wall can be seen (14). Electron microscopy has shown that the cellulosic portion is a network of microfibrils that are interlaced (32). In this work the primary walls of the cotton fibre were never exposed because the scouring and solvent-extraction processes were reasonably mild.

2.1.2 The stages in the CPNF process

In the previous chapter, it has been stated that there are three stages in the CPNF process giving rise to three sample types (figure 1.2). For the purpose of this work, the SEM study was confined to the initial stage of the greige (raw) cotton fibres and the final stage of the bleached and scoured fabric.

There was no difference expected to be found between the greige (raw) cotton fibres and the fibres in the patterned fabric of the second stage. The only difference between the two stages ((a) & (b) of figure 1.2) is that the fibres in the patterned fabric have been in brief contact with the cold water of the water jets. This process is not vigorous enough to effect changes in the cotton fibre surface.

The final stage in the production process of cotton CPNF involves the combined purification process of scouring and bleaching to remove non-cellulosic materials. The chemicals used in this process are sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), hydrogen peroxide (H_2O_2) and a detergent. The combined action of the sodium salts is a scouring one.

It is reported that concentrations of sodium hydroxide as low as 1% will remove pectins completely in kier boiling (34). Cotton waxes are also removed through kier boiling (34). Hydrogen peroxide is the bleaching agent.

2.1.3 Experimental variations of the cotton fibre

Experimental variations of the cotton fibre were prepared by various procedures (details in chapter 3). Following types of cotton fibres were examined

1. washed cotton fibres
2. solvent - extracted (dichloromethane) cotton fibres

3. washed and solvent-extracted cotton fibres
4. Absorbent cotton wool BPC* (see Appendix 8)

The washing and solvent-extraction processes used removed non-cellulosic materials from the fibre surface.(14,34). The cotton wool was used as a control because it represents cotton fibre that has been bleached and scoured to its purest form.

Electron microscopic examination of the abovementioned fibre variants was performed in order to examine them for changes as compared to the raw cotton fibres.

2.2 OTHER FIBRES

In the course of this study, attempts were made to apply the CPNF manufacturing process to various synthetic fibres (see figure 1.3) that are commonly used in the production of conventional nonwoven fabrics using adhesive bonding and thermal bonding.

* Absorbent Cotton Wool British Pharmaceutical Codex is prepared from cotton, which consists of the epidermal trichomes of *Gossypium herbaceum* L. and other cultivated species of *Gossypium* (Fam. Malvaceae). The seeds are removed mechanically and the trichomes freed from fatty matter by treatment with alkali, bleached, washed, and mechanically loosened and separated to form a fleecy mass of soft white filaments which consist almost entirely of cellulose.

The experiments were conducted on a pilot laboratory line (Details are given in chapter 8). The fibres investigated were:

1. Grilene fibre
2. Chisso EA fibre
3. Enka Bico fibre
4. Standard viscose rayon fibre

The details of the fibres are given in figure 1.3 in Chapter 1.

Electron microscopic examination of the four types synthetic fibres was conducted to investigate their morphology, and findings were related to the pilot laboratory production of CPNF.

2.3 EXPERIMENTAL DETAILS

The bulk of the micrographs were taken by Mrs Wendy Baier on a Hitachi S-430 scanning electron microscope. Instrument operating voltages are recorded on the individual micrographs. The samples were mounted horizontally on specimen stubs and coated with gold. Some of the micrographs were taken by Mr Robin Cross of the Electron Microscope Unit at Rhodes University.

2.4 RESULTS

Figure 2.1 - Greige (raw) cotton (magnification: 80x)

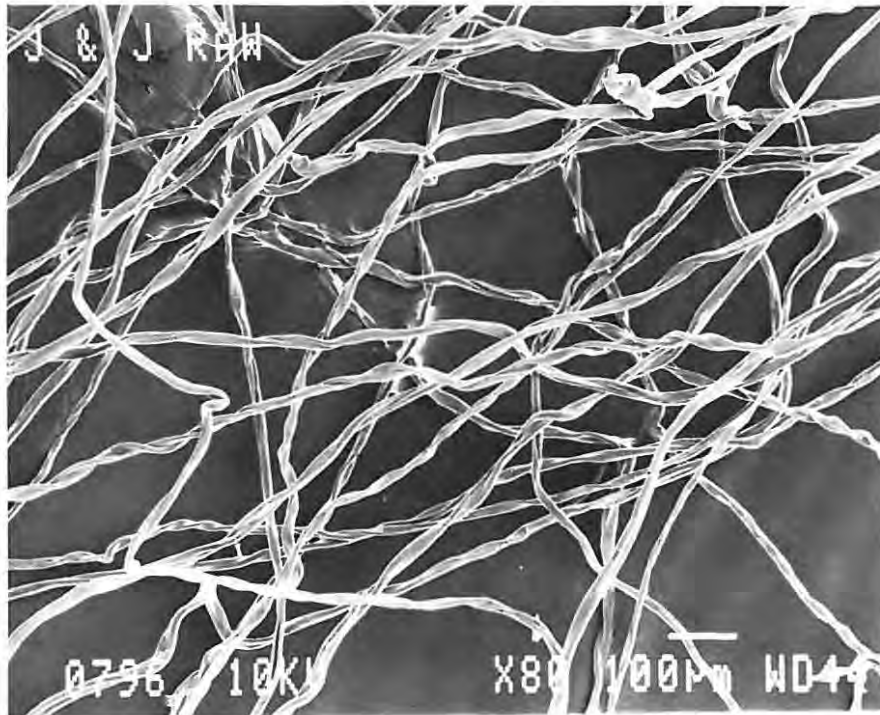


Figure 2.2 Scoured and bleached cotton CPNF (factory produced)
(magnification 80x)

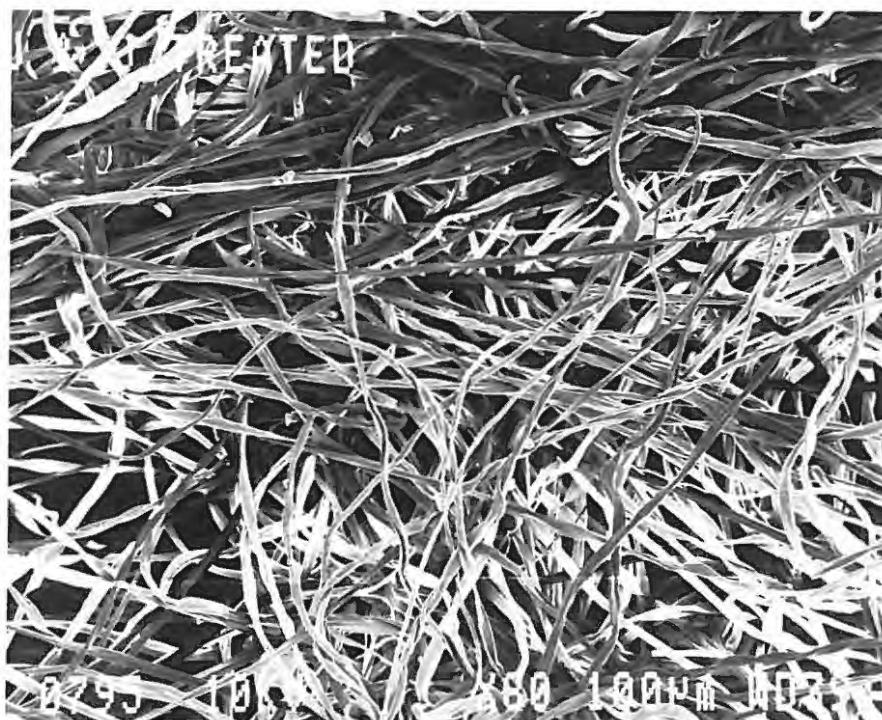


Figure 2.3 - Greige (raw) cotton (magnification 3000x)



Figure 2.4 - Scoured and bleached cotton CPNF (factory produced magnification 3000x)



Figure 2.5 - Greige (raw) cotton (magnification 100x)

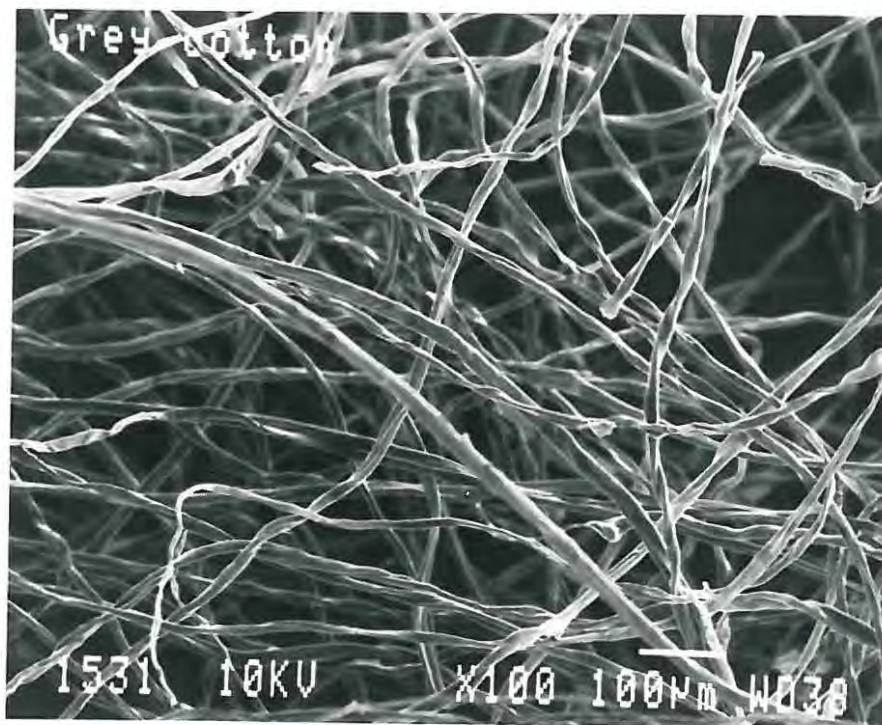


Figure 2.6 - Washed cotton (magnification 100x)

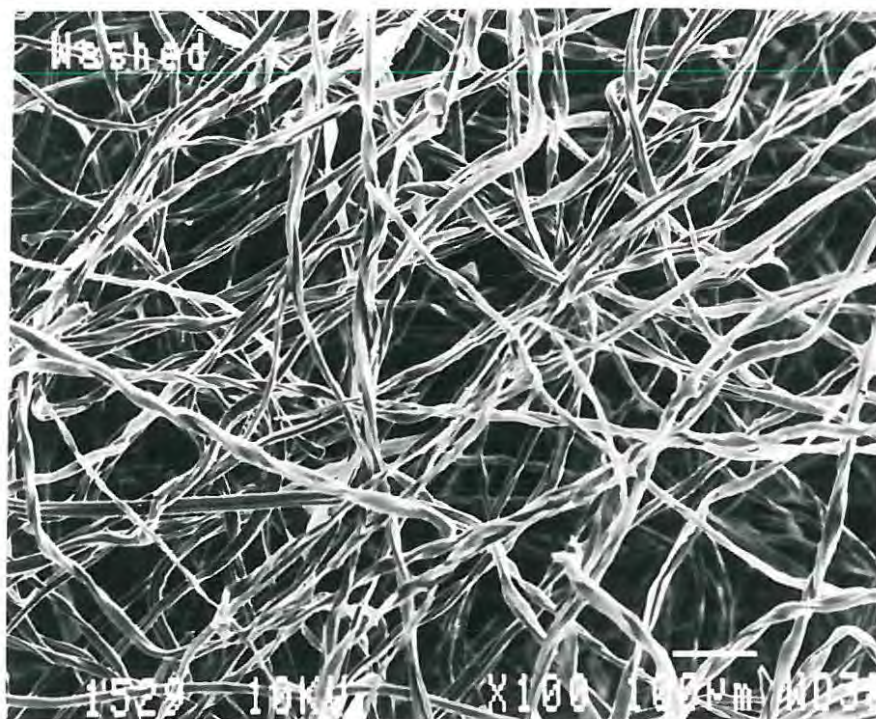


Figure 2.7 - Solvent-extracted cotton (magnification 100x)

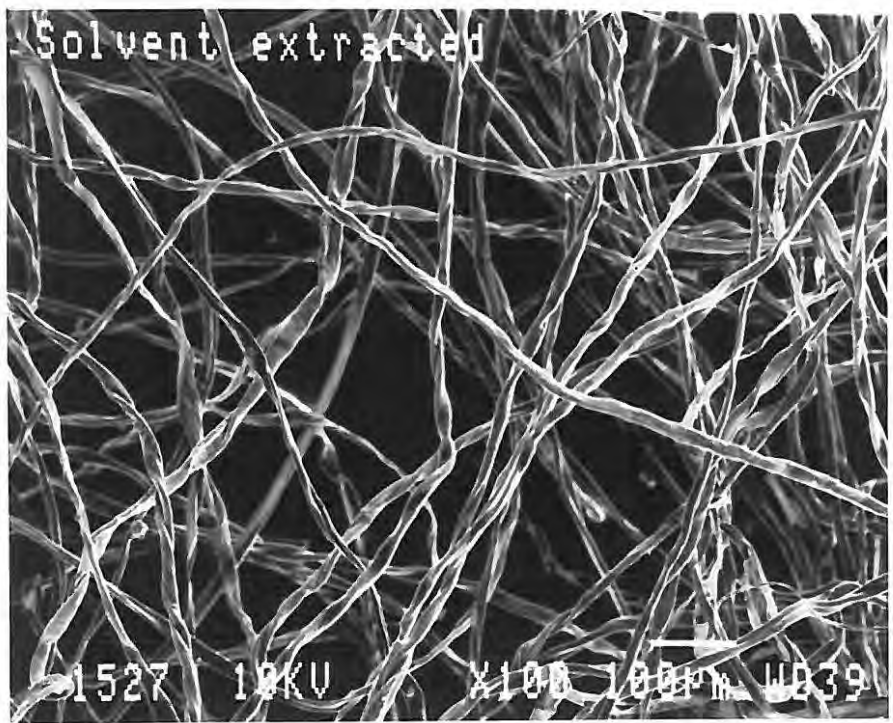


Figure 2.8 - Washed & solvent-extracted cotton (magnification 100x)

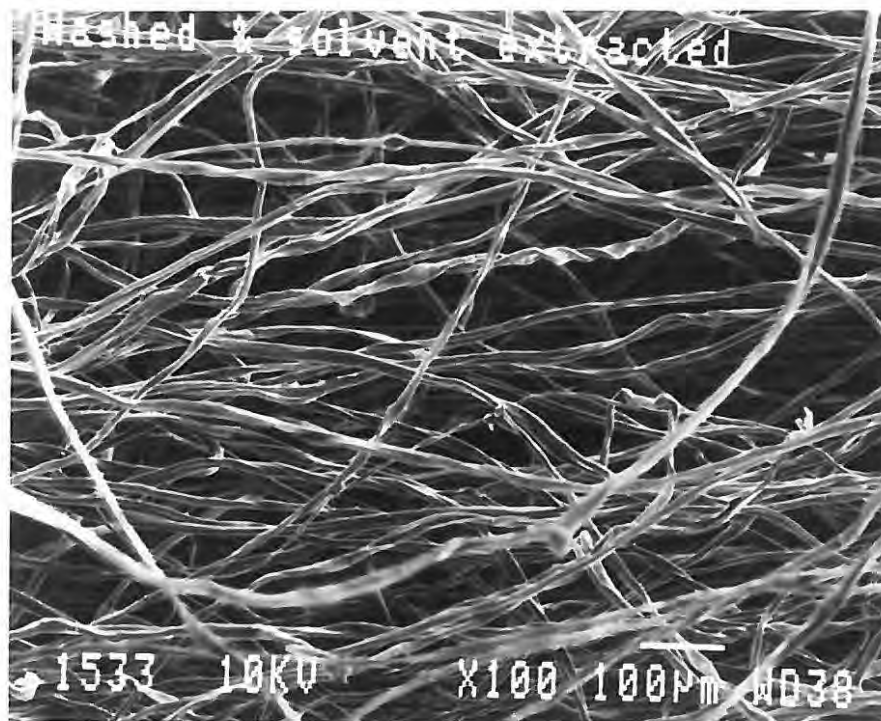


Figure 2.9 - Greige (raw) cotton cotton (magnification 1000x)

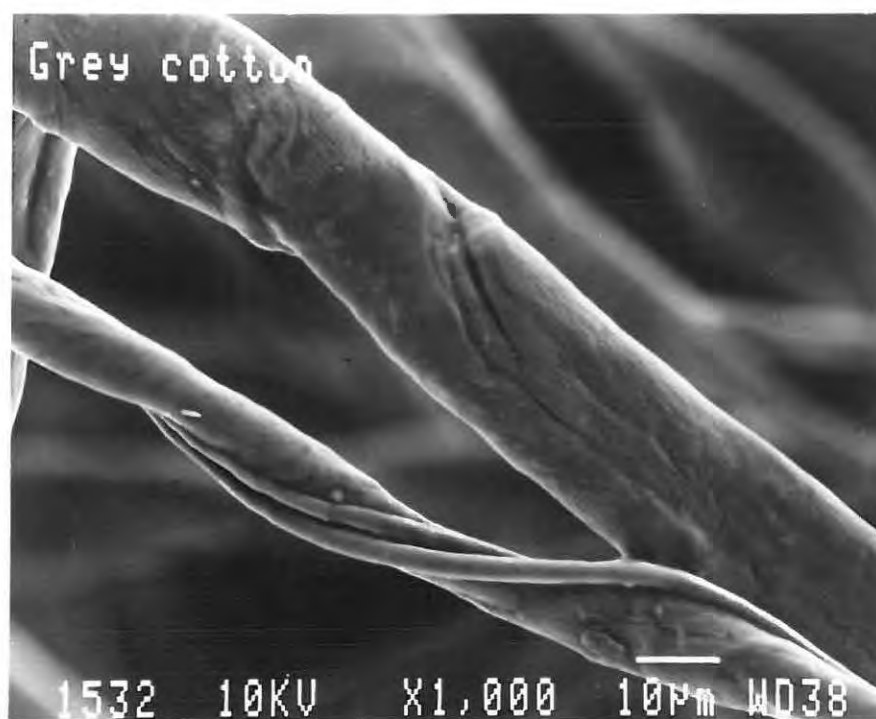


Figure 2.10 - Washed cotton (magnification 1000x)

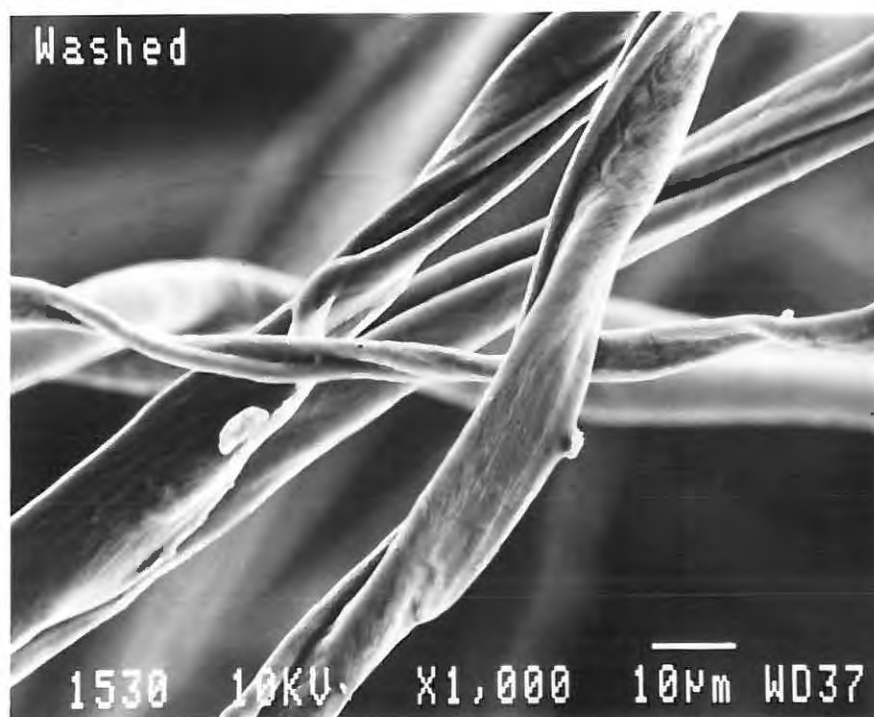


Figure 2.11 - Solvent-extracted cotton (magnification 1000x)

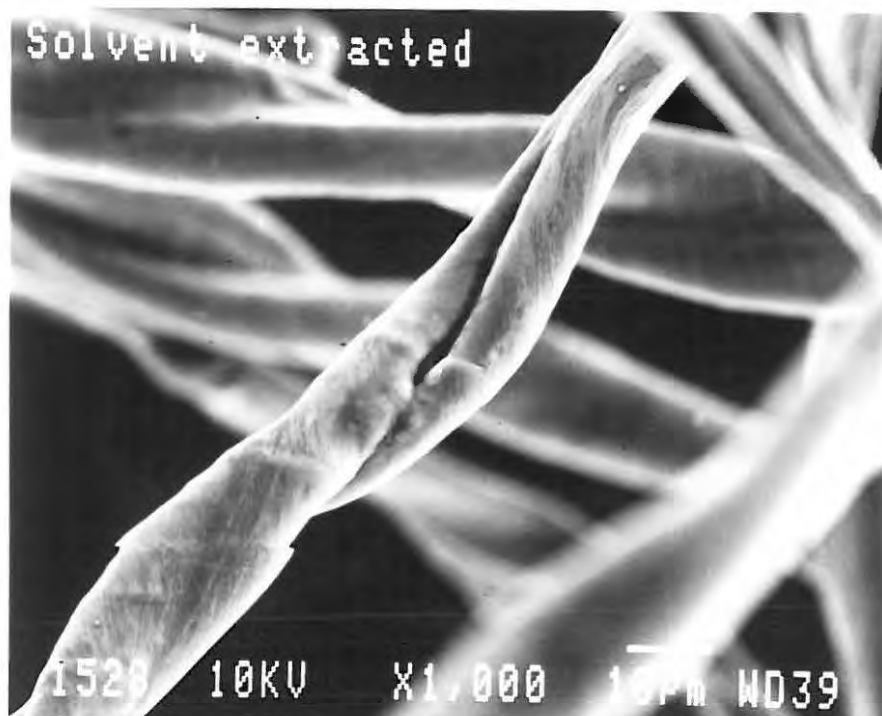


Figure 2.12 - Washed & solvent-extracted (magnification 1000x)



Figure 2.13 - Chisso EA CPNF (magnification 36x)

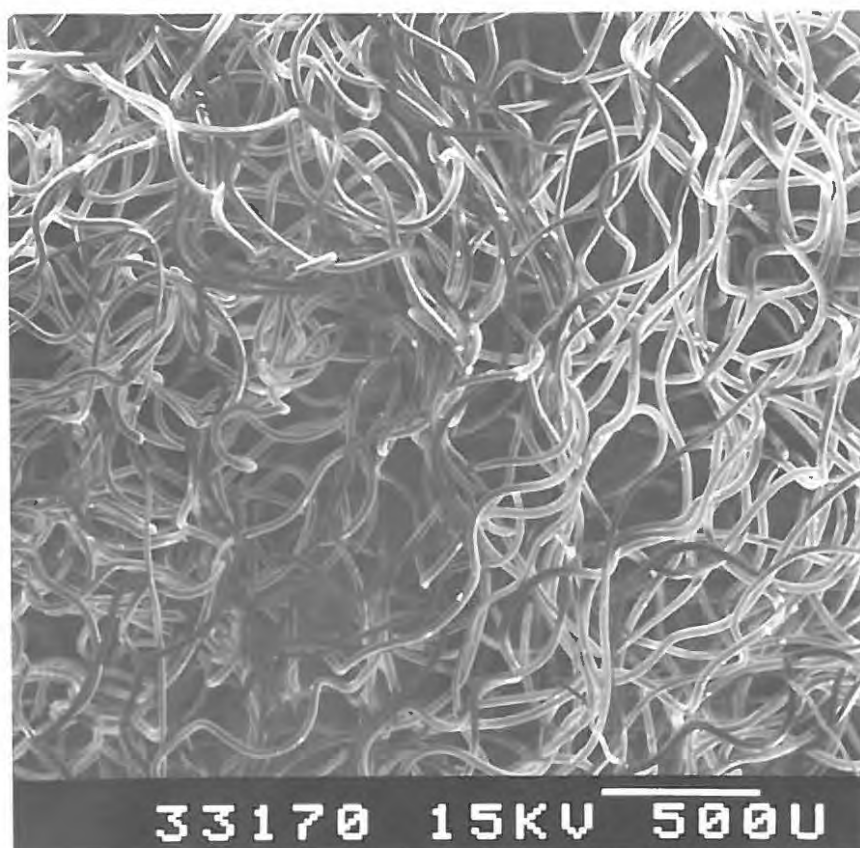


Figure 2.14 - Enka Bico CPNF (magnification 35x)

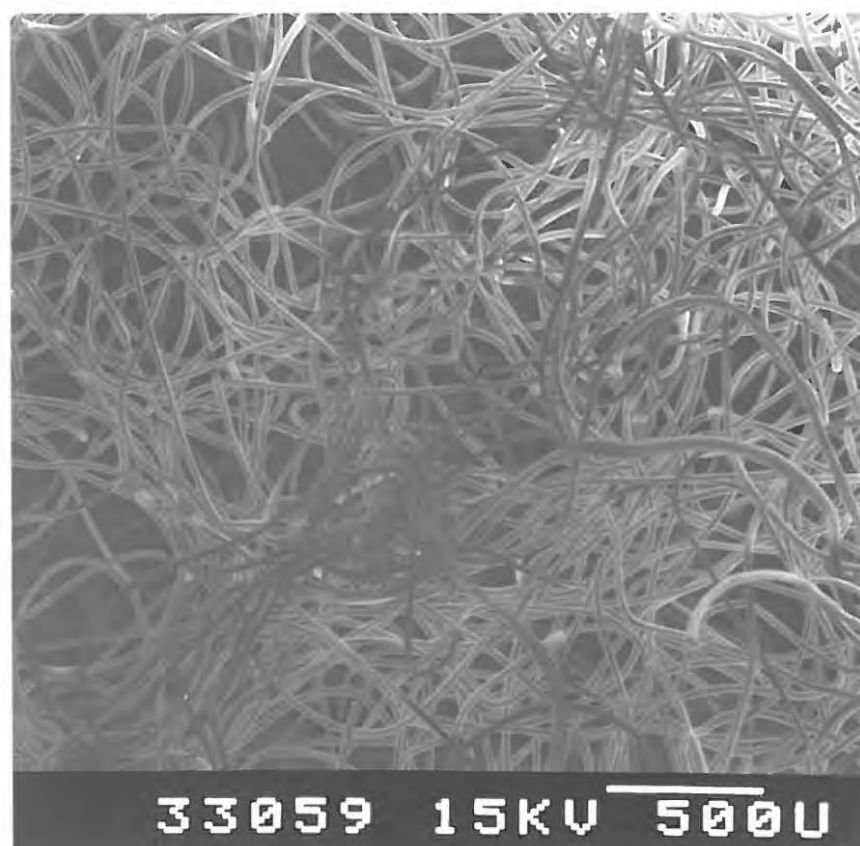


Figure 2.15 - Absorbent cotton wool CPNF (magnification 36x)

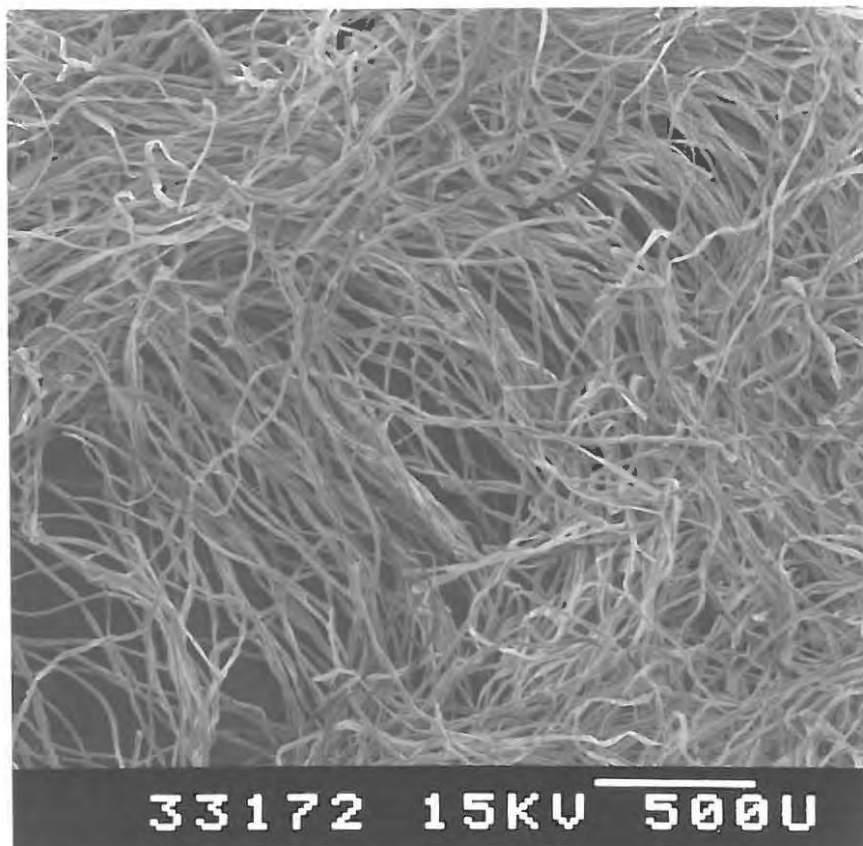


Figure 2.16 - Greige (raw) cotton CPNF (magnification 35x)

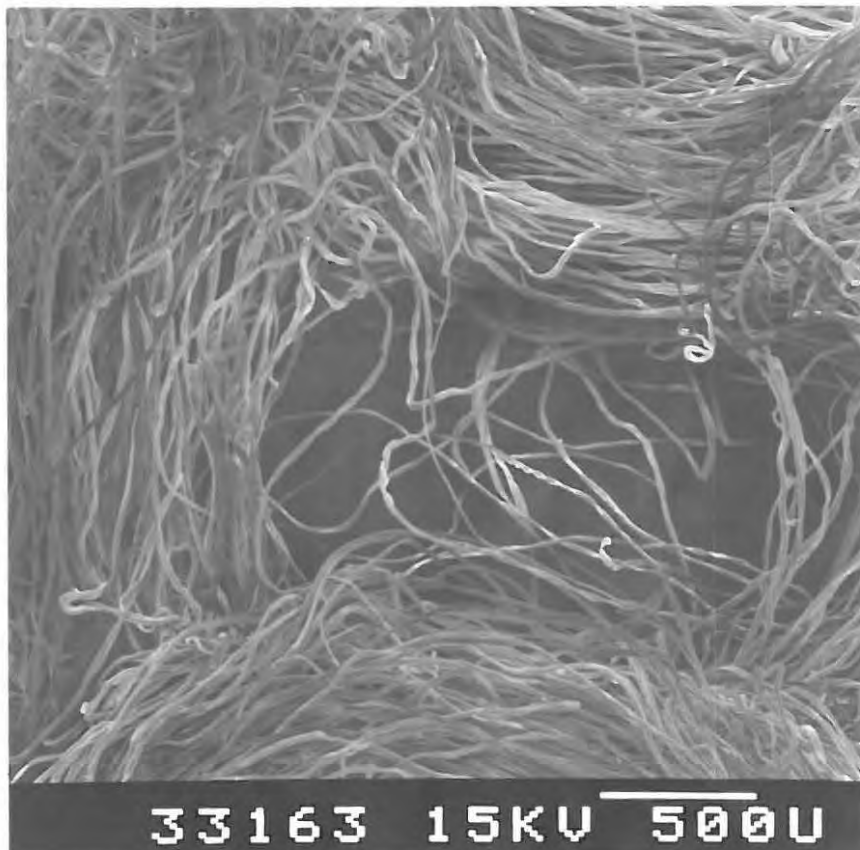


Figure 2.17 - Viscose rayon (10 minutes) CPNF (magnification 35x)

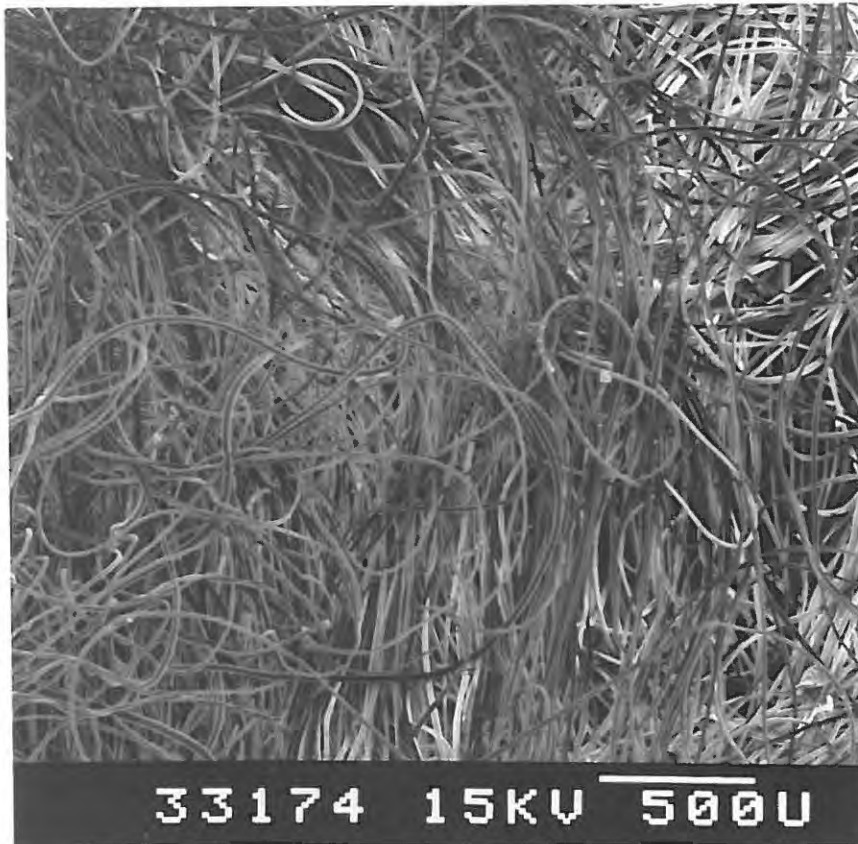


Figure 2.18 - Viscose rayon (20 minutes) CPNF (magnification 35x)



Figure 2.19 - Chisso EA CPMF (magnification 100x)

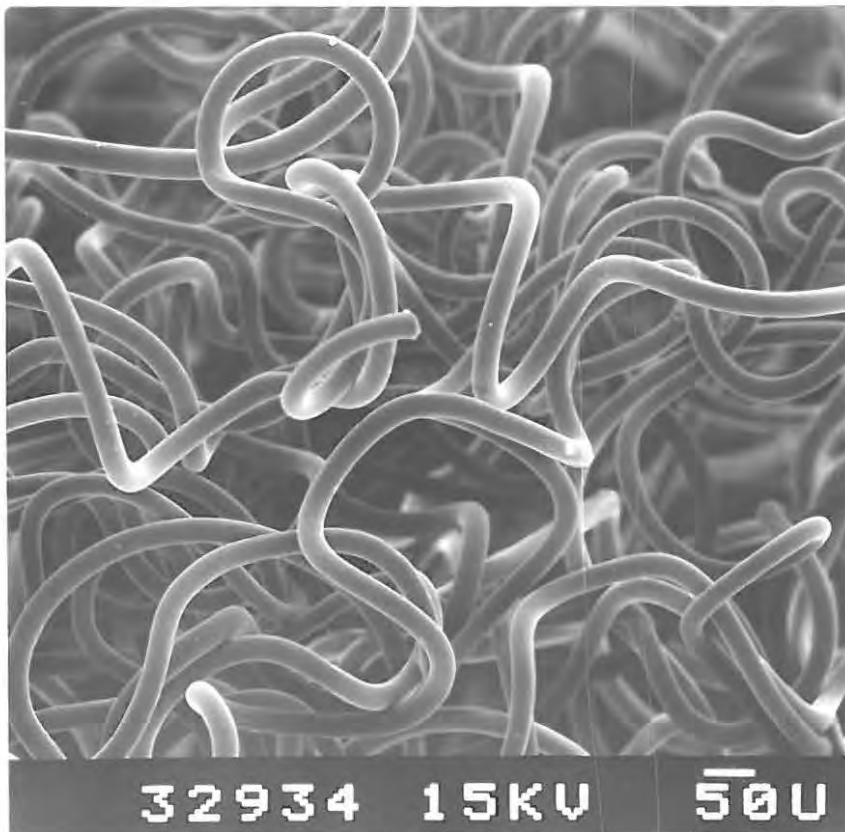


Figure 2.20 - Enka Bico CPMF (magnification 100x)

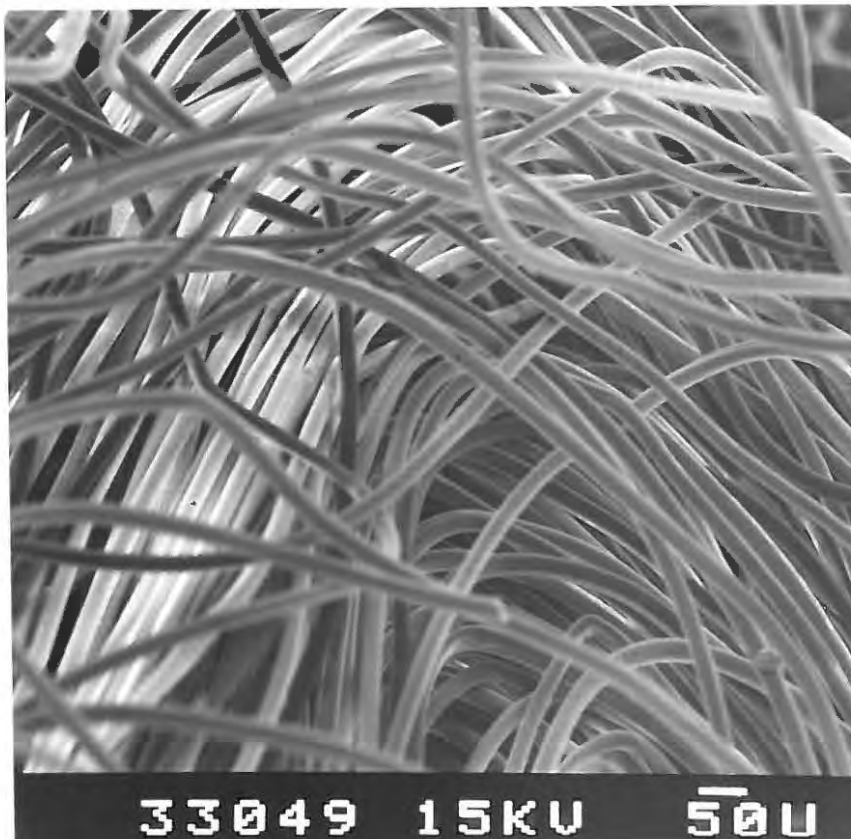


Figure 2.21 - Absorbent cotton wool CPNF (magnification 100x)

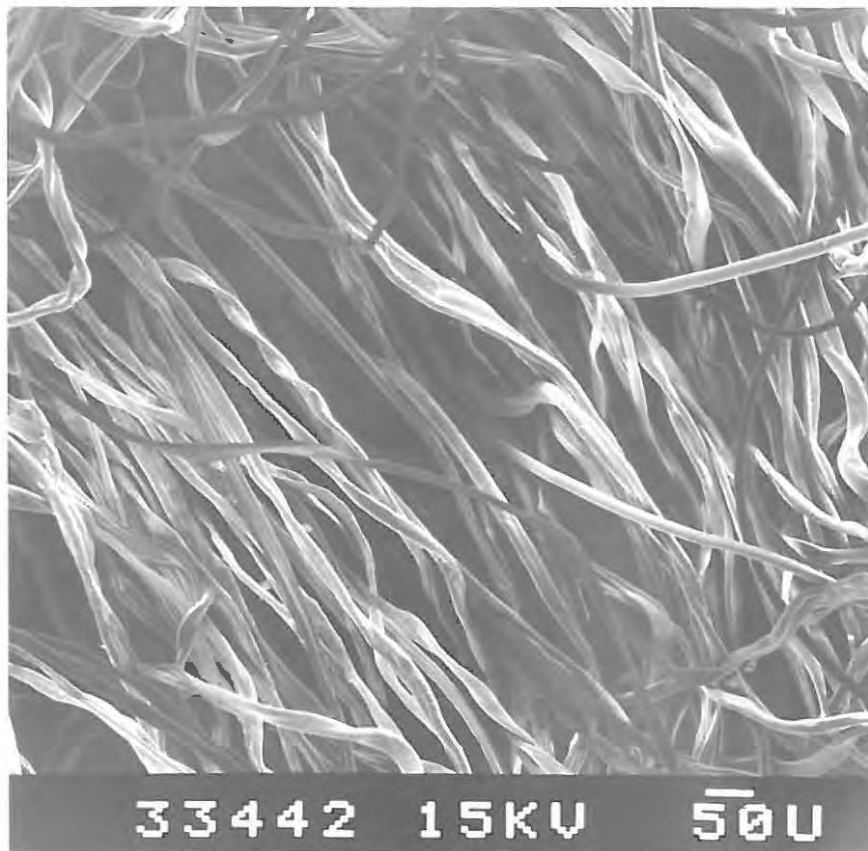


Figure 2.22 - Greige (raw) cotton CPNF (magnification 100x)

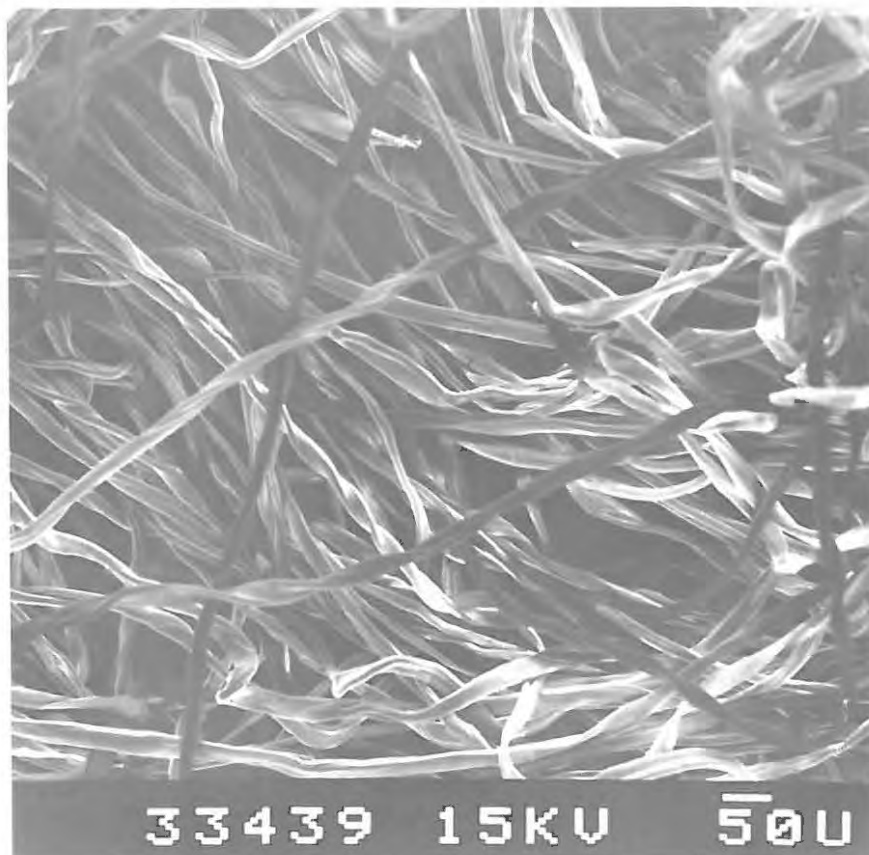


Figure 2.23 - Viscose rayon (10 minutes) CPNF (magnification 100x)

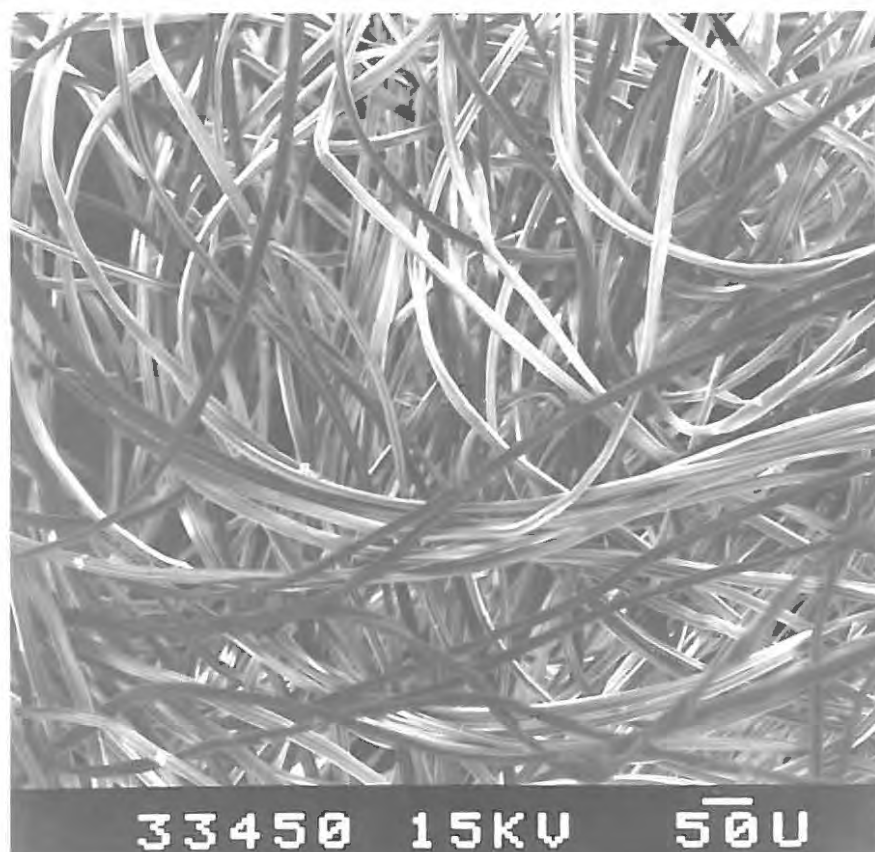


Figure 2.24 - Viscose rayon (20 minutes) CPNF (magnification 100x)

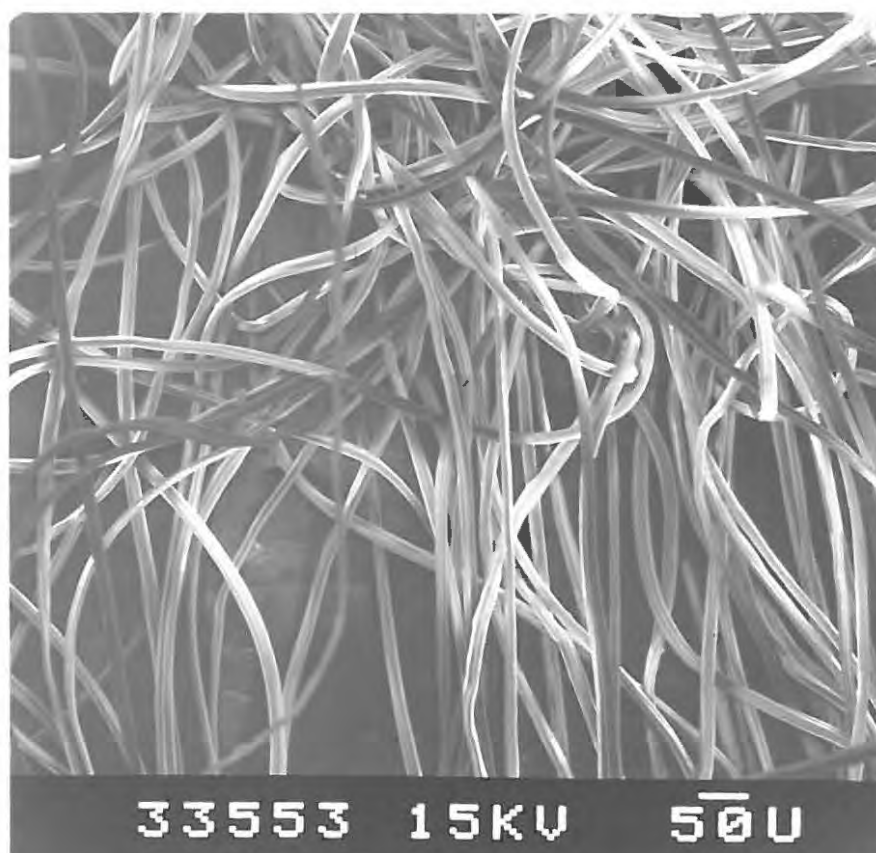


Figure 2.25 - Chisso EA fibre (magnification 1000x)

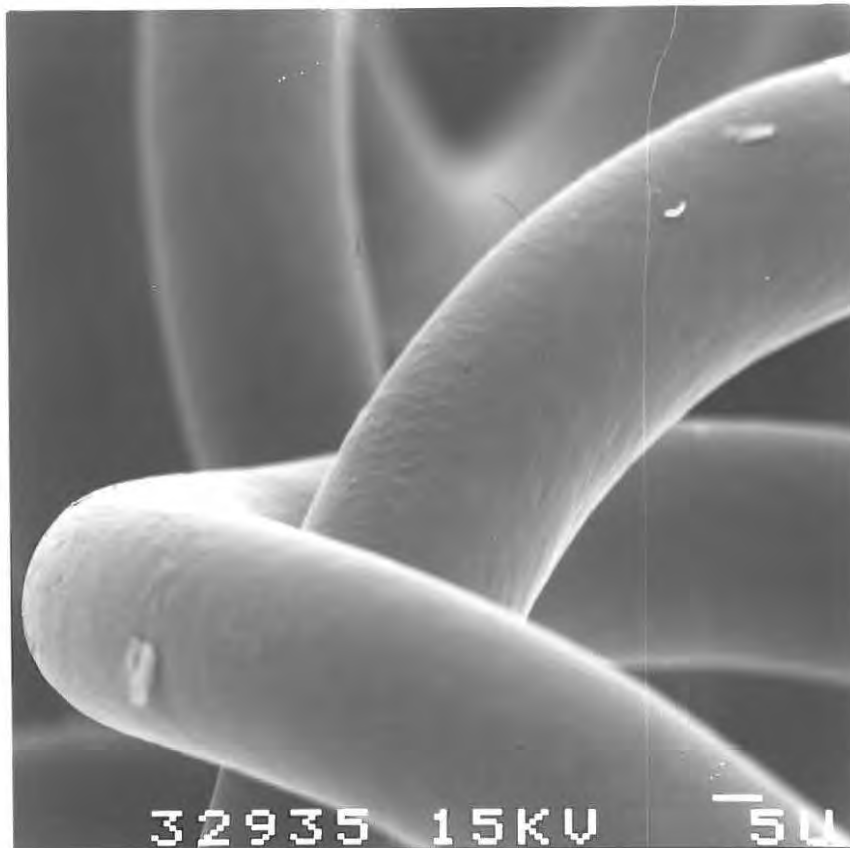


Figure 2.26 - Grilene fibre (magnification 1000x)

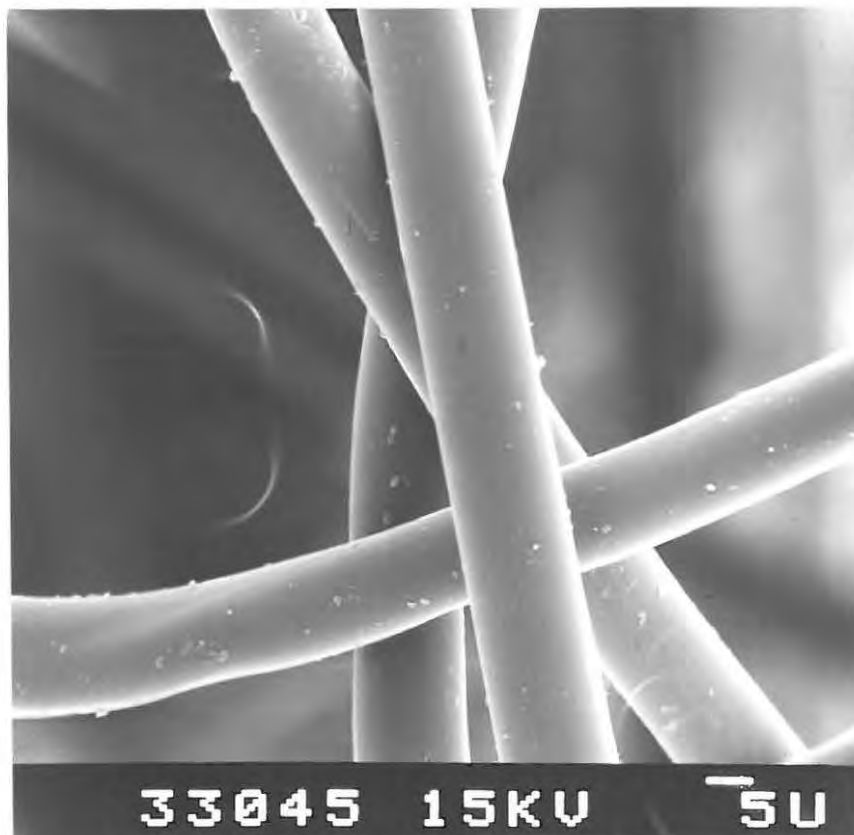


Figure 2.27 - Greige (raw) cotton fibre (magnification 1000x)

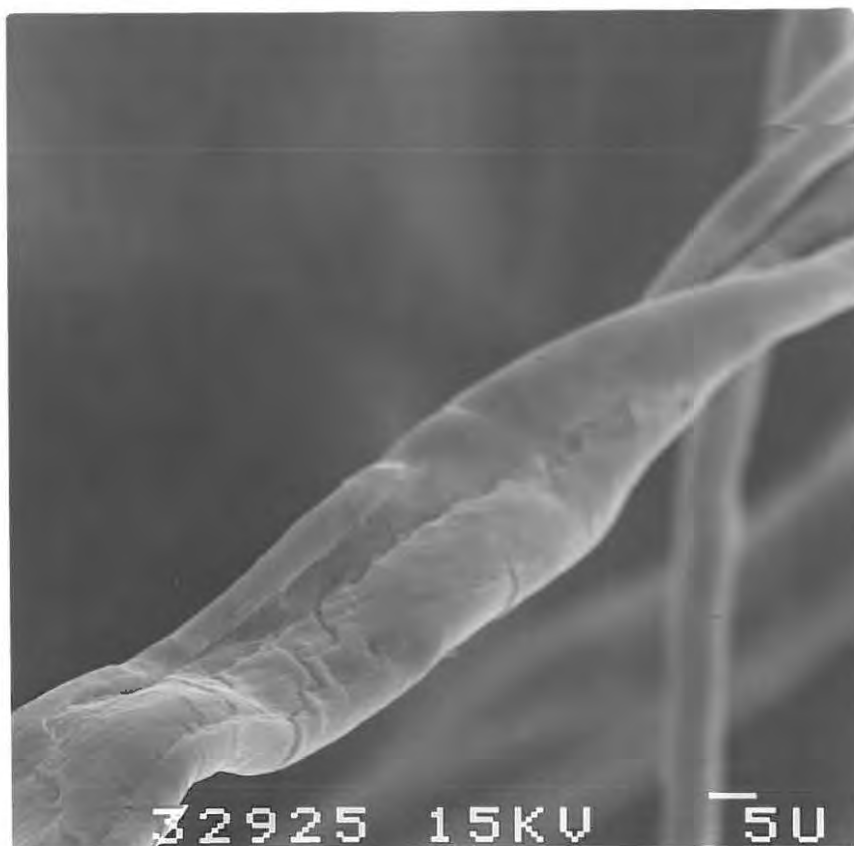


Figure 2.28 - Viscose rayon fibre (magnification 1000x)

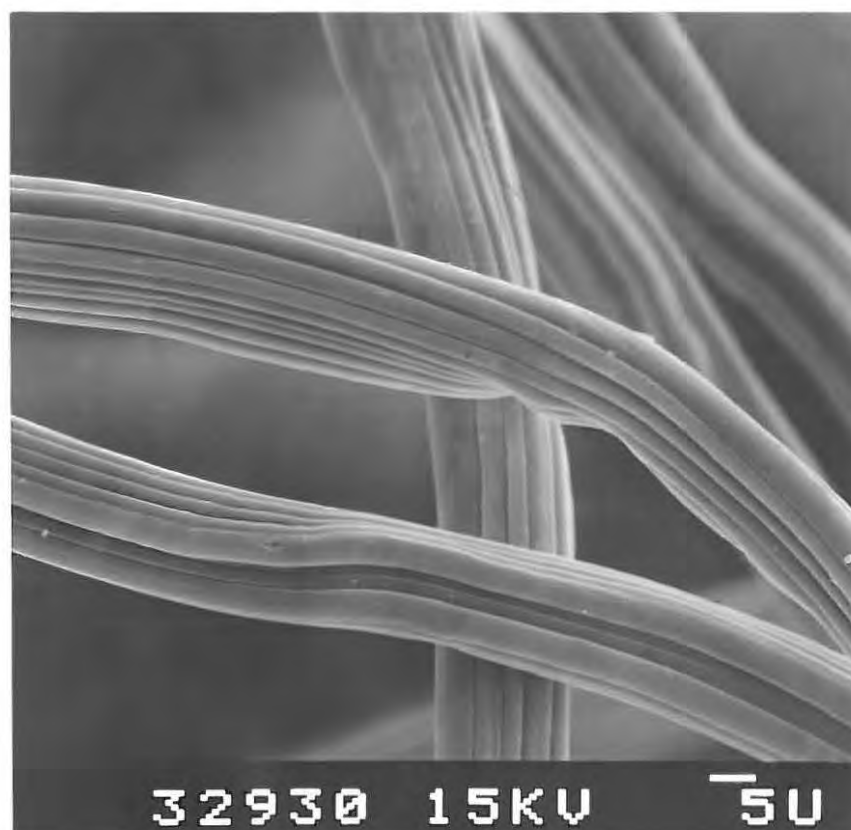


Figure 2.29 - Enka Bico fibre (magnification 1000x)

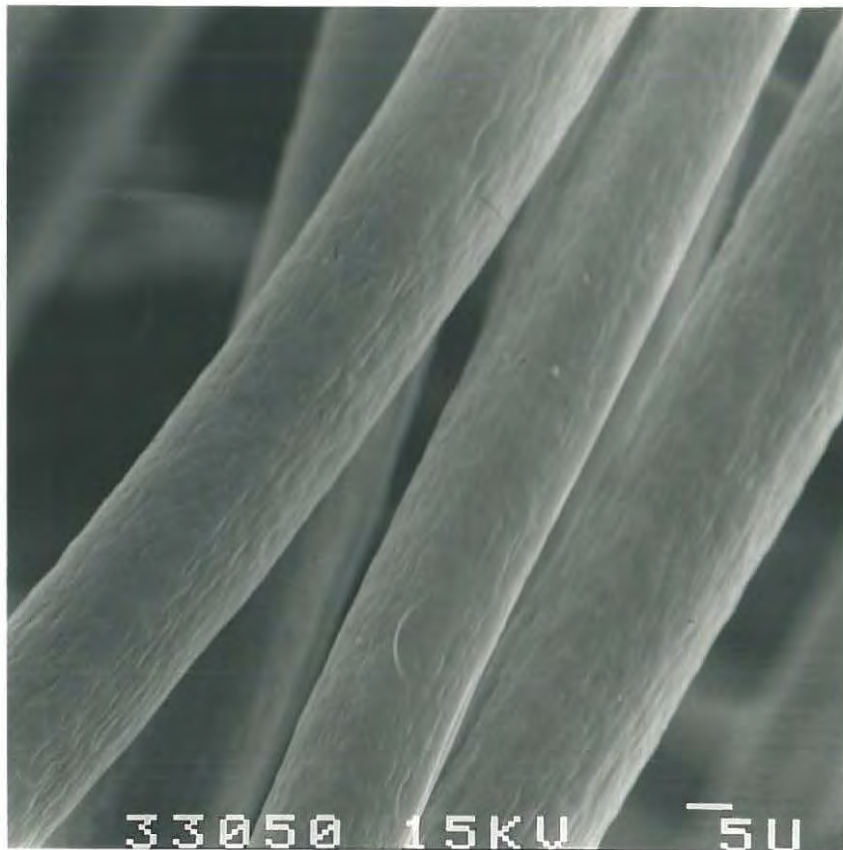


Figure 2.30 - Absorbent cotton wool (magnification 1000x)

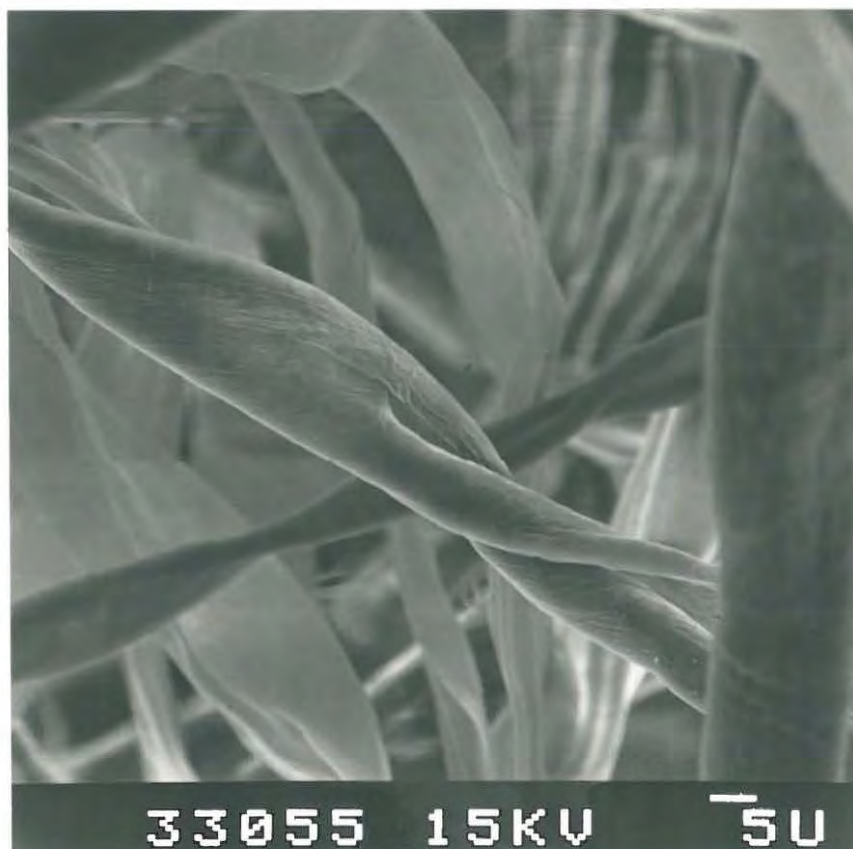


Figure 2.31 - Grilene fibre (magnification 5000x)

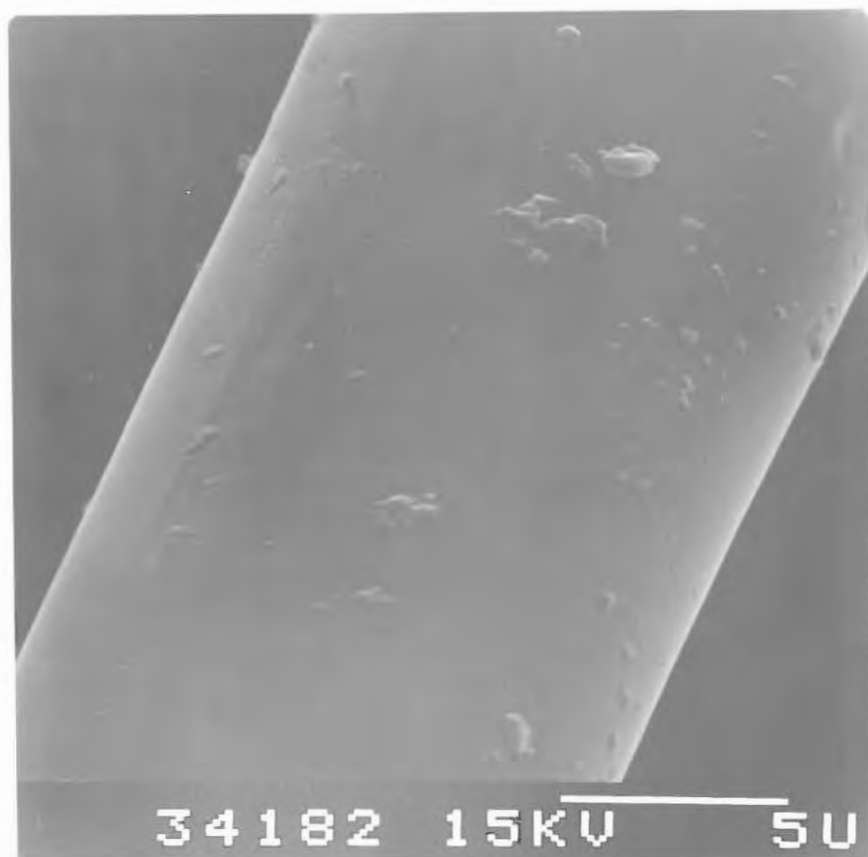


Figure 2.32 - Chisso EA fibre (magnification 5000x)

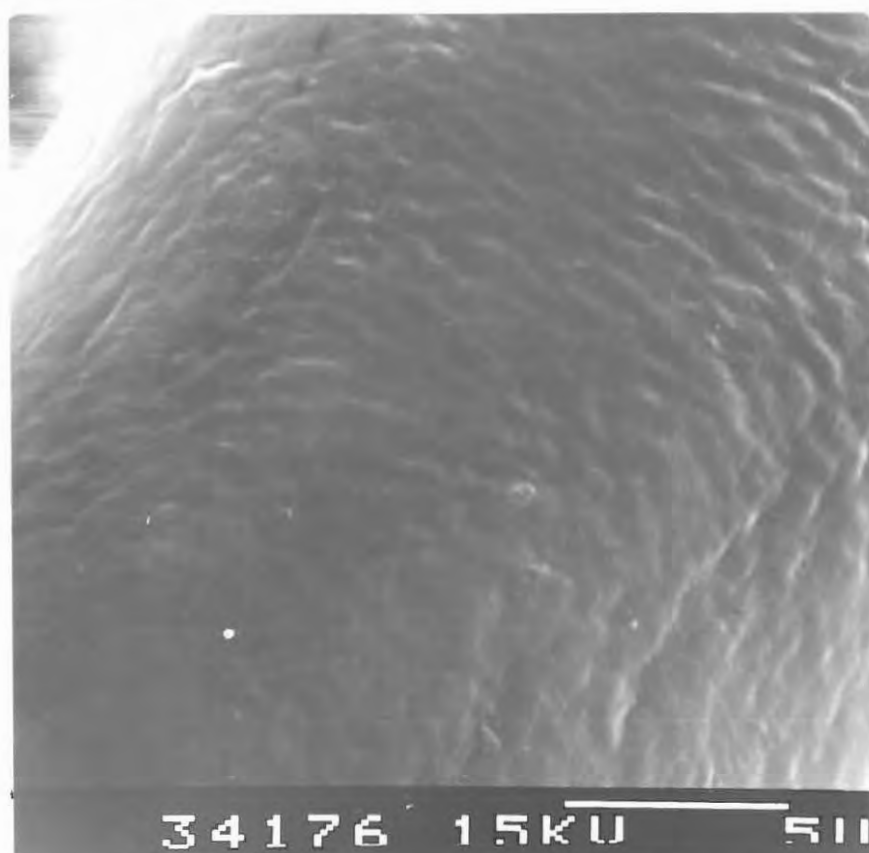


Figure 2.33 - Enka Bico fibre (magnification 5000x)

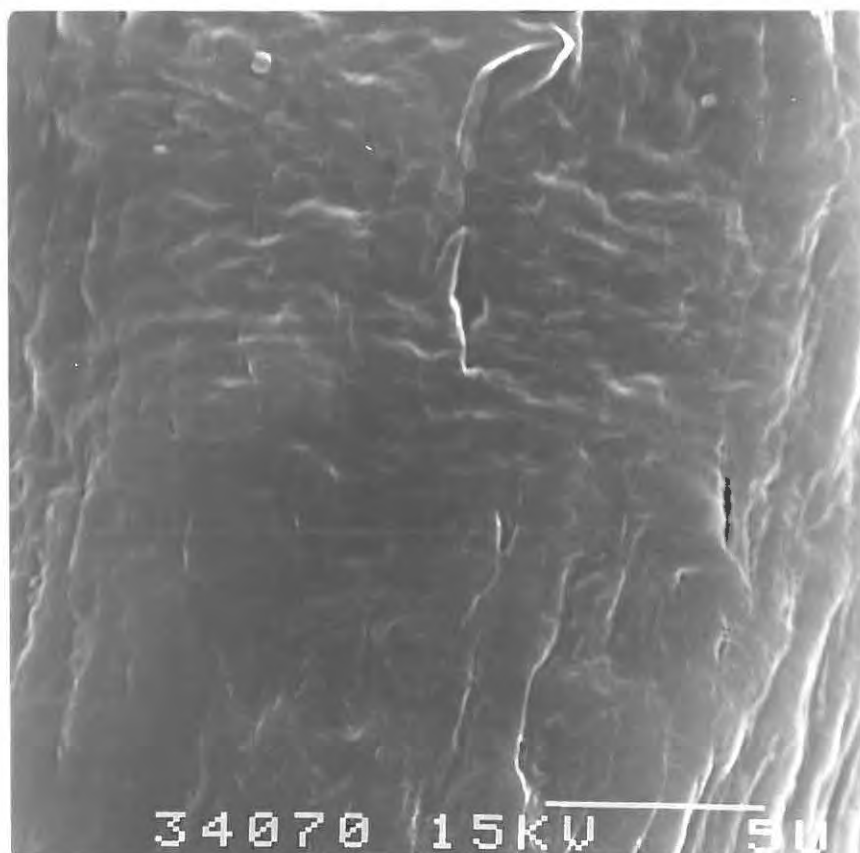


Figure 2.34 - Absorbent cotton wool (magnification 5000x)

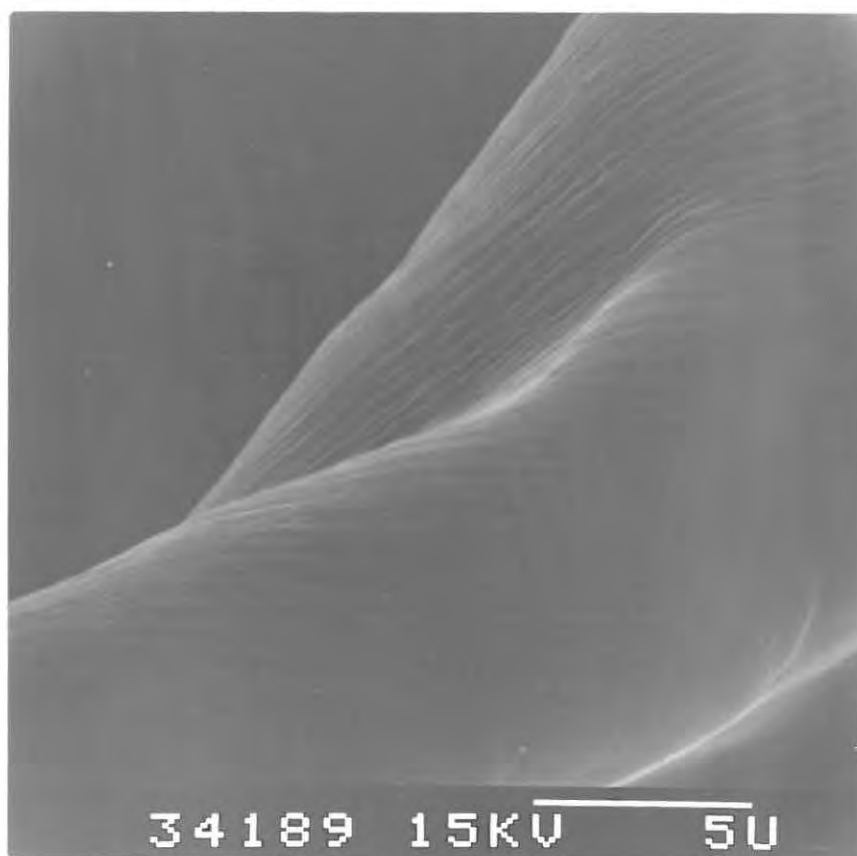


Figure 2.35 - Greige (raw) cotton (magnification 5000x)

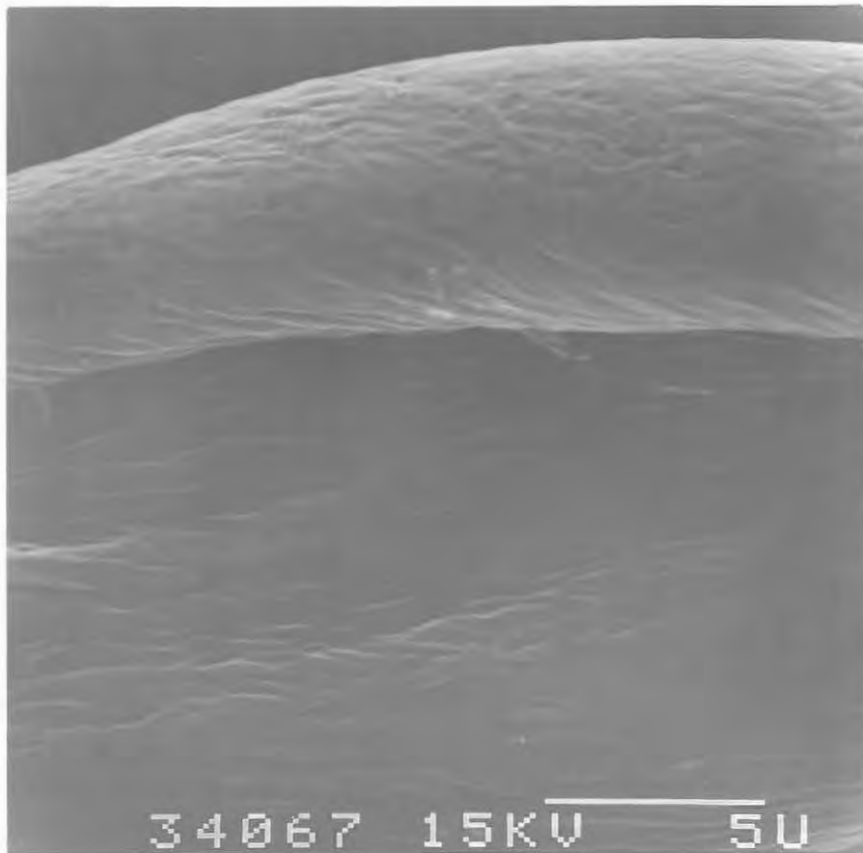
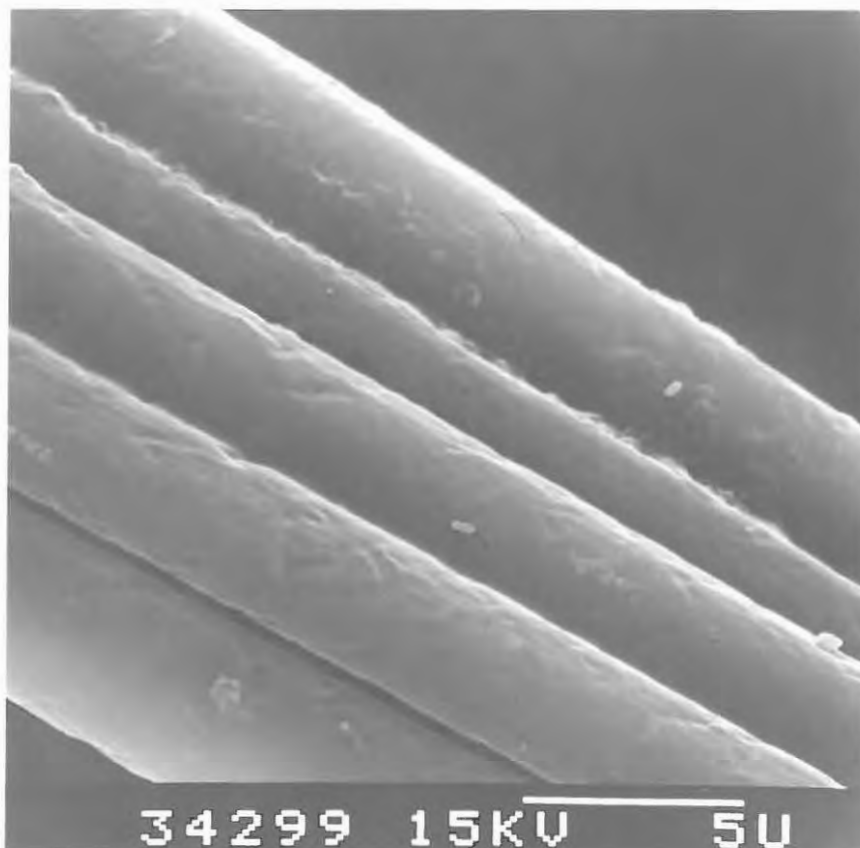


Figure 3.36 - Viscose rayon (magnification 5000x)



2.5 DISCUSSION

2.5.1 The cotton fibre in the stages of the cotton CPNF process

The SEM micrographs obtained in this work confirm the flat ribbonlike structure of cotton as well as the convolutions observed by other workers (13,32,34). These features could be seen clearly at the lower magnifications (figs 2.1 & 2.2). The reversals in the direction of twist of the convolutions could also be seen clearly (fig 2.1). The same characteristics could be seen in the micrographs of the cotton fibre that has been scoured and bleached in the final stage of the CPNF process (fig 2.2). More detailed examination at higher magnification (figs 2.3 and 2.4) revealed the wrinkled or corrugated aspect observed in the surface replica studies of other workers (13, 34). A slight difference was noticed between the greige (raw) cotton and the scoured and bleached fibres when magnification was taken further still (figs 2.34 & 2.35).

2.5.2 The cotton fibre variants (washed, solvent-extracted, solvent-extracted and washed, and absorbent cotton wool BPC)

Again in all the samples the flat ribbonlike structure of cotton could be clearly seen in the low magnification micrographs (figs 2.5 to 2.8). The convolutions and their accompanying features were there in all the experimental variations of the cotton fibre.

As before, the higher magnification micrographs (figs 2.9 to 2.12) failed to reveal any significant differences in the surfaces of the cotton fibre variants. The only apparent difference was in the washed and solvent-extracted fibre (fig 2.12), where a portion of the primary wall seemed to have lifted off the fibre at the bottom.

2.5.3 The other fibres

It was not possible to use the man-made fibres in the factory process because it would have interfered with normal production. As a result a specially designed pilot laboratory line was used to take these fibres. The cotton fibres were also subjected to this process to allow a proper comparison.

2.5.3.1 Spatial arrangements of Cotton and Synthetic fibres in the experimental fabrics

In this work the fabrics were made with a patterning time of 10 minutes (for details see chapter 8).

Low magnification (35X) micrographs of the experimental fabrics were taken in order to compare the spatial or geometric arrangement of the fibres in the webs that could not be patterned, to the geometric arrangement of the fibres in the webs that were patterned. The micrographs of the web of Chisso EA fibres (fig 2.13) that could not be patterned showed a mat of randomly arranged fibres.

The fabric made from the Enka fibre (fig 2.14) showed a barely discernible pattern. A successful CPNF could not be made with absorbent cotton wool. The fabric made with the absorbent cotton wool did not have a clear pattern, but there was a degree of fibre orientation diagonally across the micrograph (fig 2.15). In a successfully produced CPNF, the water jets will produce a hole.

The unbleached, entangled fabric made with greige (raw) cotton showed a well-defined hole that is typical of CPNF (fig 2.16). In the rayon fabric, there was a degree of fibre orientation to be seen, but no clear pattern formation (fig 2.17). In contrast, the rayon fabric made with the longer entangling time (20 minutes) exhibited the characteristics of a successful CPNF. This included a well-defined hole that is typical of CPNF.

When the fabric samples were examined under a higher magnification than before (100X), the main difference between Chisso EA fibres (fig 2.19) which was one of the fibres that could not be patterned and the fibres in the other fabrics (figs 2.20 to 2.24) was in the complete absence of any parallel fibres in it. The Chisso EA fibres had a springy, bulky quality to them in contrast to the bundles of close parallel fibres that could be seen in the other figures.

It would seem that this lack of compressibility and the resistance to orientation that the Chisso EA fibre has, are responsible for its inability to pattern.

2.5.3.2 The surfaces of the fibres used in the experimental fabrics

The examination of the fibre surfaces under high magnification (figs 2.25 to 2.30) revealed differences in their fibre surfaces. The Grilene fibre (fig 2.26) had the smoothest surface of all the fibres, followed closely by the Chisso EA fibre (fig 2.25). The Enka fibre surface (fig 2.27) showed some degree of roughness, looking almost like a slightly pitted surface. All three fibre types were cylindrical in shape. As before, there were slight differences between the absorbent cotton wool fibre and the greige (raw) cotton fibre (fig 2.28 to 2.29) respectively. The rayon fibre had deep ridges of almost uniform width along its length (fig 2.30). These ridges resulting in a surface roughness from the extrusion process, was most likely the reason for the ability of the rayon to form a successful CPNF.

The differences in the fibre surfaces were confirmed when the highest magnification (5000X) used in this SEM study was used to take the micrographs shown in figures 2.31 to 2.36.

2.6 CONCLUSION

The investigation using scanning electron microscope studies did not reveal any significant differences between the surface of cotton in its raw state and the surface of the cotton fibre in the final stage of scoured and bleached fabric in the CPNF process. The procedures used to remove water-soluble matter and waxes from the greige (raw) cotton did not had any detectable effect on the appearance of the cotton fibre surface. The SEM photographs were unable to distinguish between absorbent cotton wool and greige (raw) cotton.

The SEM study however showed very significant differences in the spatial arrangement of the fibres in the webs that could not be patterned and the fibres in the fabrics that were patterned with varying degrees of success. The SEM study also showed a distinct difference in the appearance of the surface of the fibres that could not be patterned and those that could be patterned. The surfaces of the fibres that could not be patterned (Grilene, Chisso, Enka) are generally smoother than the surfaces of the fibres that could be patterned (cotton and rayon).

The degree of success of CPNF patterning of fibres does seem to be related to the degree of roughness observed from the SEM micrographs. (See table 2.1 below).

TABLE 2.1

FIBRE	DEGREE OF SUCCESS IN PRODUCING CPF TYPE	DEGREE OF SURFACE ROUGHNESS
Grillon	Unsuccessful	Very smooth
Chisso EA	Unsuccessful	Fairly smooth
Enka	Limited success	Slightly pitted but not rough
Cottonwool	Limited success	Rough
Rayon	Successful	Smooth with ridges
Greige (raw) cotton	Successful	Rough

The inability of the cottonwool to form a successful CPNF must be due to another factor. The waxy, oily feel of the scoured and bleached CPNF as opposed to the dry feel of the cottonwool gives some hint as to the possible cause. This will be dealt with in Chapter 6 when the friction properties are discussed.

CHAPTER 3 - DSC AND CHEMICAL ANALYSIS OF COTTON SAMPLES

3.1 INTRODUCTION:

DSC was used to investigate possible differences in the cotton fibre before, during and after processing. The cotton fibre was thus analysed at the three different stages:

- i) the initial stage of greige (raw) cotton fibre
- ii) the intermediate stage of patterned fibre (unbleached CPNF)
- iii) the final stage of scoured and bleached fabric

DSC was selected because other workers have used thermal analysis techniques in the investigation of both natural and synthetic fibres (35,36,37,38,39).

3.2 EXPERIMENTAL:

3.2.1 Materials:

The greige (raw) cotton, unbleached CPNF and scoured and bleached CPNF samples were obtained from a production line. These were all sampled per batch i.e. for every batch of CPNF selected for analysis, the cotton was sampled at the three different stages of the process already mentioned above.

All laboratory samples were prepared from a single batch of greige (raw) cotton approved for CPNF production. This is the same cotton from which the fibre samples for friction tests have been taken. The growth area of the cotton cannot be revealed because it is proprietary information. Absorbent cotton wool BPC, a medical grade cotton, was used as a control sample. A pulp sample from SAICCOR (with 92,62% Alpha Cellulose) was used as reference sample.

3.2.2 Materials Preparation

A variety of laboratory samples were prepared in order to investigate the effects of some treatments that were used to vary the cotton fibres. Washed fibre was prepared to produce a fibre whose water-soluble matter had been extracted. Solvent-extracted fibre was prepared to produce a fibre with as much cotton wax as possible removed from the surface. Washed and solvent-extracted fibre was prepared to evaluate the effect of the combination of the abovementioned treatments. Water-soaked fibre was prepared to produce a fibre that had been contact with water alone, without any heat. Caustic-treated fibre (2% NaOH) was prepared to produce a fibre which had been subjected to an alternative means of removing wax, similar to the scouring process.

The washed samples were prepared by boiling greige (raw) cotton in distilled water (weight ratio of cotton to water 1:10) for 30 minutes followed by drying it at room temperature.

The solvent-extracted samples were prepared by Soxhlet extraction of greige (raw) cotton fibre according British Standards test method (9) using analytical grade dichloromethane (MERCK). The soaked samples were prepared by soaking greige (raw) cotton in enough distilled water or solvent (dichloromethane) to cover a 2g sample placed in a 100ml beaker. The samples were left for 24 hours in the soaking liquid of choice and later dried at room temperature. The caustic-treated fibre was prepared by boiling the greige (raw) cotton in 2% NaOH (weight ratio of cotton to solution 1:10) for 30 minutes followed by rinsing the fibre with cold distilled water until the rinse water was neutral to litmus paper.

3.2.3 Sample preparation

A sample mass of 10mg was used for all analyses. The cotton fibre was chopped finely with a pair of scissors and passed through a 300 micrometer (aperture size) sieve. The Absorbent cotton wool BPC used as a control was prepared in the same method described for the greige (raw) cotton fibre. The fibre samples and the fluffed pulp were pressed firmly on the aluminium sample pans. The unbleached fabric samples and the bleached fabric samples were cut with an office punch. The fabric was folded into layers before punching to product a disc. The resultant discs were the correct diameter to fit the aluminium sample pans snugly.

3.2.4 Instrumentation

The apparatus used was a DuPont 990 Thermal Analyser system equipped with a temperature programmer, an X-Y plotter and a DSC module. A heating rate of 10°C/minute was used throughout and the heating range was from ambient to 575°C. All analyses were performed in an inert atmosphere of dry nitrogen (500 kPa) at a flow rate of 10ml/min. The sample pans were covered with aluminium lids with a hole pierced through.

3.2.5 Chemical Analysis

The water-soluble extractables of the greige (raw) cotton samples were determined in order to find out what could possibly be extracted by the water from the cotton during the patterning process. Although the experimental conditions were harsh (extraction with boiling distilled water) compared to processing conditions, the results are useful because they give an indication of the possibilities.

3.2.5.1 Determination of % water-soluble extractables

The cotton fibre was first conditioned at room temperature in the air-conditioned laboratory (20-22°C, 50⁺5% relative humidity). A 2g sample (weighed to the next 0.1mg) was placed in a clean 400ml beaker. 200ml distilled water was added to the beaker and the level marked.

The sample was boiled for 30 minutes with frequent stirring while adding water to maintain the original level when necessary. The hot liquid was filtered through Whatman No 1 filter paper into a clean dry 250ml beaker of known weight. (The beaker was dried for 1 hour in an oven set at 105°C before being weighed). As much water as possible was squeezed from the sample using a glass rod.

The filtrate was then boiled down gently on an electric hot plate until the beaker was almost dry, before being transferred to an oven set at 105°C. The beaker was dried for 3 hours before being cooled in a dessicator and weighed. The soluble extractables were determined from the mass of the dried residue as a percentage of the mass of the cotton sample.

In order to check whether any waxy substances were removed during the extraction of water-soluble matter, some of the dried residue was analysed by DSC. A very small peak was noted at 51°C, showing that there was a very small amount of wax removed during the extraction with boiling distilled water.

3.2.5.2 Analysis of water-soluble extractables by Atomic Absorption

The water-soluble extractables were analysed by Atomic Absorption in order to identify and determine the concentration of the major cations. The dried residue was dissolved, the solids filtered off, and the analysis performed in the usual manner. This was done in order to determine which inorganic salts were responsible for the differences noted in the DSC thermograms of the samples from the three stages of the CPNF process. The atomic absorption analysis was performed by Mr Heine Kruger of LIRI, Rhodes University, on the dried water-soluble matter.

3.2.5.3 Determination of metal content in cotton samples

The greige (raw) cotton, unbleached CPNF and bleached CPNF from one batch, were analysed by SAWTRI, Port Elizabeth, together with a sample of the greige (raw) cotton used for the laboratory samples, for metal content analysis. This was done in order to determine the change in concentration of the inorganic content as the CPNF process proceeds from greige (raw) cotton to the final bleached product. The analysis was performed on the sulphated ashes of the cotton samples, using an X-ray Energy Dispersive Spectrometer (XEDS).



3.2.5.4 Identification of the Anions in the water-soluble extractables

Qualitative tests for the presence of nitrates, sulphates and phosphates were performed on the water-soluble extract. The extract was obtained by boiling a 20g raw cotton in a litre of distilled water for 30 minutes, filtering it through Whatman No 1 filter paper while hot and boiling it down to 200ml to concentrate it. The concentrate was allowed to cool to room temperature before being tested.

To test for nitrates, 10ml of the filtrate was acidified with concentrated sulphuric acid, a pinch of copper turnings added and the mixture heated. Red fumes are produced on heating in a positive test for nitrates. The test was negative.

To test for sulphates, about 2ml of barium chloride solution was added to 10ml of the filtrate. The test is positive if a white precipitate which is insoluble in hydrochloric acid is formed. The test was positive.

To Test for phosphates, the pH of the filtrate was tested before adding about 2ml of silver nitrate solution to 10ml of the filtrate. The test is positive if a yellow precipitate which is soluble in dilute nitric acid (as distinct from chlorides which give a precipitate which is insoluble in nitric acid).

Before dilute nitric acid was added to the filtrate, the pH was again tested. An acid solution denotes the presence of acid phosphate (H_2PO_4^- or HPO_4^{2-}) while a neutral solution denotes the presence of normal phosphate (PO_4^{3-}). The test was positive for acid phosphate (H_2PO_4^- or HPO_4^{2-})*.

Clearly only the solutions of KH_2PO_4 are acidic after the addition of silver nitrate solution.

Presence of Chlorides

When the test for phosphates was performed (see previous page), although nearly all the precipitate dissolved on the addition of dilute nitric acid, a very dilute suspension remained. This confirmed the presence of trace quantities of chloride in the water-soluble extract.

* A quick experiment was run to check whether pH measurement could be used in the test, to distinguish between H_2PO_4^- , HPO_4^{2-} , and PO_4^{3-} ions. 10ml volumes of weak solutions of the potassium salts of the above anions were prepared in test tubes. The pH of the solutions was measured before the addition of silver nitrate solution (K_3PO_4 solution pH = 12.03, K_2HPO_4 solution pH = 9.10 and KH_2PO_4 solution pH = 4.5) The pH was then measured after the addition of the AgNO_3 solution (K_3PO_4 solution was 11.52 ; K_2HPO_4 solution was 7.72 ; KH_2PO_4 solution was 2.96).

3.2.5.5 Analysis of Water-soluble extractables by Ion Chromatography

The water-soluble extract was prepared by boiling a 5g sample in 500ml distilled for 30 minutes while topping it up to retain the original volume. The hot extract was filtered through Whatman No 1 filter paper into a clean dry beaker and boiled down to about 25ml before being transferred to a clean sample bottle. Two samples were prepared in this manner and analysed by Prof Bob Hasty of UNISA by Ion Chromatography. A Dionex 4000 ion chromatograph with a conductivity detector and a AS 4A column was used. The eluent was a carbonate/bicarbonate solution.

The results of the ion chromatography analysis identified the presence of phosphate, sulphate, chloride, nitrate and fluoride ions in the ratio of 30:50: 30:1,5 mass per volume.. Their concentrations were also given. The technique could not distinguish between HPO_4^{2-} , H_2PO_4^- and PO_4^{3-} . The results support the qualitative analysis in section 3.2.5.4.

3.2.6 DSC Analysis

The samples from the three stages of CPNF from different batches were analysed by DSC. After the cations present in the water-soluble substances had been identified, the individual effects of the sulphates, chlorides and the orthophosphates of the metals concerned (K, Ca, Mg, and Na) on the washed cotton were also investigated.

This was done by soaking a bleached CPNF sample (all samples were soaked in a 1% solution of the salt) and leaving it to dry for 24 hours at room temperature before being analysed. Some samples were also subjected to DSC analysis after soaking in 10% solution of the KCl and KH_2PO_4 salts in order to investigate the effect of the higher salt concentration on the hygroscopic properties. The cotton samples treated with the higher concentration (10%) were dried for 2 hours in an oven set at $105^{\circ}C$. The control sample was also similarly dried and the DSC analysis repeated.

3.3 RESULTS

DSC Thermograms

Figure 3.1 - Greige (raw) cotton

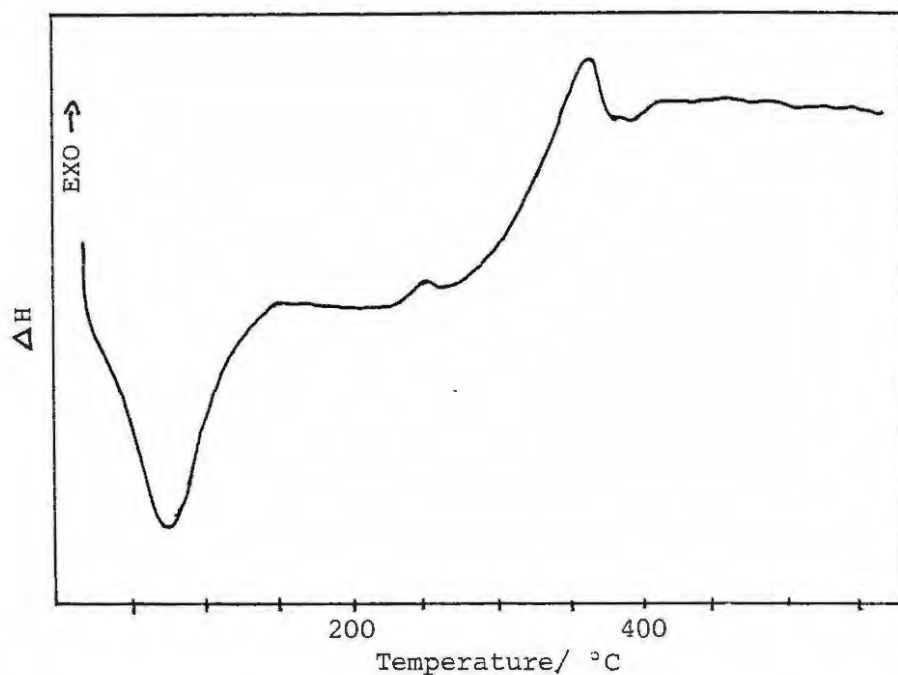


Figure 3.2 - Unbleached Cotton CPNF

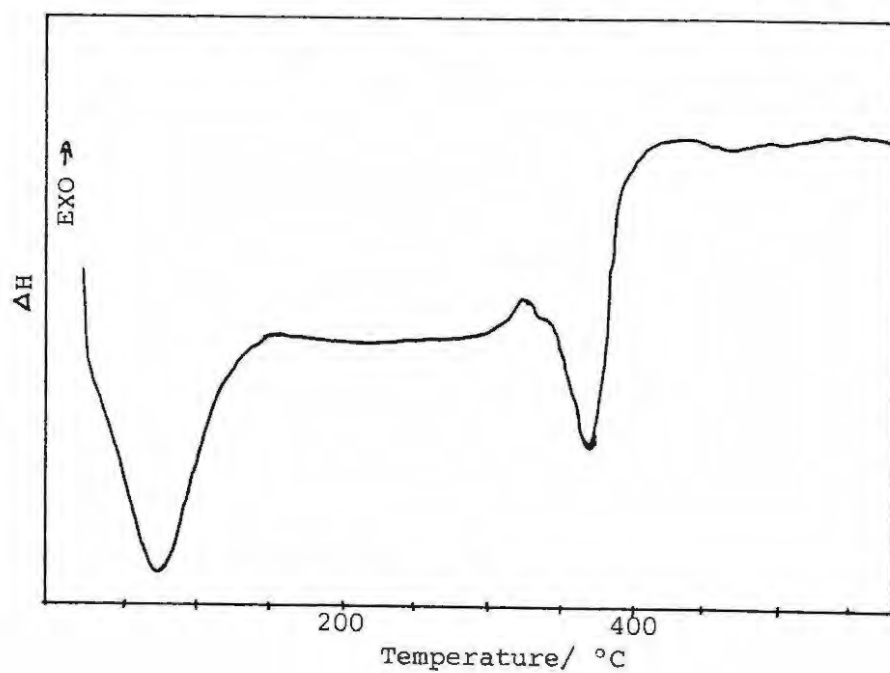


Figure 3.3 - Bleached Cotton CPNF

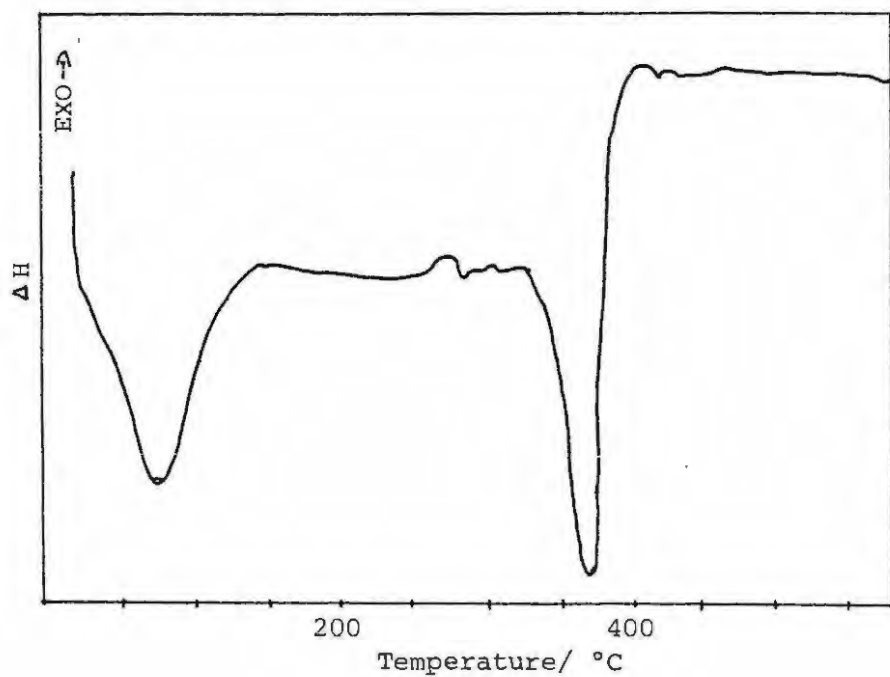


Figure 3.4 - Oven-dried, Bleached Cotton CPNF

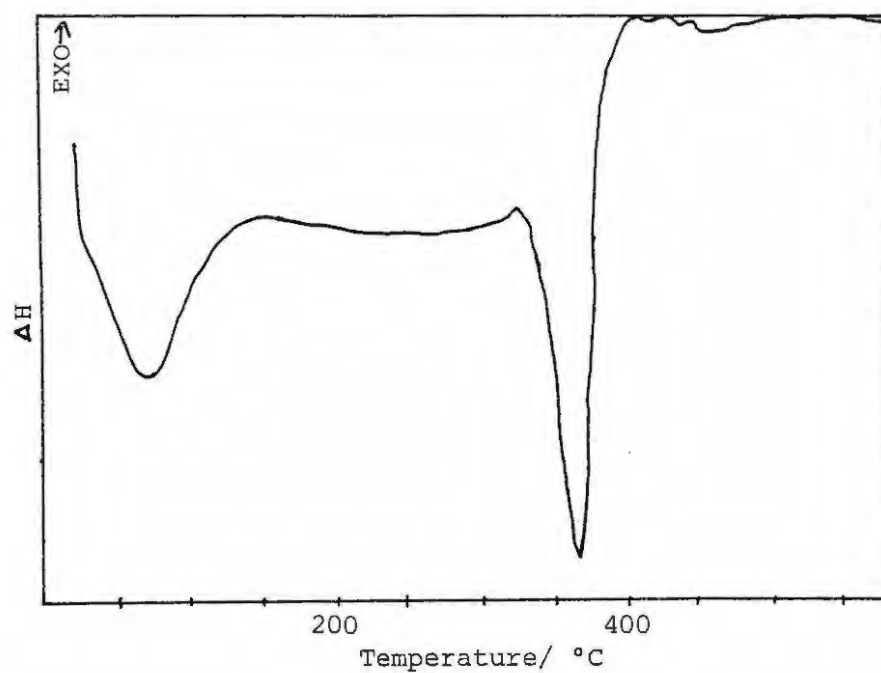


Figure 3.5 - Bleached Cotton CPNF soaked in 1% KCl solution and dried at room temperature

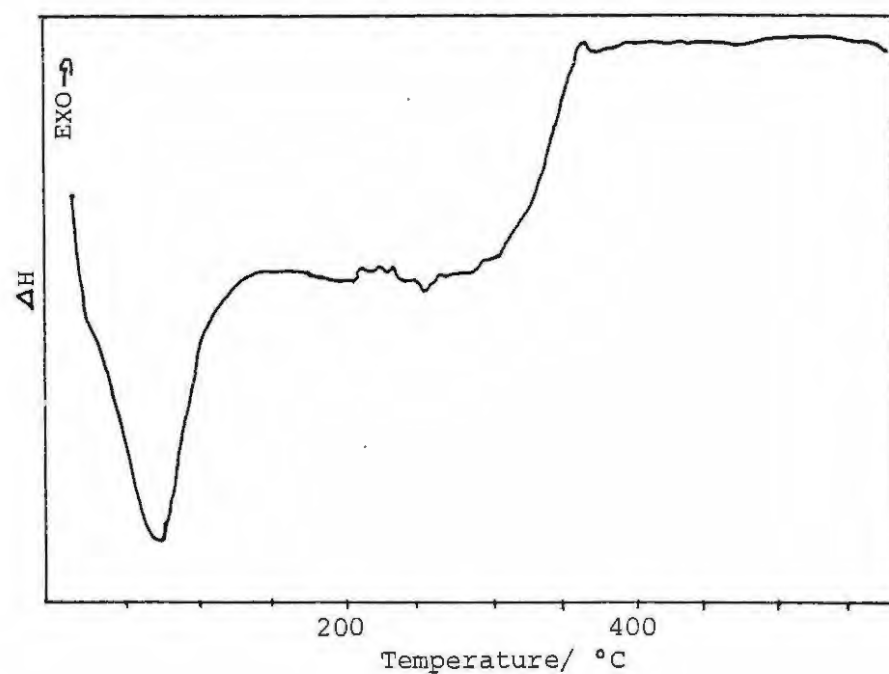


Figure 3.6 - Bleached Cotton CPNF soaked in 10% KCl solution and dried at room temperature

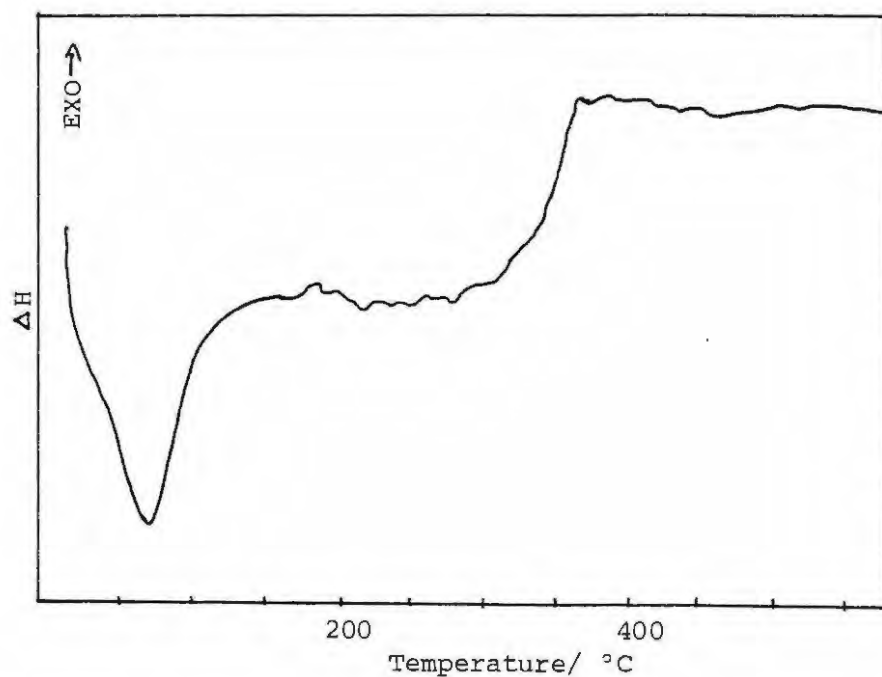


Figure 3.7 - Bleached Cotton CPNF soaked in 1% K_2HPO_4 solution and dried at room temperature

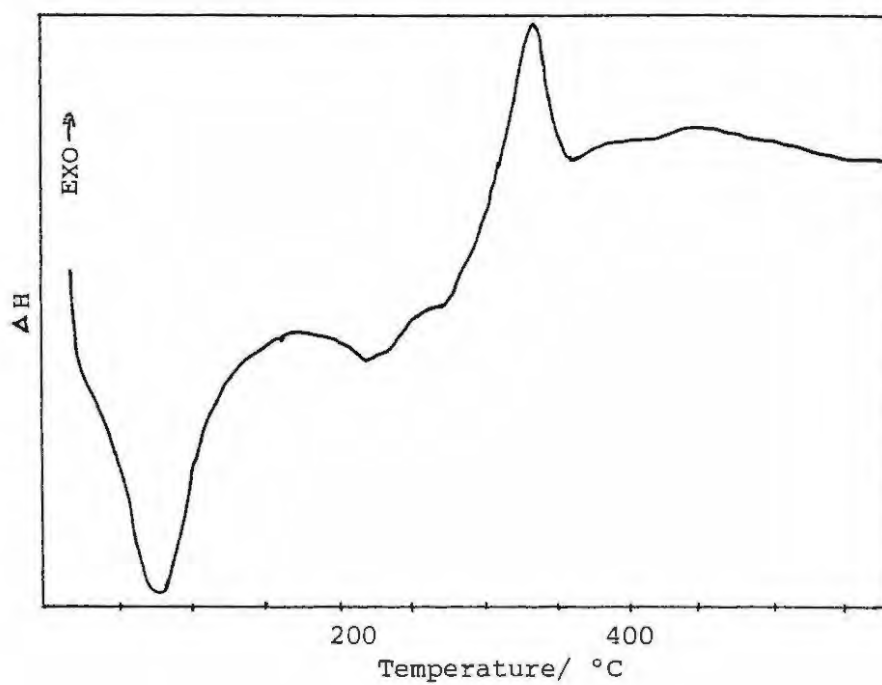


Figure 3.8 - Bleached Cotton CPNF coated in 10% K_2HPO_4 solution and dried at room temperature

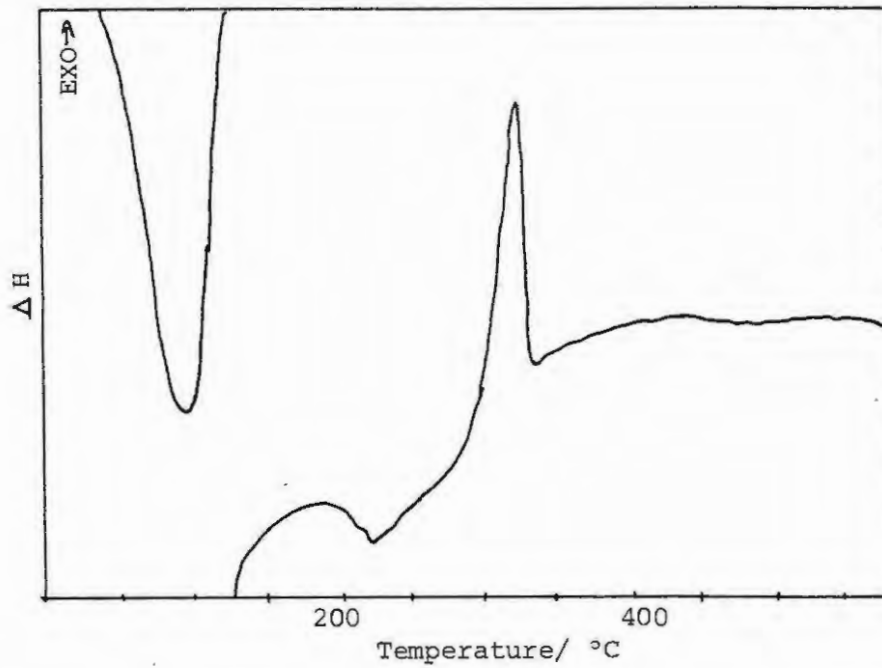


Figure 3.9 - Oven-dried Bleached Cotton CPNF soaked in 10% K_2HPO_4 solution

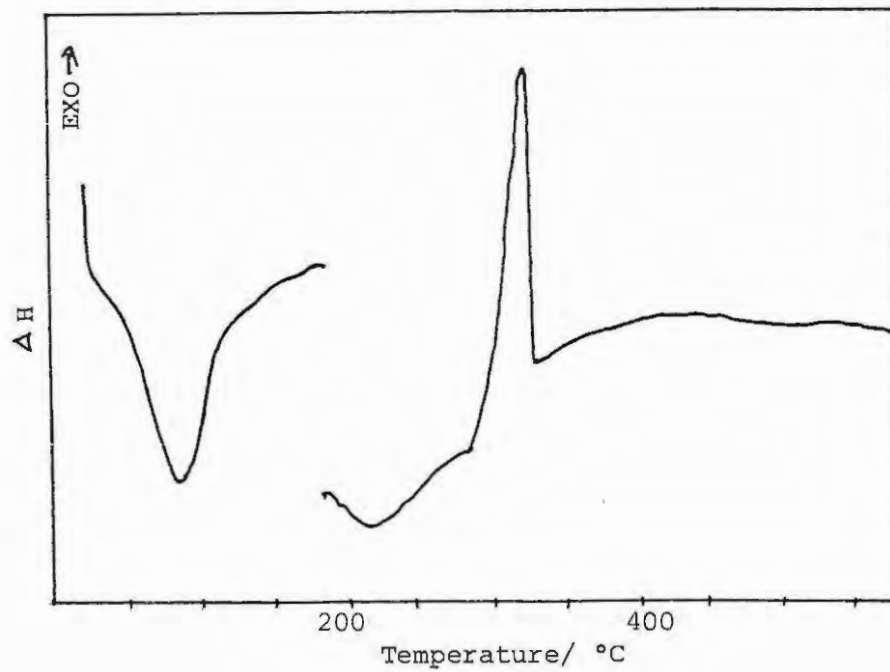


Figure 3.10 - Absorbent Cotton Wool BPC

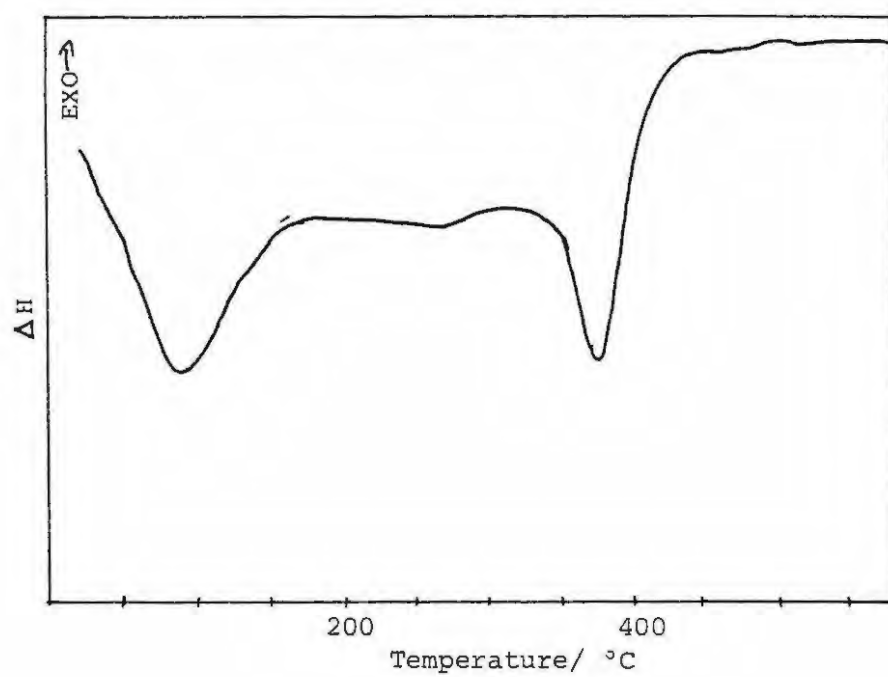


Figure 3.11 - Washed Cotton Fibre

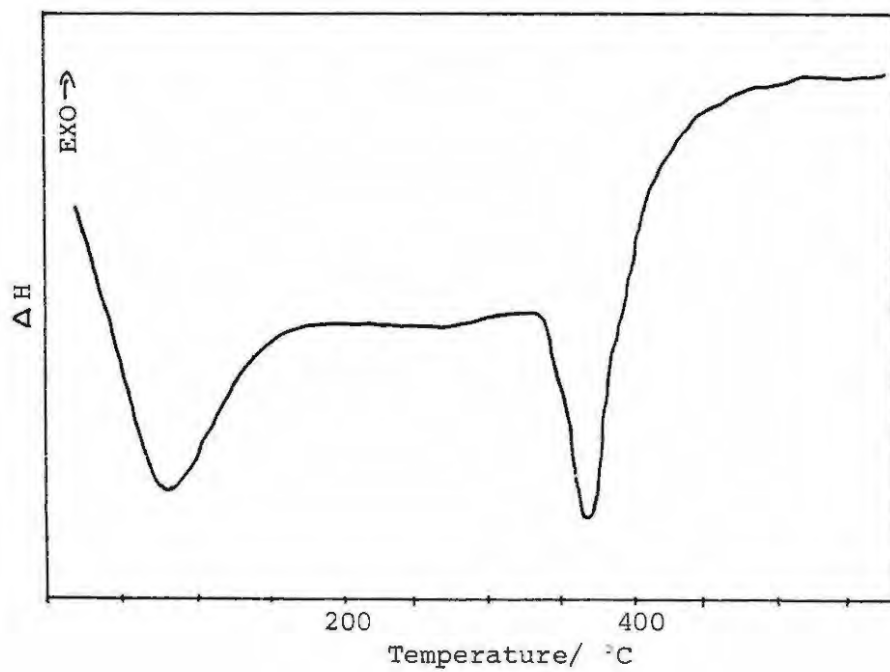


Figure 3.12 - Solvent-extracted Cotton Fibre

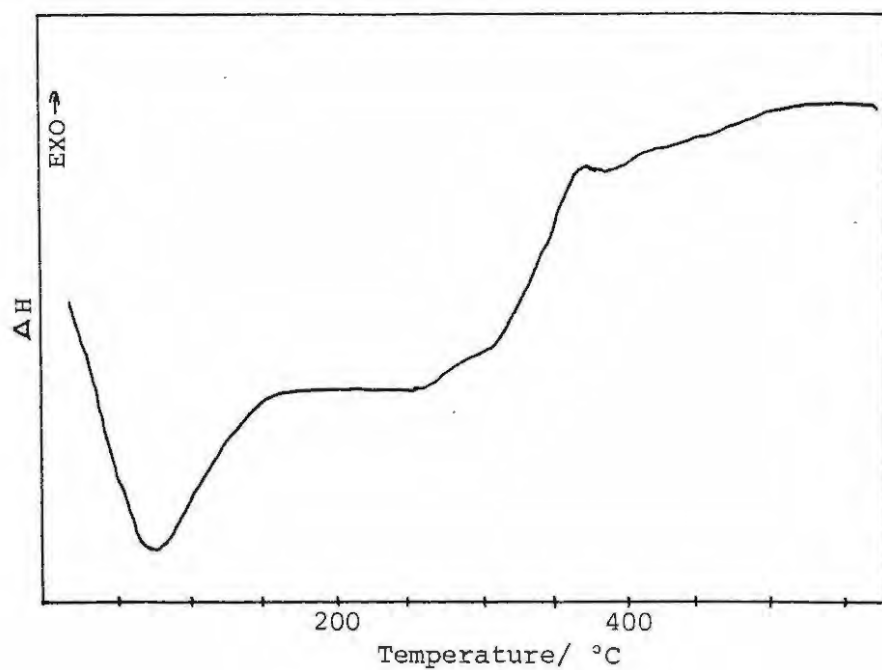


Figure 3.13 - Cotton Fibre treated with 2% NaOH solution

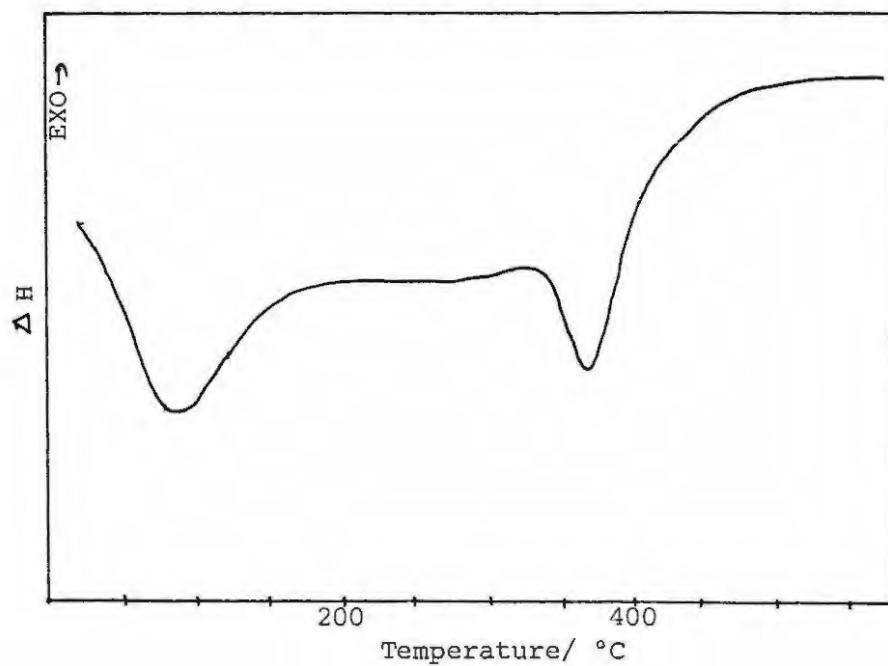


Figure 3.14 - Washed, Solvent-extracted Cotton Fibre

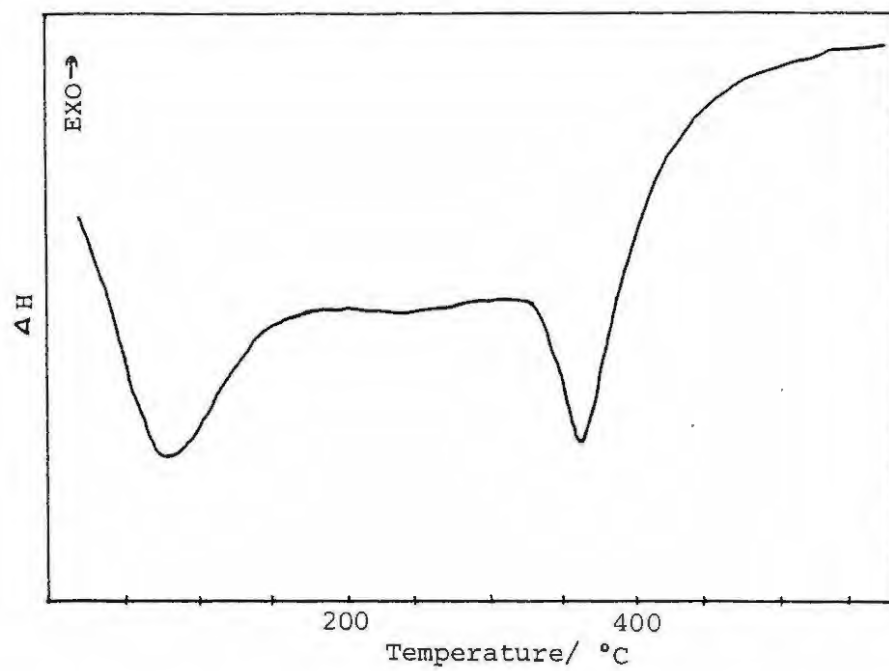


Figure 3.15 - Water-soaked Cotton Fibre

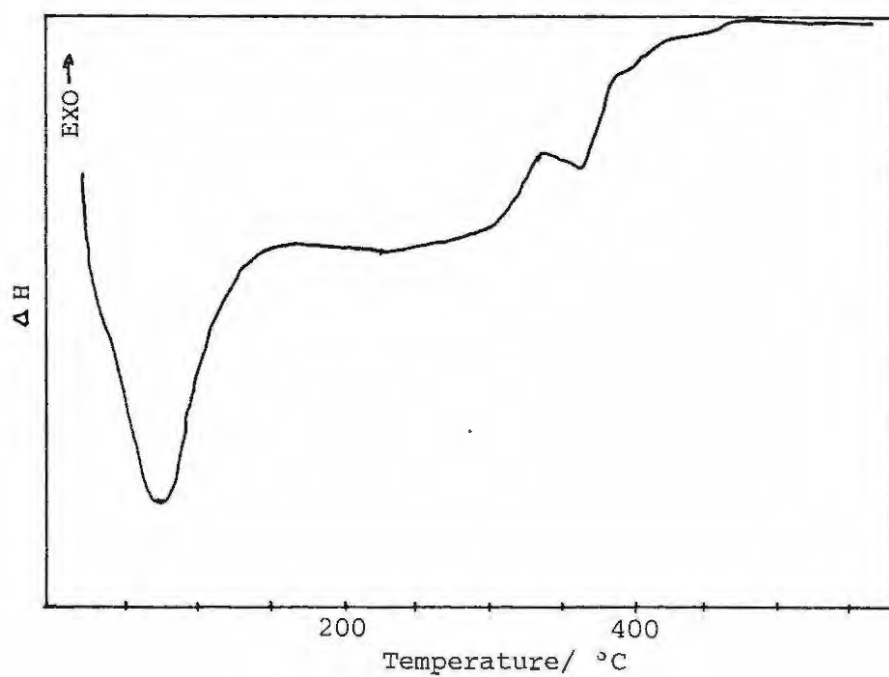


Figure 3.16 - Solvent-soaked Cotton Fibre

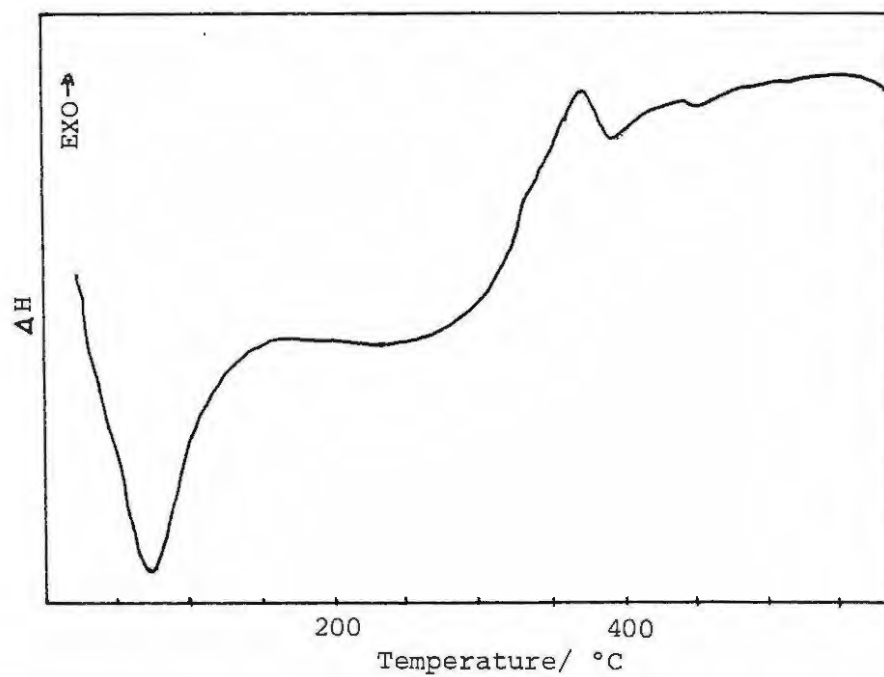


Figure 3.17 - Fluffed Pulp

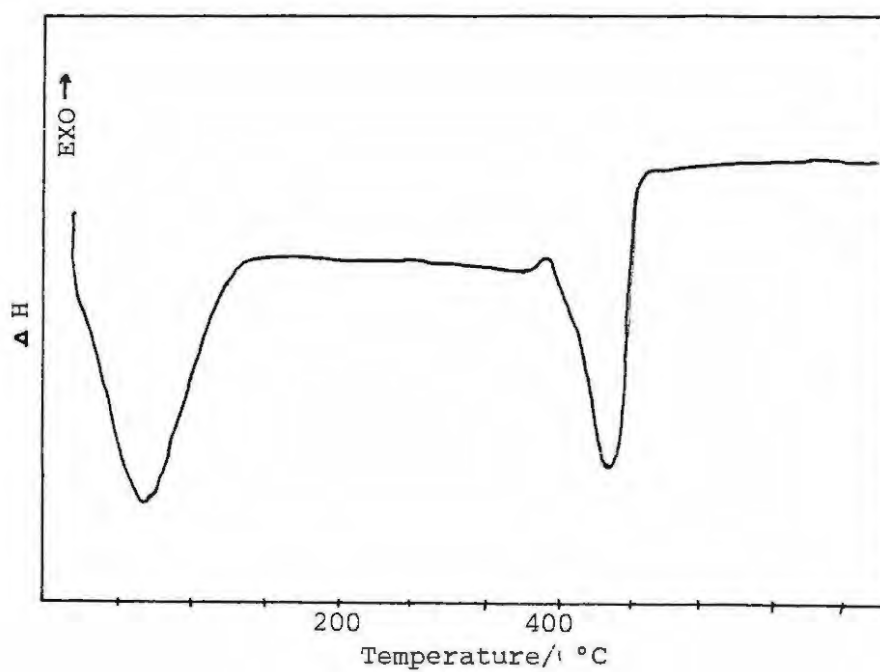


Figure 3.18 - Bleached Cotton CPNF soaked in 1% KH_2PO_4 solution

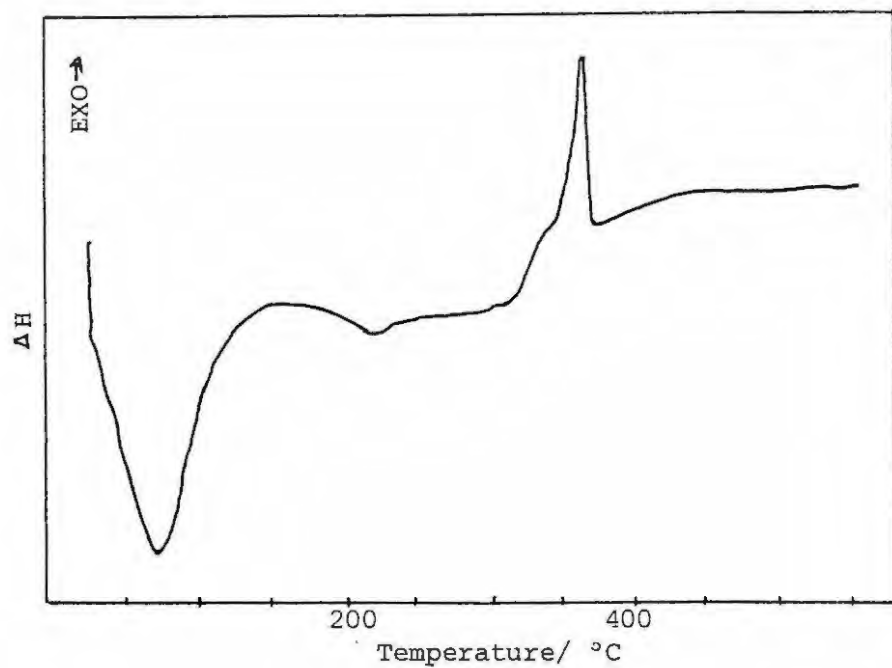


Figure 3.19 - Bleached Cotton CPNF soaked in 1% $\text{K}_3\text{PO}_4 \cdot 7\text{H}_2\text{O}$ solution

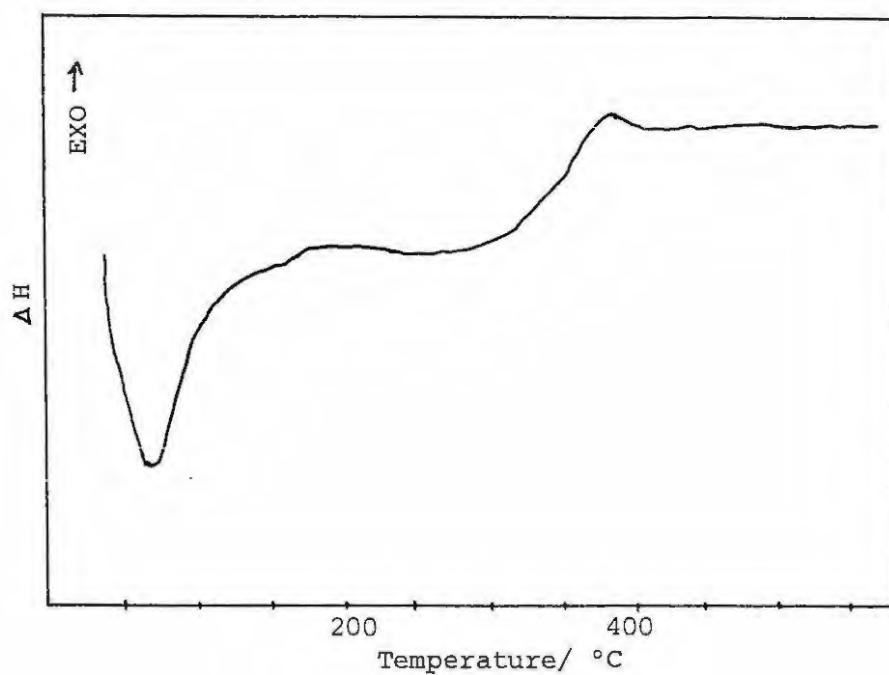


Table 3.1 - Results of DSC Analysis of the cotton samples

		MOISTURE		ONSET OF		PYROLYSIS	
SAMPLE		PEAK	TYPE	PYROLYSIS		PEAK	TYPE
1.	Raw cotton	75°C	Endothermic	292°C		367°C	Exothermic
2.	Unbleached CPNF	78°C	Endothermic	344°C		372°C	Endothermic
3.	Bleached CPNF	76°C	Endothermic	343°C		369°C	Endothermic
4.	Ovendried						
	bleached CPNF	72°C	Endothermic	344°C		372°C	Endothermic
5.	Bleached CPNF						
	soaked in 1%						
	KCl solution	75°C	Endothermic	320°C		368°C	Exothermic
6.	Bleached CPNF						
	soaked in 10%						
	KCl solution	73°C	Endothermic	332°C		371°C	Exothermic
7.	Bleached CPNF						
	soaked in 1%						
	K ₂ HPO ₄ soln	79°C	Endothermic	296°C		342°C	Exothermic
8.	Bleached CPNF						
	soaked in 10%						
	K ₂ HPO ₄ soln	95°C	Endothermic	293°C		325°C	Exothermic
9.	Overdried						
	bleached CPNF						
	soaked in 10%						
	K ₂ HPO ₄ soln	88°C	Endothermic	300°C		326°C	Exothermic
10.	Absorbent						
	Cotton Wool						
	BPC control	93°C	Endothermic	355°C		384°C	Endothermic

Note: "Bleached CPNF" refers to the scoured and bleached fabric of the final CPNF stage.

Table 3.1 continued

SAMPLE	MOISTURE		ONSET OF PYROLYSIS	PYROLYSIS	
	PEAK	TYPE		PEAK	TYPE
11. Washed cotton	83°C	Endothermic	350°C	374°C	Endothermic
12. Solvent- extracted cotton	77°C	Endothermic	325°C		Exothermic
13. Cotton treated with 2% NaOH soln	90°C	Endothermic	350°C	377°C	Endothermic
14. Washed, solvent extracted cotton	83°C	Endothermic	345°C	378°C	Endothermic
15. Water-soaked cotton	77°C	Endothermic	349°C	372°C	Endothermic
16. Solvent- soaked cotton	62°C	Endothermic	298°C	378°C	Exothermic
17. Fluffed pulp from SAICCOR	69°C	Endothermic	331°C	358°C	Endothermic
18. Bleached CPNF soaked in 1% KH_2PO_4 soln	73°C	Endoethermic	293°C	337°C	Exothermic
19. Bleached CPNF soaked in 1% $\text{K}_3\text{PO}_4 \cdot 7\text{H}_2\text{O}$ soln	72°C	Endothermic	293°C	344°C	Exothermic

NOTE: "Bleached CPNF" refers to the scoured and bleached fabric of the final CPNF stage.

Table 3.2 - % Water-soluble extractables content of cotton

<u>Sample No</u>	<u>% Water soluble extractables</u>
1	2,77
2	2,62
3	2,20
4	2,28

Table 3.3 - Metal content of water-soluble extractables expressed as % mass of dried solids (AA determination)

<u>Metal Identified</u>	<u>% Content of dried solids</u>	<u>% content* in cotton</u>
Potassium (K)	34,21	0,86
Sodium (Na)	1,30	0,03
Magnesium (Mg)	3,67	0,09
Calcium (Ca)	0,46	0,12

* These are estimates based on a sample mass of 5g from the raw cotton and an average % water-soluble extractables of 2,5%

Table 3.4 - Anions identified in water-soluble extract (Ion Chromatography)

<u>Anion Identified</u>	<u>% Contents in mg per litre</u>
Phosphate	30
Sulphate	50
Chloride	30
Nitrate	1,5
Fluoride	Trace

NOTE: Technique could not distinguish between HPO_4^{-2} , H_2PO_4^- or PO_4^{3-}

Table 3.5 - Metal content of cotton samples from the three stages of the CPNF process
(XEDS determination)

Samples	Element concentration (mg/g cotton)							
	Na	Mg	Al	Si	K	P	Ca	Fe
A ₁ greige (raw) cotton	0,00	0,45	0,01	0,00	2,29	0,04	0,42	0,00
A ₂ unbleached CPNF	0,01	0,61	0,05	0,09	0,15	0,18	1,10	0,01
A ₃ scoured & bleached								
CPNF	0,02	0,11	0,02	0,08	0,02	0,02	0,24	0,00
B greige (raw) cotton	0,00	0,98	0,07	0,02	3,37	0,21	0,46	0,00

Note: Samples A₁, A₂ and A₃ were sampled from different stages of the same batch.

3.4 DISCUSSION

3.4.1 The moisture loss reaction

All the thermograms begin with a prominent endotherm which was completed at an average temperature of 155°C for the samples shown in figures 3.1 to 3.7, and at 185°C for figures 3.8 to 3.12 with the highest temperature being 210°C in figure 3.13. This endotherm is due to moisture loss in the sample (16, 17, 40). The temperatures at which the maximum rate of moisture loss occurred in the various samples are listed in Table 3.1.

These peak temperatures are in the range 62 - 95°C. The peak temperature occurs at 69°C in the pulp reference sample and at 93°C in the absorbent cotton wool BPC. The analysis conducted in this study showed that the peak temperature of the moisture loss endotherm is determined by the moisture content of the sample when analysis parameters such as sample size, sample pan geometry, furnace atmosphere, heating rate etc are kept constant.

This is best illustrated by comparing figures 3.3 and 3.4 as well as figures 3.8 and 3.9. In figure 3.3, the peak temperature of the moisture loss endotherm occurs at 76°C. An oven-dried sample from the same fabric (figure 3.4) exhibits a moisture loss peak at 72°C, a four degree shift to the left compared to the sample before it was oven-dried (figure 3.3). The peak temperature of the moisture loss reaction shifts to the right, to higher temperature, with increasing moisture content in the samples.

The fabric samples which were soaked in potassium hydrogen orthophosphate (K_2HPO_4) solution were hygroscopic and felt damp to the touch after being dried at room temperature like the other samples. The hygroscopic effect increased with concentration, as can be seen by comparing the size of the moisture loss peaks for samples of nearly similar masses (10,2mg and 10,4mg) in figures 3.7 and 3.8 respectively. The oven-dried fabric that was soaked in 10% K_2HPO_4 solution had a moisture loss peak at 88°C, seven degrees lower than was observed in Figure 3.8 before the sample as dried.

The differences in the moisture content of the samples were found to be responsible for the variation in the temperatures at which the moisture loss reaction is completed. This temperature shifted to higher values for those samples that had a higher moisture content. This is best illustrated by the control sample in figure 3.10 (moisture loss reaction completed at 185°C) and the orthophosphate solution treated CPNF (moisture loss reaction completed at 180°C for 10% concentration).

After the moisture loss peak, the next feature in the thermogram is a shallow endothermic region in the 150°C - 300°C range, whose size varies. This reaction may be due to the decomposition of the waxes found in the cotton fibre surface (16). The thermograms of the CPNF samples that were soaked in K_2HPO_4 solution (see figs 3.7 to 3.9) were different from the rest in that they exhibited a small but well-defined peak in the region of 185°C to about 265°C, with peak positions in the 214°C - 224°C region.

The position of this peak is influenced by the concentration of the K_2HPO_4 solution, and it shifts to higher temperature with increasing concentration. It is also affected by moisture content, with peak position shifting to lower temperatures when the concentration of the potassium salt solution remains constant (see figures 3.8 and 3.9).

3.4.2 The Thermal Degradation of the cotton samples

In the region beyond 290°C, the thermogram of the scutched greige (raw) cotton (figure 3.1) which is the first stage of the cotton CPNF process differs from the thermograms of the second stage (unbleached CPNF) and the third stage (scoured and bleached CPNF) seen figures 3.2 and 3.3 respectively. The greige (raw) cotton exhibits an exothermic peak which starts at 282°C, has a peak at 367°C and is completed at 385°C. The unbleached CPNF and the bleached CPNF both exhibit a large endothermic peak which starts at 344°C, has a peak at about 372°C and is completed by 415°C (refer to Table 3.1). These results are in agreement with the findings of other workers (16, 17, 40).

The thermogram of the oven-dried bleached CPNF exhibits an endothermic peak whose peak has shifted to a slightly lower temperature at 369°C. The decomposition of cellulose occurs in this region and has been found to be an exothermic reaction in raw cotton and an endothermic one in scoured or washed cotton.

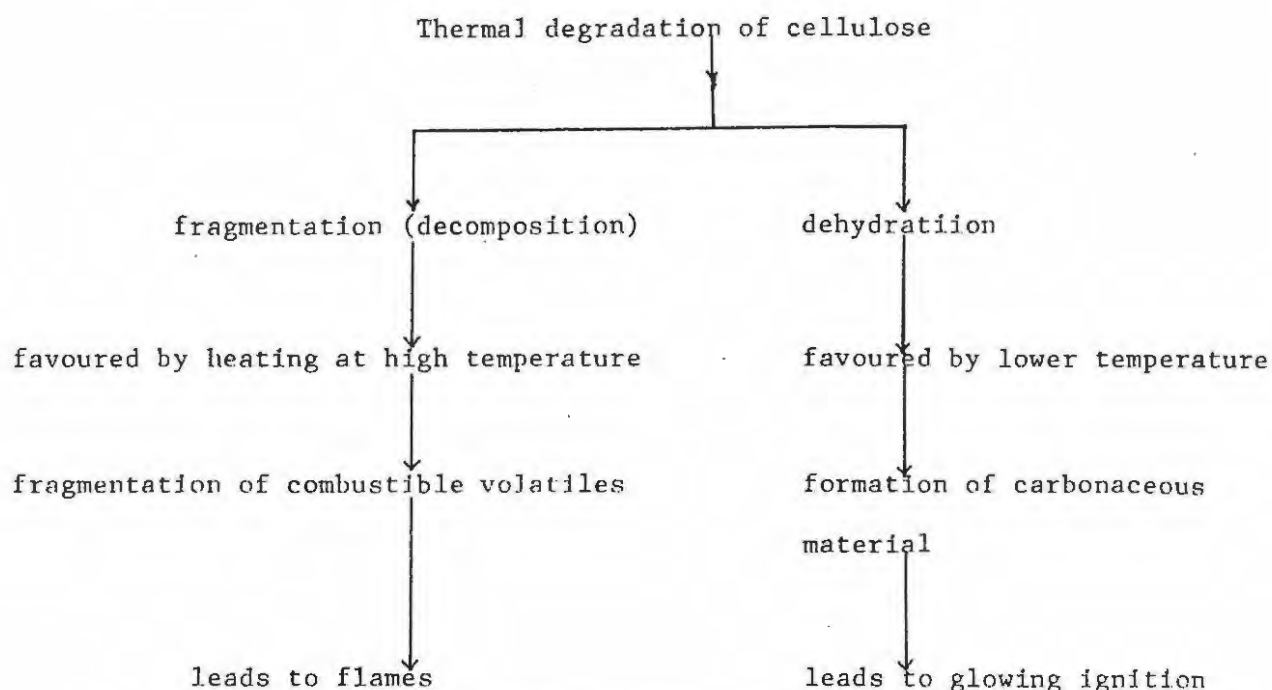
The unbleached CPNF differs from the greige (raw) cotton in that it was washed by water during the patterning process performed by the water jets in the CPNF manufacturing process. The bleached CPNF was further purified by the combined scouring and bleaching process that produces the final fabric. The thermograms in figures 3.1, 3.2 and 3.3 show that the exothermic contribution decreases in going from the greige (raw) cotton to the bleached CPNF. This indicates that there are two processes occurring during the degradation of cotton, one exothermic and the other endothermic.

The pyrolysis of pure cellulose in a nitrogen atmosphere at a heating rate of 10°C/min exhibits a large endotherm at about 392°C (40).

The Absorbent Cotton Wool BPC, used as a control in this study exhibited a large endothermic peak which started at 355°C, with the maximum rate of reaction occurring at 384°C. The pyrolysis of the control sample was completed at about 458°C (see figure 3.10). The SAICCOR pulp used as reference had its endothermic peak at 358°C while its pyrolysis was completed at 390°C (see figure 3.17).

3.4.3 The mechanisms of the thermal degradation of cellulose

There are two pathways that the thermal degradation can follow depending on whether the heating of the cellulose is done at a high temperature or at a low temperature (40). The two pathways can be presented by the following diagram:



Copied from (40)

It is thought that the two reactions perhaps compete with each other at all temperatures, depending on the conditions of the sample and the temperature. The initial degradation reactions include depolymerisation, hydrolysis, oxidation, dehydration and decarboxylation (40). The moisture loss that is observed initially in all the thermograms presumably occurs at random along the chain of glucose units (17). Levoglucosan is the major volatile product of the pyrolysis of cellulose (17, 40). Other pyrolysis products include water, organic acids, aldehydes and ketones as well as gaseous products such as CO , CO_2 , CH_4 and H_2 (40). The residue left after pyrolysis is a solid product which is carbonaceous matter or char.

3.4.4 Factors that influence the thermal degradation of cellulose

The thermal degradation of cellulose is influenced by (40):

1. the nature and period of heating
2. the environmental atmosphere
3. inorganic impurities
4. non-cellulosic ingredients

The first two factors were kept constant throughout this study by using the same heating rate of $10^\circ\text{C}/\text{min}$ and by using nitrogen (500 KPa) at a fixed flow rate of $10\text{ml}/\text{min}$ through out the DSC analysis. This indicates that the last two factors were responsible for the difference observed in the thermograms. Both these factors were investigated in this study. The non-cellulosic content of the cotton fibre is covered in another section of this thesis (see Chapter 4).

The level of inorganic impurities was first determined using the % water-soluble content of the cotton fibre (see Table 3.2). The values obtained range from 2,20% to 2,77% and are of the same magnitude as the level of water-extractable material reported by other workers (41). Water effects the reversal of the decomposition of cellulose from an exothermic reaction in greige (raw) cotton to an endothermic one in washed cotton by removing the soluble inorganic salts in the cotton. Analysis of the dried solids from the water-soluble extractables using Atomic Absorption Spectroscopy to determine the metallic ions removed from the cotton then followed. The analysis showed that metals present in the water-soluble extractables are potassium, magnesium, sodium and calcium in the proportions given in Table 3.3. The anion present in the water-soluble extract were identified as acid phosphate (HPO_4^{2-}) sulphates (SO_4^{2-}) and chlorides (Cl^-) using the quantitative tests described in the experimental section 3.2.5.3. These results were confirmed by the Ion chromatography results (Table 3.4) which showed the presence of additional anions, nitrates and fluoride. The level of the nitrates was so low as not to be detectable by qualitative means (refer section 3.2.5.4).

The determination of the metallic content of the cotton fibre at the stages of the CPNF process that was performed by SAWTRI (see Table 3,5) showed that potassium was a major component of the inorganic content of greige (raw) cotton. These results also show that the potassium content of the cotton decreases sharply in going from the greige (raw) cotton to the final bleached CPNF. Potassium content in cotton is known to be in the form of highly soluble salts (16, 40, 41) and it is therefore not surprising that the contact with water results in such a sharp decrease in the potassium content of the cotton at the different stages of the CPNF process. The fact that the concentration of other elements appears to increase in going from the greige (raw) cotton to the unbleached CPNF could be due to some of the inorganic impurities in the processing water being absorbed by the cotton.

3.4.5 The exothermic effect of some potassium salts on the thermal degradation of bleached cotton CPNF

Experiments were conducted with the phosphate, sulphate and chloride salts of potassium to find out which one had an exothermic effect on the endothermic decomposition of bleached cotton CPNF. Only KH_2PO_4 , K_2HPO_4 , $\text{K}_3\text{PO}_4 \cdot 7\text{H}_2\text{O}$ and KCl were found to have an exothermic effect on the decomposition of the bleached cotton CPNF (see figures 3.5 to 3.9, 3.18, 3.19). The exothermic effect was greater for the higher concentrations of the salt solutions. The profile of the DSC curve of the CPNF samples treated with the K_2HPO_4 , KH_2PO_4 and $\text{K}_3\text{PO}_4 \cdot 7\text{H}_2\text{O}$ solutions was found to be similar to that of greige (raw) cotton, while that of KCl treated CPNF samples was quite different.

Other workers have produced exotherms from purified cotton by adding ionic potassium in the form of the chloride and malate salts (16). They controlled wet weight gain by careful centrifugation before drying the cotton samples for DSC analysis. Their thermogram for KCl added to purified cotton is closer to the profile of the unwashed cotton. This could be due to the fact that centrifuging the cotton samples resulted in a more uniform distribution of the added salt than just soaking the cotton. Potassium malate has been found to be present in cotton (16, 41) but was not determined in this study.

DSC analysis of K_2HPO_4 in the temperature range of interest (ambient to $575^\circ C$) showed that on its own it is thermally inactive in this region. In contrast KH_2PO_4 was found to have strongly endothermic reactions but thermal activity was complete by the time temperatures reached $250^\circ C$. $K_3PO_4 \cdot 7H_2O$ was found to be thermally active as well, with a shallow endothermic peak in the region $305-380^\circ C$. This suggests that the exothermic decomposition of bleached CPNF that has been treated with the phosphate solutions is due to the interaction between cellulose and phosphate anions. The observed exothermic reaction is not a resultant of individual thermal reactions of the anions and the cotton samples.

3.4.6 The Effect of the laboratory treatments on the thermal behaviour of the greige (raw) cotton

As explained earlier, cotton was subjected to various treatments in the laboratory during the course of this study, in an attempt to effect changes in the surface of the cotton fibre. These treatments have already been described in the experimental section and the thermograms of the treated cottons are shown in figures 3.11 to 3.16.

The shape of the DSC curve for solvent-extracted fibre (figure 3.12) is very similar to that of greige (raw) cotton (figure 3.1). The only difference between the two samples is that most of the wax has been removed from the solvent-extracted fibre by extraction with dichloromethane. They both did not have contact with water.

The moisture loss peak of the solvent-extracted cotton occurred at 77°C, while that of the greige (raw) cotton occurred at 75°C. The onset of pyrolysis also occurred at the 325°C, but the exotherm was too shallow for any accurate determination of the pyrolysis peak temperature.

The thermograms for the washed cotton (figure 3.11) and the washed, solvent-extracted cotton (figure 3.13) have very similar characteristics (see Table 3.1). Their reaction temperatures are all lower than those of the purified cotton (Absorbent Cotton Wool BPC). Both these samples confirm that the effect of water on the cotton fibre is to reverse the pyrolysis of greige (raw) cotton from an exothermic reaction to an endothermic one.

The treatment with 2% NaOH solution was used to remove some of the wax on the cotton fibre surface. NaOH is a well-known scouring agent for greige (raw) cotton (18, 41). The moisture loss peak temperature is at 90°C, which shows that the removal of wax by the NaOH treatment makes the fibre more absorbent than the solvent-extraction process does.

The thermogram of the water-soaked fibre (figure 3.1) shows the effect of water alone on the cotton fibre, without any heat or mechanical action. The moisture loss peak occurs at about the same temperature as in the greige (raw) cotton (see Table 3.1) but although small, the pyrolysis peak is endothermic. The soaking treatment has the same effect as the washing. The solvent-soaked cotton sample has a thermogram (figure 3.16) that is similar to that of greige (raw) cotton in figure 3.1.

3.5 CONCLUSION

DSC analysis has shown that the effect of the impinging water jets that pattern the greige (raw) cotton in the CPNF process is to reverse the pyrolysis of cellulose from an exothermic reaction in the greige (raw) cotton to the exothermic one found in unbleached and bleached cotton CPNF. The analysis performed by AA and XEDS on the water-soluble extracts, the greige (raw) cotton, unbleached cotton CPNF and bleached cotton CPNF have confirmed that this reversal is due to the removal of water-soluble inorganic salts. Some of the inorganic salts responsible for the exothermic effect observed in the thermal decomposition of greige (raw) cotton have been identified as potassium phosphate salts (K_2HPO_4 , KH_2PO_4 and $K_3PO_4 \cdot 7H_2O$).

CHAPTER 4 - NON-CELLULOSIC ORGANIC CONTENT OF THE COTTON FIBRE

4.1 INTRODUCTION

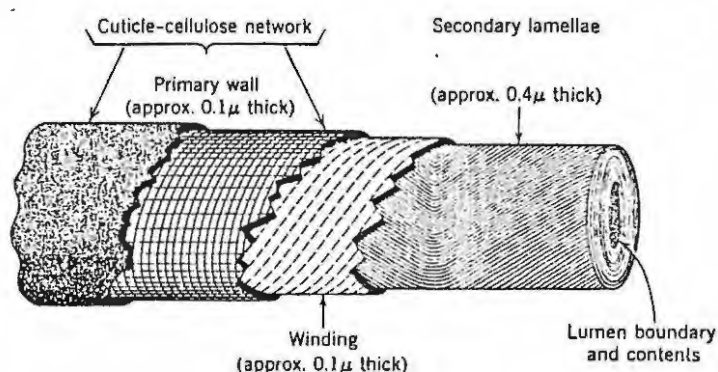
4.1.1 The Structure of the cotton fibre

The cotton fibres used in commerce are the seed hairs of plants belonging in the genus *Gossypium* of the Mallow family (20). The fruiting body or boll cracks open when growth is complete, resulting in a "fluffy, ball-like appearance" of the open boll (20). The cotton fibre is a single, long, cylindrical cell containing some living protoplasm in the cell liquid when the bolls crack open (20). The open bolls are picked and sent to ginneries for cleaning to remove seeds, and foreign matter.

The dried cotton fibre is in the form of a flattened cylindrical ribbon with many twists along its length (20,32). The collapse of the cylindrical structure is due to the loss of liquid protoplasm in the centre of the fibre. The places at which the twists occur are known as convolutions (20,42). The cross section of the mature cotton fibre before drying consists of six parts (20,42):

1. the cuticle
2. the primary wall
3. the winding layer
4. the secondary layer
5. the lumen wall
6. the lumen

Fig 4.1 - Schematic diagram of cotton fibre structure (Copied from (20)).



The above diagram illustrates the concentric arrangement of the six layers. Of interest to this study are the first two layers which are the cuticle and the primary wall. These contain the non-cellulosic substances (20,41,43). The non-cellulosic constituents that are of concern to us are the natural products that are formed in the normal growth process of the cotton fibre.

The non-cellulosic organic content is present as pectins, proteins and cotton waxes. The cuticle is mostly a waxy layer but it may also contain pectins and proteins (20,44). The primary wall is mostly cellulose but it is both covered by and impregnated with the wax, pectins and proteins found in the cuticle (20).

The % composition of the natural non-cellulosic constituents is typically in the following quantities on a dry-weight basis (45)

proteins (nitrogen x 6,25)	1,3%
pectic substances	0,9%
ash	1,2%
wax	0,6%
organic acids	0,8%
sugars	0,3%
others	0,9%

4.1.2 Pectins and proteins in the two outer layers of the cotton fibre

The name pectin is used to describe the complete class of pectin substances which contain chain molecules of d-galacturonic acid as the primary constituent. They often occur as insoluble calcium, magnesium or iron salts as well as soluble sodium, potassium and ammonium salts (43). The pectin substances can be subdivided into protopectins, pectins and pectin substances. Although water alone does not remove all the pectins, their degree of extraction in neutral water varies. Pectins are practically entirely removed by the dilute caustic solutions used kier boiling (20,43). It has been shown that the removal of pectins from cotton fibres affects neither the tensile strength nor the cellulose fluidity in cuprammonium hydroxide (20).

The proteins found in the cotton fibre are assumed to be residual protoplasm left in the lumen after the fibre has dried (20,43). Very little is known about the composition and properties of the protein found in the cotton fibre, but protein amino acids as well as free amino acids have been discovered in the cotton fibre and in its primary wall preparations (41). It has been found that boiling the cotton fibre in dilute sodium hydroxide solutions reduces the nitrogen content significantly. It is deduced from this fact that this treatment also reduces the protein content of the cotton (20,41).

On the basis of the information obtained on the pectins and proteins in the cotton fibre, they were not seen as important when considering its surface properties. Thus an in-depth chemical analysis of these pectins and proteins was not part of this study.

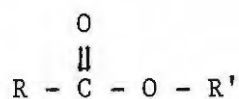
4.2 COTTON WAXES OF THE OUTER TWO LAYERS

Perhaps the most important component of the outer two layers of the cotton fibre is the wax. It is responsible for a number of attributes which affect the processing of the cotton fibre (6,20).

- (1) it lubricates the fibre surface
- (2) it decreases the tendency of the fibres to cling together
- (3) it reduces the fibre-fibre friction
- (4) it lowers the tensile strength of yarn and fabric
- (5) it affects the wetting behaviour

The fibre characteristics of cotton in the lint form vary according to the cultivar and variety, growing area, season and conditions, time, method of harvesting and ginning conditions (6,45). A similar variation in wax content and components is to be expected. Other greige cotton samples were evaluated together with the greige cotton as a starting point. These samples were of different cultivars, manufacturers and growing areas.

A wax is defined as an ester of a long chain alcohol and a long chain fatty acid, with the structure (46):



Analyses of cotton wax have shown that it contains all the even-numbered carbon primary alcohols, with n-triacontanol ($\text{C}_{30}\text{H}_{61}\text{OH}$) the alcohol present in the largest amounts. It also contains all the even-numbered fatty acids from C_{24} to C_{34} , with n-tetracosanoic acid ($\text{CH}_{24}\text{H}_{48}\text{O}_2$) the fatty acid present in the largest amounts, (20,43). Other substances which have been found in cotton wax include glycerol, resinous materials, amyirin ($\text{C}_{30}\text{H}_{50}\text{O}$) and hydrocarbons with probably heptacoxane among them (43).

The non-cellulosic material that can be extracted from cotton fibres with organic solvents such as chloroform, carbon tetrachloride, benzene, dichloromethane and other organic solvents is classified as wax (6,20,21). Dilute sodium hydroxide solutions can also remove some of the wax from the fibre (6,14,20,21). Cotton wax is classified as a plant cuticle wax (20). The wax content of mature cotton varies from 0,4 to 1,35% with 0,5% being a typical value (6,20).

The residual wax content after bleaching by either the hydrogen peroxide method or the hypochlorite method is in the range 0,3 to 0,5% (20). For cotton CPNF, the maximum permissible wax content after bleaching is 0,3%.

The % cotton wax was determined at the three stages of the CPNF process in order to determine,

- (1) the % wax content of the cotton fibre at the different stages of the process
- (2) whether there was any change in the composition of the wax from the different stages of the process

Cotton wax was obtained by evaporating the dichloromethane extract from the fibre samples to dryness. It was analysed by Infrared (IR) analysis to characterize the wax samples from the different stages to as to be able to detect changes in the components of the wax. Differential Scanning Calorimetry (DSC) was used to determine the melting points and % composition (using peak areas) of the wax samples. The cotton wax was also analysed by a Gas Chromatograph - Mass Spectrometer (GC-MS) in an attempt to identify its components.

4.3 EXPERIMENTAL

4.3.1 Materials

The greige (raw) cotton was sampled from a single batch of cotton fibre approved for CPNF production on a manufacturing line.

This is the same cotton used throughout this investigation. The source of the cotton cannot be revealed because it is proprietary information. Samples were taken from the greige (raw) cotton, unbleached CPNF and scoured and bleached CPNF stages of four batches.

4.3.2 Instruments

The IR analysis was performed on a Beckman Acculab 10 IR spectrophotometer using NaCl windows. The samples were prepared as thin melted films.

DSC analysis was performed on the DuPont 990 Thermal Analyser with a DSC cell using open Aluminium sample pans. Nitrogen set at 500 KPa and with a flow rate of 10ml/min was used as a purge gas. The GC-MS analysis was performed by Mr Aubrey Sonneman of the Chemistry Department, Rhodes University, using a Hewlett-Packard instrument.

4.3.3 Solvent-Extraction of the cotton fibre

The solvent-extraction was performed according to a British Standards test method (21) using analytical grade dichloromethane (Merck). The extractions were performed on 5g samples that had previously been dried to a constant weight in a convection oven set at 110°C. The % extractables or wax content was determined by evaporating the solvent-extract to dryness and calculating it as a percentage of the mass of the dried sample.

4.4 RESULTS

Table 4.1 - % Cotton wax of cotton fibre samples (based on dry fibre mass)

CULTIVAR	GROWTH AREA	% COTTON WAX
Acala	Barberton	0,49
Acala 1517	Jan Kempdorp	0,72
Rebel	Jan Kempdorp	0,44
Turbo	Zimbabwe	0,47
Tongaat Albacala	Swaziland	0,45
Orange Kook	Uppington	0,36
Greige (raw) cotton	Unknown	0,62
		0,58
		0,61
		0,52
		0,55

Table 2.1 - % Cotton wax of cotton fibre at the 3 CPNF stages

BATCH NO	RAW COTTON	UNBLEACHED CPF	SCOURED AND BLEACHED CPF
794	0,53	0,53	0,21
	0,51	0,52	0,31
	0,53	0,38	0,32
	0,59	0,45	0,45
834	0,52	0,50	0,35
	0,52	0,52	0,29
	0,55	0,48	0,37
	0,57	0,52	0,32

Table 4.1 continued

812	0,76	0,53	0,10
	0,67	0,53	0,1
	0,61	0,41	0,3
	0,62	0,47	0,2
815	0,53	0,53	0,1
	0,57	0,48	0,2
	0,52	0,56	0,2
	0,66	0,53	0,2

Table 4.3 - DSC results of wax from random raw cotton samples

SAMPLE	GROWTH AREA	NO OF MELTING PEAKS	PEAK	% PEAK
			TEMP	AREAS
Deal A Acalar	Barberton	2	57,8°C	25,9
			70,8°C	74,1
Acala 1517	Jan Kempdorp	2	54,0°C	20,2
			74,8°C	79,8
Deal A2 Rebel	Jan Kempdorp	2	55,0°C	22,4
			76,0°C	77,6
Turbo	Zimbabwe	2	54,5°C	17,5
			75,6°C	82,5
Tongaat Albacala	Swaziland	2	58,0°C	24,5
			74,6°C	75,5

Table 4.3 continued

Orange Kook	Uppington	5	37,0°C	0,2
			53,8°C	0,5
			58,0°C	3,7
			66,5°C	0,4
			74,0°C	90,4
Greige (raw) cotton	-	4	37,0°C	0,2
			56,4°C	9,1
			74,0°C	90,5
			83,0°C	0,2

Table 4.4 - DSC results of wax from the mixed batches of CPNF samples

SAMPLE	NO OF MELTING PEAKS	PEAK TEMPERATURES			% PEAK AREA		
		°C					
Batch 812	3	38,0	36,5	36,0	7,9	4,6	7,1
Greige (raw)		53,8	54,0	51,5	17,1	13,1	15,4
cotton		68,5	71,5	68,0	75	82,3	77,5
Batch 812	3	33,5	33,0	absent	2,0	4,0	absent
Unbleached CPNF		54,9	53,5	59,0	17,2	20,9	42,0
		75,7	75,8	76,0	80,8	74,7	58,0
Batch 815	3	38,0	33,0	36,0	13,4	3,7	8,3
Greige (raw)		54,0	53,6	53,6	15,8	14,4	13,2
cotton		68,6	76,8	70,5	70,8	81,9	78,5

Table 3.4 continued

Batch 815	3	36	36	1,1	2,2
Unbleached CPNF		55,5	54,5	17,7	16,4
		75,5	75,6	81,2	81,4

Figure 4.2 - DSC thermogram of cotton were extracted from greige (raw) cotton

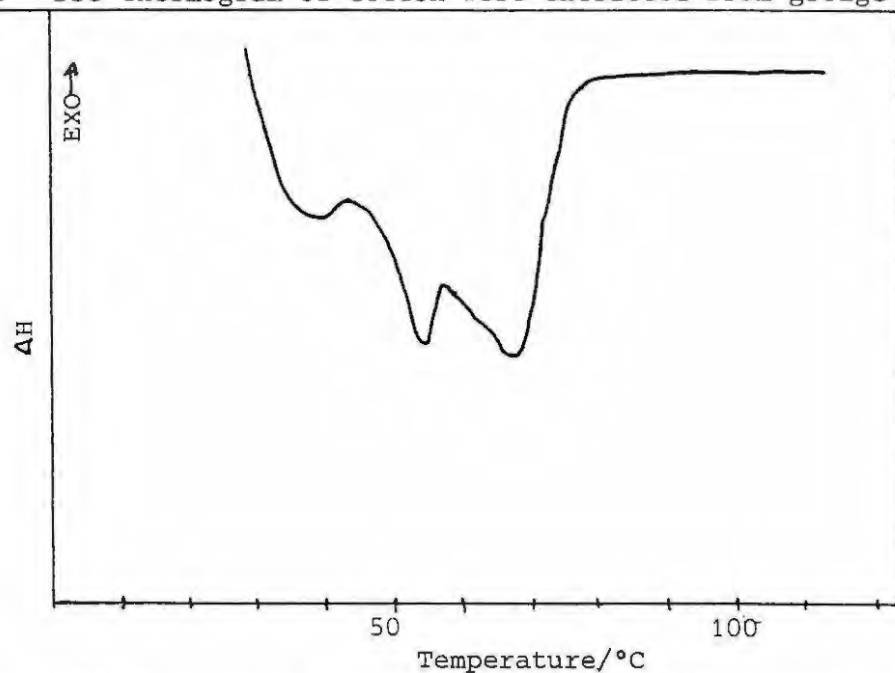


Figure 4.3 - DSC thermogram of cotton wax extracted from unbleached CPNF

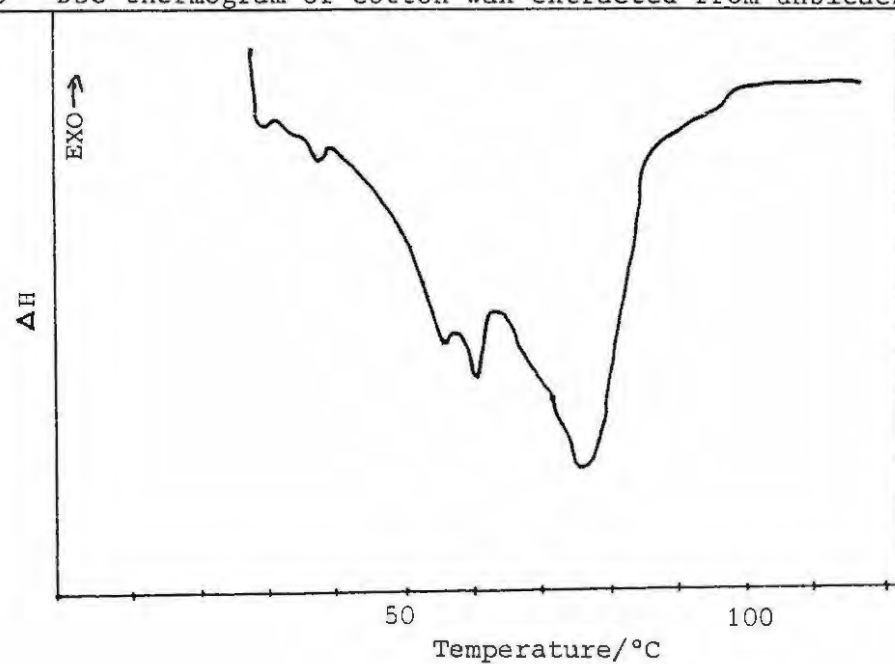


Figure 4.4 - IR graph of cotton wax extracted from greige (raw) cotton

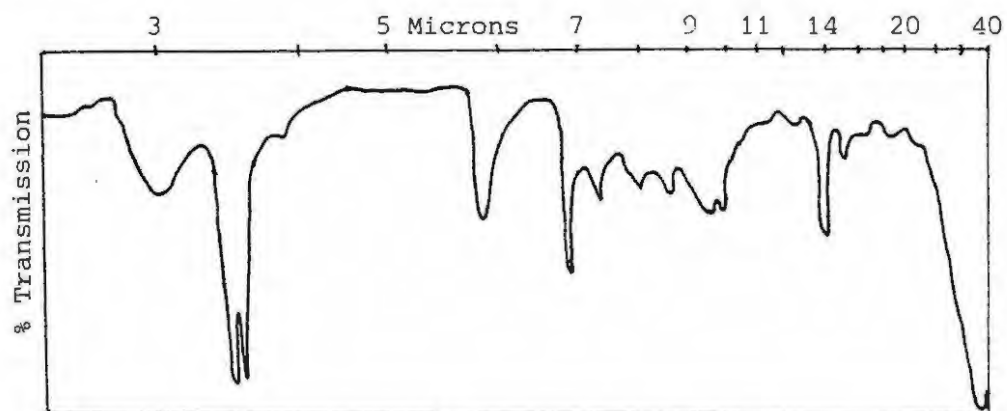


Figure 4.5 - IR graph of cotton wax extracted from unbleached CPNF

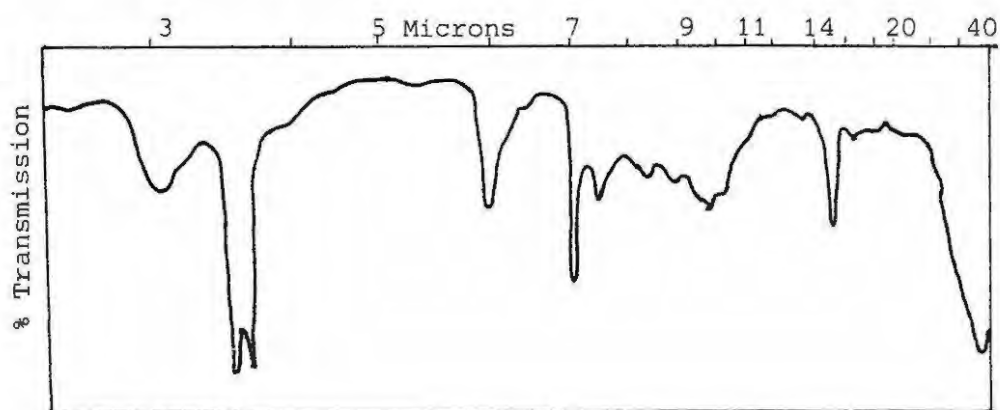
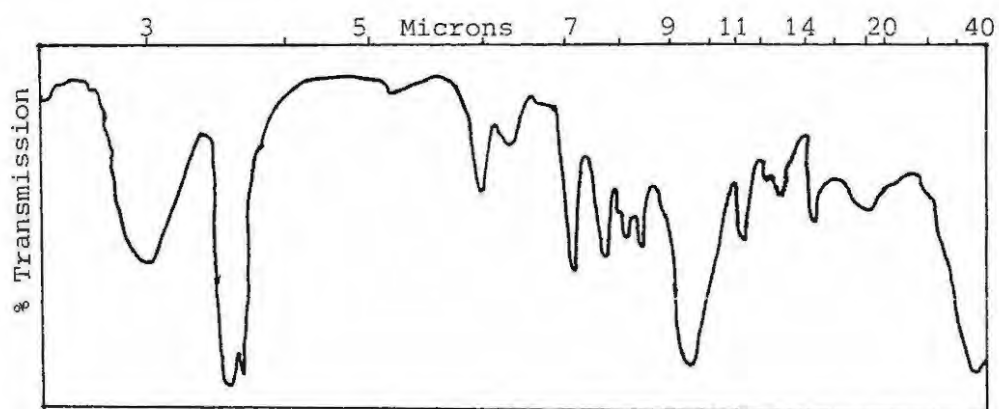


Figure 4.6 - IR graph of cotton wax extracted from bleached CPNF



4.5 DISCUSSION

4.5.1 Cotton Wax content

The wax content values of the randomly selected samples given in Table 4.1 show that their wax content ranges from 0,36% to 0,72%. This variation in the results is due to the differences in cultivars and also possibly due to differences in growth areas. The wax content of the greige (raw) cotton samples is close to the value (0,6%) said to be typical of mature cotton (6,20) (see Table 4.1). As expected, the wax content of the finished product i.e. bleached CPNF (see Table 4.2) is often lower than that of the greige (raw) cotton.

This is due to the effects of the combined scouring and bleaching process that reduces the amount of wax on the cotton fibre surface. The chemicals used include sodium hydroxide and hydrogen peroxide. Sodium hydroxide is a well-known scouring agent that is very effective in the removal of wax (20,37,41). Hydrogen peroxide is a common bleaching agent (41,45). A surfactant that acts as a wetting agent is also one of the chemicals.

4.5.2 DSC Analysis of the cotton wax

DSC analysis revealed that a common feature of all the cotton wax samples from the different cotton samples is two major endotherms that have peaks in the 53-58°C range and in the 70-77°C (see Tables 4.3 & 4.4).

Of the randomly selected samples (Table 4.1) only samples 2 and 6 had other melting endotherms in addition to these. The additional peaks were very small and were only detectable at higher instrument sensitivities.

The mixed raw cotton samples from the different batches (see Table 4.4) also exhibit the two major components whose peaks are noted in the 52-56°C region and in the 68-76°C region (see figures 4.2 and 4.3). The other peak is found in the region 33-38°C and is a minor peak. The peak occurring around 37°C was also noted in random sample no 6.

The major component of the cotton wax is the one which melts in the 68-76°C range. It is present in percentages ranging from 75% to 81,4%. The component which melts in the 52-56°C range is present in quantities which range from 13% to 21%. The minor peak in the 33-38°C region is present in quantities which range from 1% to 8%. One of the wax samples from the unbleached CPNF did not have this component. The other two components were found in nearly equal quantities in this sample.

The wax from the raw cotton and unbleached CPNF was a light brown hard tacky wax. The residue obtained from the bleached CPNF was a light brown oily residue that could not be analysed on the DSC in the temperature (ambient to 100°C) that was selected. Attempts to analyse it simply gave a flat sloping base with no discernible peaks.

It is assumed that the solvent-extract from the bleached fabric is contaminated by the surfactants and other additives that are used in the bleaching and scouring process.

The melting points observed with the cotton wax samples fall within the region defined by the overlap of the reported melting points which are in the (68-90°C) region (20,46).

In fact the two major endotherms with peaks at 52-56°C and 68-76°C would be detected as a single melting point probably at round 70°C using standard laboratory apparatus .

4.5.3 IR Analysis of cotton wax

The samples were analysed in the 2,5 - 40 micron range (see figures 4,4 to 4,6). The major absorption bands occur at wavelengths near 3; 3,5; 5,8; 6,3; 6,9; 7,3; 7,9; 8,9; 9,3 and 20 microns in the greige (raw) cotton (figure 4,4) and unbleached CPNF (figure 4.5) samples. The band near 3 microns is due to oxygen-hydrogen linkages (O-H) and the band near 5,8 microns is due to the carbon-oxygen double band (C=O). The bands at 3,5 microns, 6,9 and 7,2 microns are due to carbon-hydrogen linkages. The bands at 8,9 and 9,3 microns are due to the carbon-oxygen single bonds (C-O) found in alcohols, esters and ethers. The results of this analysis confirm the presence of the functional groups of alcohols, esters, carboxylic acids and possibly ethers. This is in agreement with information obtained from literature (20,47).

The fact that the spectrum of cotton wax extracted from greige (raw) cotton is virtually identical to the IR spectrum of the cotton wax extracted from the unbleached implies that reduced % wax in unbleached CPNF is simply due to a decrease in the wax content, and not due to loss of a particular component.

The IR spectrum of the residue from the solvent-extraction of the bleached CPNF (figure 4.5) shows some additional absorption bands that were absent in the spectra of the previous absorption bands of the previous two stages. This was to be expected because its appearance was different. The additional bands are near 10,5 and 11,7 microns and assumed to be due to the residual chemicals from the scouring and bleaching process. It is most likely that the surfactant is responsible for these bands.

4.5.4 GC-MS Analysis of the cotton wax

The GC-MS analysis revealed 3 peaks at mass numbers 256, 392 and 509. These are equivalent to C_{16} , C_{28} and C_{34} respectively. They are identified as (48).

1. C_{16} : Palmitic Acid $C_{16}H_{32}O_2$
mp $63^{\circ}C$ mol. wt 256,43

2. C_{28} : Octacosane $CH_3(CH_2)_{26}CH_3$
mp $64,5^{\circ}C$ mol.wt 394

3. C_{34} : Cetyl Stearate $C_{17}H_{35}CO_2C_{16}H_{33}$

mp $57^{\circ}C$ mol. wt 508,91

4.6 CONCLUSION

Solvent-extraction analysis showed that the % cotton wax of the greige (raw) cotton used in this study is in the range 0,5 to 0,7%. The % cotton wax of the unbleached CPNF was also in the same range. This shows that the patterning process does not remove significant quantities of cotton wax from the cotton fibre surface. The IR analysis confirmed these findings because the IR graphs of the cotton wax from greige (raw) cotton and that from the unbleached fabric were identical.

The % cotton wax found in the bleached CPNF is in the range of 0,1 to 0,4% and is lower than % cotton wax in the greige (raw) cotton. This confirms that the scouring and bleaching process removes some of the wax from the cotton fibre.

Using DSC analysis, 3 components were detected in the cotton wax extracted from greige (raw) cotton. These results are supported by the results of the GC-MS analysis which identified three components to be present in the cotton was. These are:

1. Palmitic acid $C_{16}H_{32}O_2$

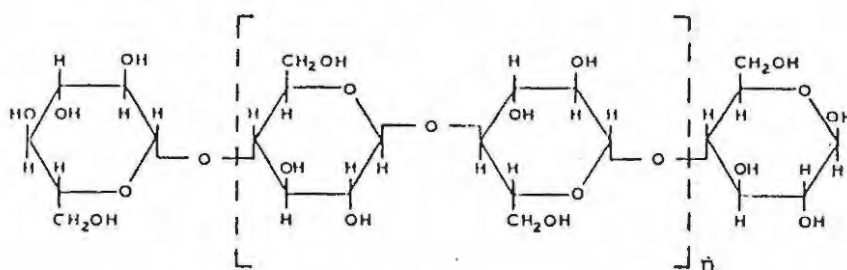
2. Octacosane $C_{28}H_{58}$

3. Cetyl Stearate $C_{34}H_{68}O_2$

CHAPTER 5 - THE DEGREE OF DEGRADATION OF THE COTTON FIBRE

5.1 INTRODUCTION

The cotton fibre is at least 85% cellulose (49), and is a polysaccharide made up of β -D(+) glucose molecules joined in linear chains (50). The accepted structural formula for cellulose is (49):



n is typically of the order of 10^4 in native cellulose, which corresponds to a molecular weight of 1590 000 (49). A typical chain length is 5×10^{-6} m (12).

Cellulose fluidity is regarded as a sensitive means of measuring the degradation of cotton or regenerated cellulose rayon yarns and textiles (18). The degradation of cotton can be caused by both mechanical and chemical damage (35). The causes of degradation in cotton include the action of heat, light, acids, alkalis, oxidising agents, reducing agents, processing conditions and laundering effects (18,51). The extent of the shortening of the cellulose chain or the reduction in the degree of polymerization depends upon the severity of the treatment that the cellulose fibres have been subjected to (18).

Solutions of long chain molecules show an enhanced viscosity compared to the solvent itself, depending on, among other factors, the chain length of the polymer (50). By definition, cellulose fluidity is the reciprocal of the viscosity of a dispersion of cellulose in a suitable solvent, expressed in c.g.s. units ie poises (49,50,52). Standard methods for cotton use either cuprammonium hydroxide or cupriethylene diamine hydroxide. The viscometers are the glass-pipette type with a fixed capillary at the end. They are described in detail in the individual standard methods (49,50).

In this study, the cellulose fluidity of cotton samples from the three stages of the CPNF process have been determined. That is, samples from the initial stage of greige (raw) cotton as well as those from the intermediate stage of unbleached entangled fibre and the final stage of scoured and bleached fabric have been analysed. This was done in order to determine whether the patterning process induced any mechanical damage in the cotton fibre.

Commercial processes usually produce fabrics that give fluidities between 4 and 5 (49). Scouring does not damage cotton fibres (49). Unless a material is exceptionally difficult to bleach or extreme whiteness is required, it should be possible to maintain fluidities within this range (52). Normal bleaching processes only degrade the cotton to a moderate extent (52,53). The fluidity of cotton bleached with sodium hypochlorite is normally not expected to exceed 8. A fluidity exceeding 8 is undesirable for commercially bleached cotton (19,49).

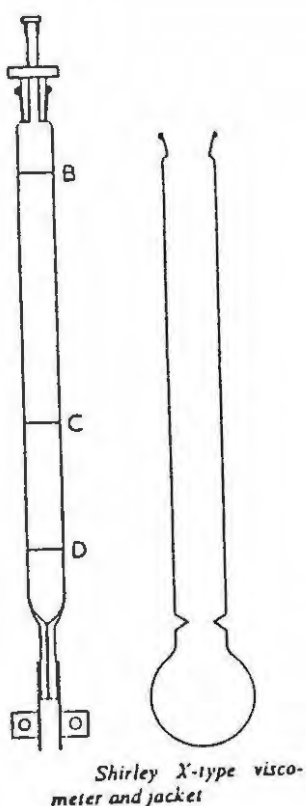
According to literature, the determination of cellulose fluidity of raw cotton gives unsatisfactory results since it contains at least 7% of non-cellulosic material (18,19,53).

In this study, any error that is introduced to the measurements by the presence of impurities such as waxes and other non-cellulosic constituents will be common to both the greige (raw) cotton samples and the unbleached patterned fabric samples. Normal values of fluidity for undamaged cotton are between 3 and 5 (19,49).

5.2 THE THEORY OF THE DETERMINATION OF CELLULOSE FLUIDITY

According to the British Standards method (50) the fluidity of a specimen of cotton is the fluidity of a solution in the standard cuprammonium solvent containing 0,5g of cotton per 100ml of solution at 2C. A weighed sample of the specimen is dissolved in a standard capillary viscometer of the type shown in figure 5.1 (50):

Fig 5.1 - Shirley X-type viscometer and jacket, copied from (19)



The solution is allowed to flow from the viscometer through the capillary after the viscometer tube and its contents have been brought to a temperature of 20°C in a thermostat. The time taken by the meniscus to fall from one fixed mark to another (B to D in above figure) is recorded as the rate of flow (9,18,19).

To calculate the fluidity, two constants first have to be determined by calibrating the viscometer tube with a liquid of known viscosity. A suitable calibration liquid is an aqueous solution containing approximately 65% by weight of commercially pure glycerol. The kinematic viscosity of this solution is approximately 8 at 20°C and is determined in a No 2 U-tube viscometer (BS188). From this measurement, the kinetic energy correction factor can be calculated from the formula (52),

$$K = 0,131 \text{ vt} \dots\dots (1)$$

where V is the volume between the top and bottom timing marks B and D and t is the time of flow of the glycerol solution.

The viscometer constant, C, can then be calculated by substituting relevant values in the formula,

$$C = F d (t - k/t) \dots\dots (2)$$

Where t is the time of flow of the glycerol solution

Fd is the kinematic fluidity given by the product of the fluidity of the glycerol solution and its density

K is the kinetic energy correction factor calculated from equation (1)

Commercially pure phenyl ethyl alcohol, further purified by distillation can also be used as a calibrating fluid. It has a kinematic fluidity of 7,14 at 20°C (52). The kinetic energy correction factory in this case is then given by the formula

$$K = 0,117 Vt \quad \dots\dots (3)$$

where t is the time of flow of phenyl ethyl alcohol and the other symbols carry the same meaning as in equation (1).

The viscometer constant, C , will still be calculated from equation (2) but t and F_d will apply to the phenyl ethyl alcohol solution (52).

To calculate the fluidity for dilute solutions of cellulose in the standard cuprammonium solvent, the density of the cellulose solution is taken as constant and equal to that of the solvent which is 0,94. This density is then substituted in equation (2) to give the fluidity, F

$$F = (C / d) / (t-k/t)$$

where $C' = C/0,94$

This equation gives the fluidity in reciprocal poises.

5.3 EXPERIMENTAL

Samples of greige (raw) cotton from 3 different batches together with corresponding samples of the unbleached CPNF and the bleached CPNF were analysed by the Textile Testing Services Division of the SABS. The fluidity of the samples was determined using cuprammonium hydroxide as a solvent (See appendix 9 for SABS test method).

5.4 RESULTS

Table 5.1 - Cellulose fluidity results obtained from the SABS

<u>Sample</u>	<u>Cellulose Fluidity</u>
801A (greige cotton)	2
801B (unbleached cotton CPNF)	2
801C (bleached cotton CPNF)	6
803A (greige cotton)	2
803B (unbleached cotton CPNF)	2
803C (bleached cotton CPNF)	5
815A (greige cotton)	2
815B (unbleached cotton CPNF)	2
815C (bleached cotton CPNF)	5

5.5 DISCUSSION

The fluidity of both the raw cotton and the unbleached CPNF samples is 2, while that of the bleached cotton CPNF samples is either 5 or 6. The results obtained indicate that the CPNF process does not damage the cotton fibre, because cellulose fluidities of undamaged cotton are normally between 3 and 5 (19,49).

It is concluded that although the cellulose fluidity below 5 results are not highly accurate (according to the SABS), the results obtained in this study are enough to indicate that no mechanical degradation occurred in the cotton fibres as a result of the patterning process in producing the CPNF.

CHAPTER 6 - THE COTTON FIBRE FRICTION

6.1 INTRODUCTION

6.1.1 Definition of Friction

Friction is defined as the resistance to motion that develops when one body slides over the surface of another (43). The waxy surface of the cotton fibre has already been described in Chapter 4, while the wrinkled appearance of its surface and the formations of convolutions along the fibre length were discussed in Chapter 2. These features, together with the cross-sectional shape and overall fibre size are the intrinsic surface factors which combine with the external structure to determine the frictional behaviour of fibres (43).

In the manufacture of nonwoven fabrics there is fibre to fibre friction as well as fibre to metal friction to consider (23). A high fibre to fibre friction is needed to provide a cohesion that facilitates the bonding process, while a low fibre to metal friction is desirable in order to card a stable web (23). The bonding process in nonwovens is essential for the integrity of the structure because the forming fibres lack the inherent strength provided by the frictional forces between yarns in woven and knitted structures (54).

Friction can be classified into two types, static friction which opposes the displacement of a body at rest against a second surface, and kinetic friction which represents the forces which oppose the forces producing uniform motion. Both quantities can be described quantitatively by using the basic form of Amonton's Law (43,55),

$$\mu = \frac{F}{N}$$

Where μ is the coefficient of friction (COF)

F is the frictional force acting parallel to the surfaces
in contact

N is the normal load between the object and the restraining
surface

μ_s is known as the coefficient of static motion is applicable
where F is the force just necessary to start motion of the
object. μ_k is known as the coefficient of kinematic friction
and is applicable when the object is in motion (55,56).

Amonton's Law assumes that frictional force is independent of the
contact area. Although this assumption is true on a macroscopic
scale, strong deviations are observed at a microscopic level i.e.
in fibre to fibre interaction (25).

6.1.2 Variables affecting fibre friction

The COF for a fibre is very dependent on the conditions under
which the measurements are made (25,55). The most important
factors related to these conditions are (55,56):

1. the area and geometry of the fibre surface

2. the nature of the fibre surface
3. the moisture absorbed

For example the COF is directly proportional to the area of contact as well as the absorbed moisture for different fibres (56). The area of contact between the fibre surface is dependent on the load and time of contact (in polymers) as well as on fibre diameter and fibre tension (25).

Other factors which affect fibre friction are humidity, temperature, history of the fibre, processing and interfibre velocity (25). Thus, in order to measure friction data and to compare data from different sources, it is essential that these factors are specified (25). The method of data calculation must also be similar. Amonton's Law is not obeyed for fibres because the ratio of frictional force F to normal load N is influenced by changes in the area of contact during measurements. The most successful relationship that has been found to fit experimental friction data is $F = an$ where a and n are constants.

6.2 EXPERIMENTAL

6.2.1 Fibre to metal friction

The following types of cotton fibres were tested by the Shirley Institute, England, for testing:

1. Greige (raw) Cotton

2. Unbleached CPNF (water-washed patterned fabric)
3. Scoured and bleached CPNF i.e. the final product
4. Scoured and bleached cotton fibre
5. Solvent-extracted fibre

Samples 1, 2 and 3 were sampled from the three different stages of the same batch of CPNF. Sample 4 was fibre that had been scoured and bleached according to the bleaching process already described in Chapter 2. The solvent-extracted fibre was prepared as described in Chapter 3. The samples were tested by the Shirley Institute according to the method described in the Shirley Institute Bulletin Volume 53 No 2 page 33 (see appendix 10), with apparatus that has been completed redesigned and rebuilt incorporating a microprocessor. The results are given in Table 6.1 and plotted in figure 6.2.

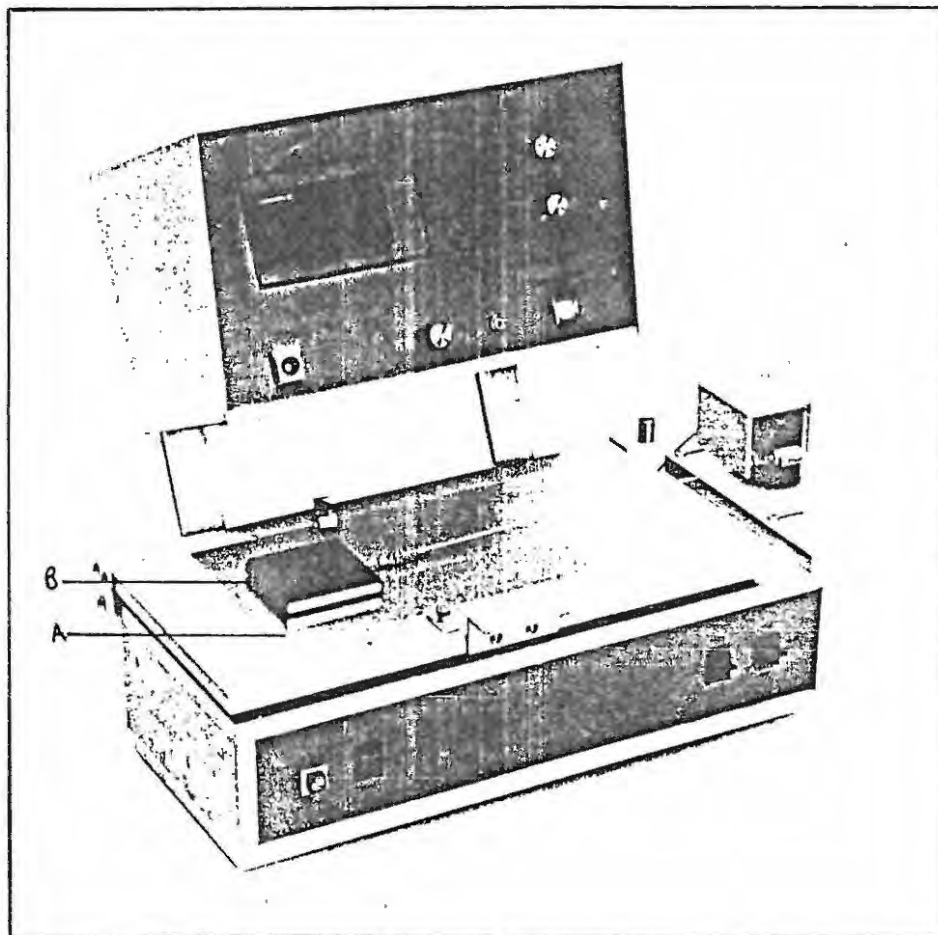
6.2.2 Fabric to fabric friction

The following fabrics were tested:

1. unbleached CPNF
2. scoured and bleached CPNF
3. experimental fabric made with Enka Bico fibre
4. experimental fabric made with Absorbent cotton wool BPC

5. experimental fabric made with greige (raw) cotton i.e. equivalent to unbleached CPNF
6. experimental fabric made with rayon fibre

Fig 6.1 - The Slip/Peel Tester used in this work



The Instruments Slip/Peel Tester Model SP105A shown in fig 6.1 was used to measure fabric to fabric friction in this investigation. This instrument is designed to measure coefficients of friction between surfaces of any type of material in accordance with the standard test method described by ASTM D-1894. (see appendix 11).

To measure the COF, both the platen (marked A) and the sled (marked B) were covered with the test fabric. A test speed of 6in/min was selected for the platen and an integrating time of 10 sec was used for the electronic readout. The instrument integrates force with respect to time, and it determines the COF as the tractive force divided by the normal force. The COF is given directly by the instrument meter.

The static COF (μ_s) is the peak of the COF that is electronically detected and stored in the instrument memory to be recalled when required. The kinematic COF (μ_k) is the average value of the COF and is obtained at the end of the run.

6.3 RESULTS

Table 6.1 - The COF results for fibre to metal friction (Shirley
Instutite). S.D. is the standard deviation of the results.

Sample	Speed	Frictional Coeff	S.D.
	m/min	μ	
Greige (raw) Cotton	1.0	.141	.021
	3.0	.153	.022
	6.0	.161	.020
	10.2	.168	.018
	13.8	.192	.017
	30.0	.232	.019
	90.0	.258	.016
	180.0	.273	.016
	300.0	.284	.018
Bleached Cotton	1.0	.161	.023
	3.0	.175	.022
	6.0	.191	.022
	10.2	.197	.026
	13.8	.210	.024
	30.0	.234	.023
	90.0	.265	.016
	180.0	.274	.018
	300	.279	.018

Table 6.1 cont.

Solvent-	1.0	.174	.022
extracted	3.0	.184	.022
fibre	6.0	.202	.022
	10.2	.212	.023
	13.8	.231	.026
	30.0	.260	.023
	90.0	.287	.022
	180.0	.295	.018
	300.0	-	-
Scoured and	1.0	.194	.018
bleached CPNF	3.0	.206	.016
(final product)	6.0	.223	.020
	10.0	.236	.027
	13.8	.243	.025
	30.0	.264	.016
	90.0	.284	.014
	180.0	.298	.016
	300.0	.307	.010
Unbleached CPNF	1.0	.204	.034
(water-washed	3.0	.209	.041
patterned	6.0	.226	.040
fabric)	10.2	.238	.043
	13.8	.250	.034
	30.0	.286	.030
	90.0	.320	.048
	180.0	.332	.034
	300.0	.349	.049

Fig. 6.2 - Graphical representation of the results in Table 6.1

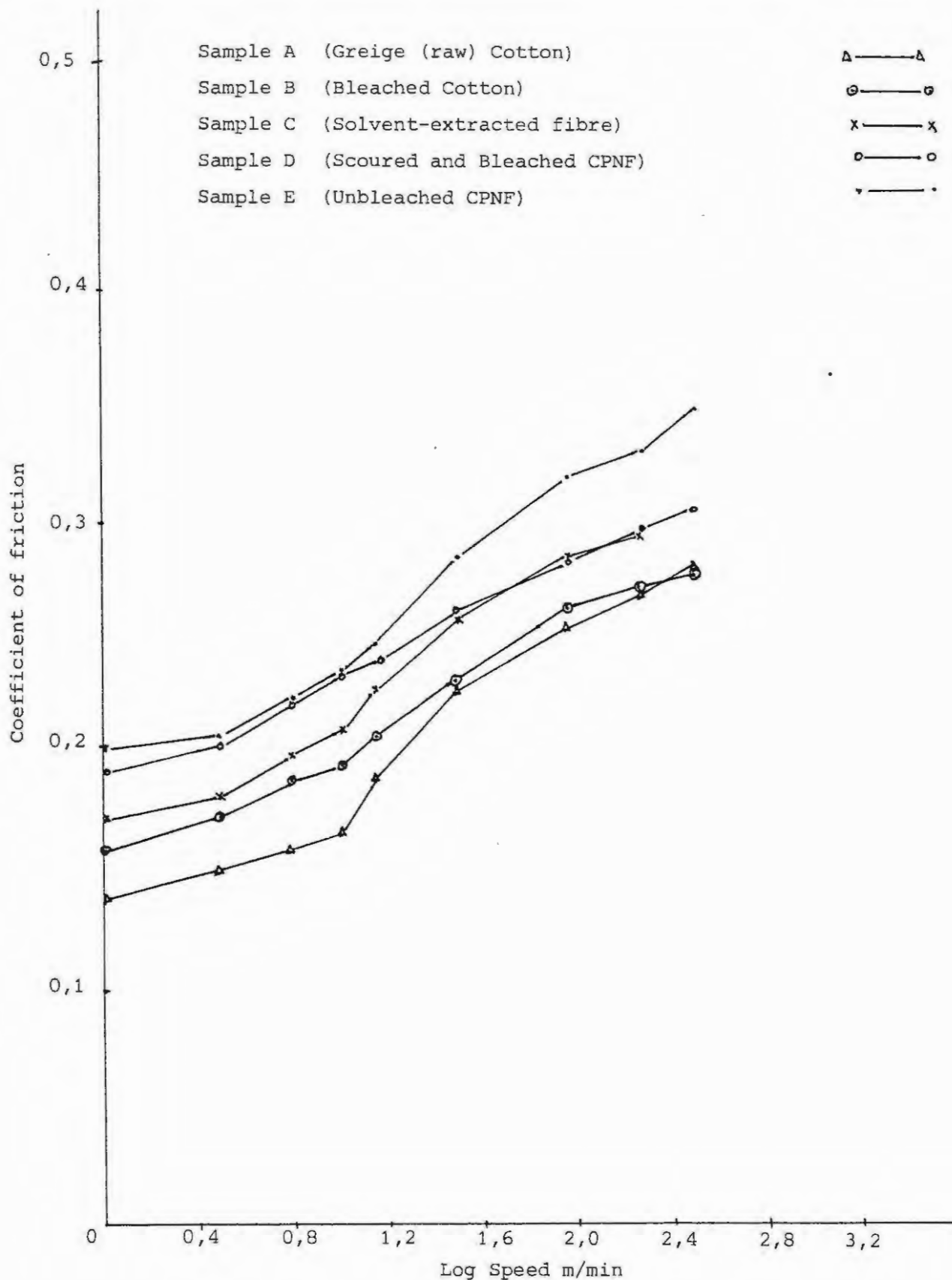


Table 6.2 - The COF results for Fabric to fabric friction (Slip/Peel Tester). The percentages given in brackets denote the relative mean deviation (RMD) of 5 readings.

Sample	Static COF	Kinematic COF
	μ_s	μ_k
Enka fabric	0,760	0,490
	(4,6%)	(3.3%)
Rayon fabric* (20 mins)	0,906	0,504
	(8,6%)	(6,8%)
Rayon fabric	1,075	0,674
	(14,8%)	(13.1%)
Greige (Raw) Cotton fabric	1,186	0,990
	(8,7%)	(4,8%)
Absorbent Cotton wool	1,327	1,097
BPC fabric	(4,7%)	(6,8%)

* The time given for the rayon fabric refers to patterning times used to make the fabric. All the other fabrics were made using an entangling time of 10 minutes.

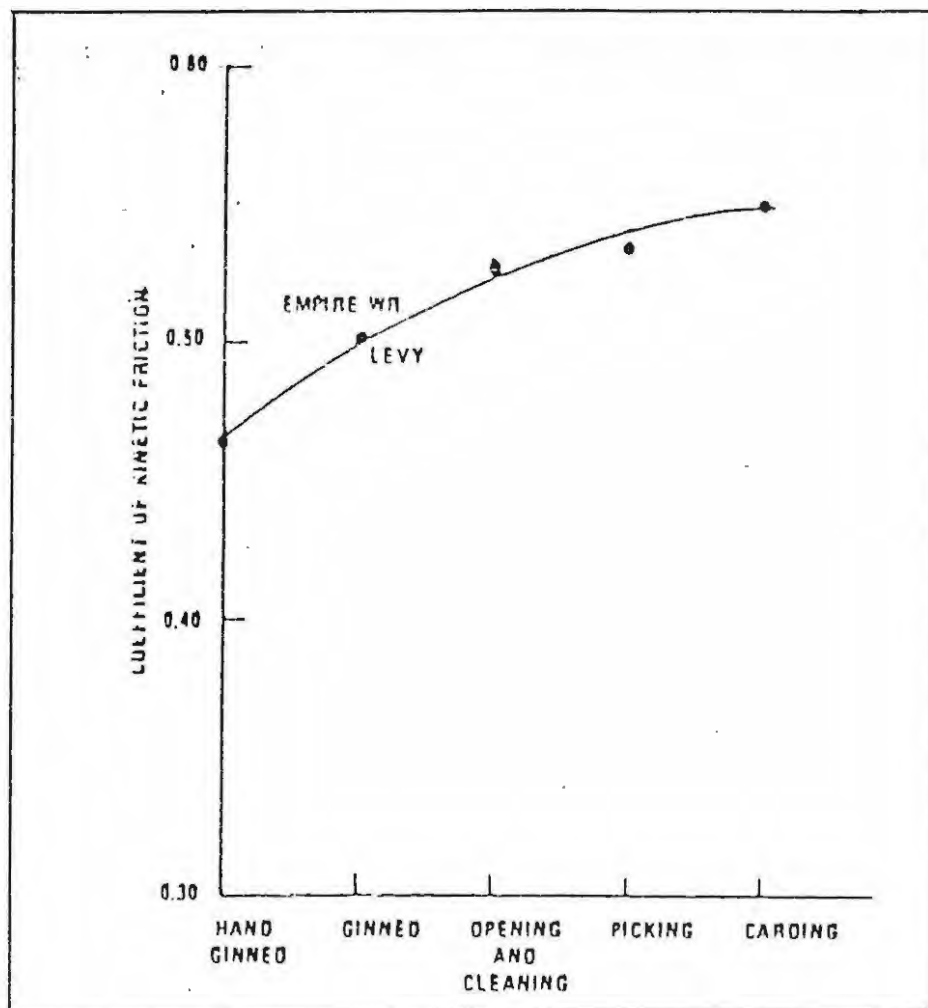
6.4 DISCUSSION

6.4.1 Fibre to metal friction

The trend followed by COF values obtained in this friction study is greige (raw) cotton $\angle$ scoured and bleached cotton fibre $\angle$ solvent-extracted cotton $\angle$ fibre from scoured and bleached CPNF.

Fig 6.3 - Effects of processing on friction (25). These results are for

Empire WR



The results show that cotton that has been ginned, scutched, carded and through the CPNF process has a higher COF than cotton which has only been through the ginning and scutching process. This result is supported by the graph in Fig 6.3 which shows the effects of processing on friction for a variety of cotton.

Although the results refer to friction measured between two crossed cotton fibres, the figure is useful in that it shows the effects of the different processes on the coefficient of friction. The carded cotton has a greater COF than the ginned and scutched cotton. It appears that each of the processes i.e. ginning, scutching and carding and possibly CPNF water jet treatment result in a roughening of the cotton-surface either by mechanical means or by removal of surface wastes and water-soluble matter.

This idea is supported by the fact that cotton surface waxes and sugars do rub off onto metal parts of processing machines (45,57). This effect does indeed cause a problem. Comparing the fibre to metal friction results, for sample B with D & E in figure 6.2 it is clear that solvent-extraction is the more effective method of removing cotton wax and results in an increase in the frictional properties of the cotton.

The role of friction in the manufacture of CPNF is vital because it is friction that holds the material together.

The tensile strength results (Chapter 8) will show that fabric made with solvent-extracted fibre is stronger than CPNF made with greige (raw) cotton when both are in the unbleached state. However this advantage is lost once both fabrics are bleached.

The bleached CPNF made from the solvent-extracted fibre is weaker than the bleached CPNF made from raw cotton. It appears as though the bleaching process has a greater deliterious effect on the more exposed solvent-extracted fibre than on the wax covered greige (raw) cotton.

Two important criteria which are pertinent here for a successful CPNF are

- (i) strength of the entangled fabric (see Chapter 8)
- (ii) the degree of definition of the pattern produced by the water jets (see Chapter 2).

The solvent-extracted fibre (sample 5) and the water-washed unbleached fibre (sample 2) produced the best CPNF's in that the fabrics produced were the strongest (see Chapter 8) and they also showed good pattern definition. The fibre which produced the least successful CPNF was the bleached cotton (sample 4). This fabric was the weakest of the cotton fabrics examined (see Chapter 8) and the pattern lacked definition.

There appears to be a correlation between the ability to make a good CPNF and the COF. The higher the friction the better the CPNF.

6.4.2 Fabric to fabric friction

The temperature and the humidity of the laboratory were $20 \pm 2^{\circ}\text{C}$ and $50 \pm 5\%$ respectively during testing. From Table 6.2 it is seen that the fibres can be arranged in order of increasing friction as follows:

Enka $\angle$ Rayon $\angle$ Greige (raw) cotton $\angle$ Absorbent cotton wool

The fabric to fabric friction results obtained in this study are much higher than the fibre to fibre friction results reported by other workers (18, 19, 20, 39). The difference in COF values is in part due to the fact that the fabric to fabric method differs from the fibre to fibre methods in the area of contact between the test surfaces is very different.

The area of contact ($41,58 \text{ cm}^2$) between the fabrics is much greater than that found in the techniques based on single fibres, tufts or slivers (25,55,56,58). Its influence on the COF values must be significant. Furthermore the results reported here were done with a very much higher traversing velocity than those reported elsewhere.

In this work the traversing velocity of the load, is 6 inches/minute (equivalent to 152mm/minute or 2,54mm/second), compared to velocities such as 0,11mm/second (18) and 9 m/second (26) used by other workers. The effect of increasing the traversing velocity is to increase the coefficient of friction (25). The normal load (200g) used in the fabric to fabric method gives a force of 4,8g/cm². Both the mass and the force it exerts are in the same range as the loads used in other methods (25,56,58).

The degree of success in producing a CPNF for these fibres has been given in Chapter 2 and is repeated here:

FIBRE	DEGREE OF SUCCESS IN PRODUCING CPNF	DEGREE OF SURFACE ROUGHNESS
Grilene	Unsuccessful	Very smooth
Chisso EA	Unsuccessful	Fairly smooth
Enka	Limited success	Slightly pitted but not rough
Abs. Cotton	Limited success	Rough
Wool BPC		
Rayon	Successful	Smooth with ridges
Greige (Raw)	Successful	Rough
Cotton		

In the attempt to produce a CPNF with the Grilene and Chisso fibres the water from the jets was retained on the fibre web thus inhibiting pattern formulation. No fabric could be produced.

The Enka and the Absorbent cotton wool fibres produced a poorly patterned fabric with a low tensile strength (see Chapter 8). Again these two materials held the water from the water jets thus inhibiting the pattern formulation. The greige (raw) cotton and the rayon both produced good CPNF's which had clear patterns and were strong (Chapter 8).

The order of success in producing a CPNF for these fabrics is similar to the order of increasing coefficient of friction for the fibres except in the case of the cotton wool. Absorbent cotton wool BPC, it must be remembered, is produced from cotton fibre which has been scoured and bleached to a very much higher degree than in conventional cotton CPNF. The surface of cotton wool is free of almost all waxes. The "feel" of cotton wool between the fingers is for example a lot drier than the feel of a conventional CPNF between the fingers. It is this wax free surface that allows the water to be held to it inhibiting pattern formulation in CPNF production.

It thus appears that the two paramount properties in determining the success of a CPNF are

- i) friction and
- ii) the ability to allow water to pass through the web ie. the surface should be hydrophobic.

6.5 CONCLUSION

The results obtained in both the fibre to metal friction study and the fabric to fabric friction study from this investigation show that the ability of the fibres to make a successful CPNF improves with increasing friction. However the cotton wool was an exception to the rule because of its hydrophilic surface. These conclusions are similar to that inferred from the SEM photographs.

CHAPTER 7 - ABSORBENCY AND WICKING

7.1 INTRODUCTION

The absorbency of cotton CPNF is its most important property as it is used as a wound dressing. Although it is used mainly with biological fluids, this investigation was confined to the cotton CPNF's absorption of water. This decision was based on the fact that apart from water being the fluid most commonly used to determine absorbency in fibrous materials, water also plays a major role in the manufacturing process of cotton CPNF.

In an absorbent fibrous material there are two sorption processes that take place. First there is the sorption of the liquid by the fibres themselves, in a manner similar to the adsorption and the absorption of vapours and gases (30). This type of process depends on the nature of the sorbing species as well as on the physical and chemical structure of the fibre (30). Secondly there is sorption into the interfibre spaces where the liquid is held by capillary forces. This type of process depends on physical properties such as fibre wettability, fibre surface energy and fibre geometry (30).

For any structure to be absorbent, there has to be a volume to hold the absorbed fluid. This volume can either be initially available or created during the absorption process (59). The absorbency characteristics of a material are a function of (59):

1. the volume available to the fluid

2. the speed with which the volume takes up fluid and retains it
3. the ability of the volume to resist removal of the fluid

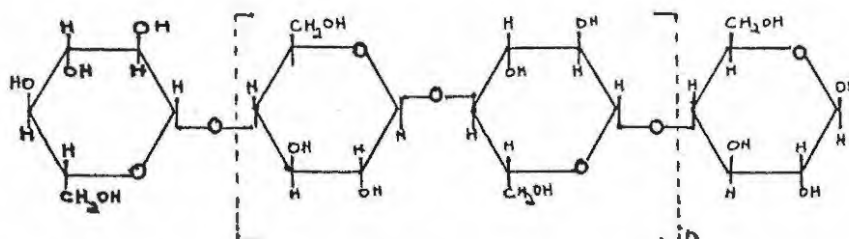
Cellulose fibres such as cotton, rayon and wood pulp have a porous structure which provides spaces where liquid can be held by capillary action (30,59). Unless specifically designed, synthetic fibres can be considered as solid rods having no accessible core and are thus not penetrable by liquids (59). Synthetic fibres can be made wettable by chemical etching, fibre finishing or mechanical treatment (30,59).

7.2 FACTORS WHICH DETERMINE THE ABSORBENCY OF THE FIBRES

7.2.1 Factors which determine the absorbency of cotton fibres

In its natural state, the outermost surface of the cotton fibre is hydrophobic because of wax and other non-cellulosic material that are found in the cuticle and the primary layer (30,43,59). The removal of the cotton wax from the cotton fibre during scouring and bleaching reduces the hydrophobic components and increase the wettability of the cotton, making it hydrophilic. This removal of waxes encourages the fibres to swell, thus increasing the volume available to the sorbing liquid (59).

The structural form of cellulose shown below (49,59),



shows that cellulose is made of a chain of anhydroglucose units.

Each unit shown has three free hydroxyl groups which act as principal sorption sites for water molecules (36, 41).

Water that is directly sorbed is firmly held on the hydroxyl groups by hydrogen bonding, while additional water is held less firmly by secondary polar interactions (36). Cellulose in cotton has a degree of crystallinity in the range 70 - 90% and it is generally assumed that sorption occurs in the non-crystalline or amorphous regions (30,59). The amorphous regions in the average cotton fibre account for 30 - 40% of its total volume (59). It must be remembered that crystallites have regions of imperfection and so these regions will also be penetrable by water (30).

An important feature of cotton is the fibrillar structure of cellulose that can be observed clearly once the non-cellulosic components of the cuticle and the primary wall have been removed (60). There is a helical orientation in the fibrils and they spiral about the lumen at an almost constant angle (20° to 30°) to the fibre axis (59,60).

The fibrils reverse the direction of the helix at intervals along the fibre and these reversal regions coincide with the convolutions that are seen in the dried collapsed fibre (59,60). Sorbing species such as water open a network of channels or pores by acting as agents for interfibrillar and intrafibrillar swelling and thus increasing the volume available for sorption in the cotton fibre (61).

7.2.2 Factors which determine the absorbency of the synthetic fibres

The Enka fibre is a bicomponent of polyethylene and polyester, with the lower melting point (polyethylene) most likely to be the sheath. Thus it is basically a polyolefin fibre at the surface. Polyolefin fibres are not absorbent fibres because unlike cellulosic fibres, they have no specific sorption sites for moisture (30). Sorption can be enhanced in polypropylene fibres by chemical modifications such as copolymerization with other monomers or by grafting on other polymers (30). It can also be enhanced by applying a hydrophilic finish on the fibre, thus improving its wettability to give it reasonable wicking and absorbent properties (59).

The Grilene fibre is a polyester fibre. Like the polyolefin fibres, polyester fibres have no sorption sites for moisture (30). However polyester fibre is used in many liquid absorbency applications because of its ability to produce fine fibres (36). This fine structure can be seen in the micrographs shown in Chapter 2 (SEM study). Polyester also forms irregular or noncircular cross-sectional shapes easily. Both these characteristics enable it to form interfibre spaces where capillary action is effective in the fibrous web or network, resulting in a high liquid absorbency (30).

The rayon is a regenerated cellulose fibre with a much lower degree of crystallinity (30 - 50%) than natural cotton cellulose, and a shorter molecular chain length (30,60).

Earlier in this chapter, it was said that the absorbency of cellulose was determined by the amorphous regions in the molecular structure (30). It is thus understandable that rayon with its lower degree of order has a higher absorbency capacity than cotton. However, a number of processes exist for manufacturing rayon and these result in end-products with different properties (59). Cross-sections in rayon fibres vary from the irregular serrated in regular rayon to a "smooth, almost round, all core cross-section" in cuprammonium rayon (41). Rayon also has a fibrillar structure but without the helical orientation found in cotton (60).

7.3 EXPERIMENTAL

7.3.1 Materials

10 Batches of the finished cotton CPNF product were sampled from a manufacturing line, and evaluated for absorbency properties. The fabrics that were manufactured using the cotton fibres prepared in the laboratory were also tested. They were made from the following cotton fibres and were prepared according to the methods described in Chapter 3:

- i) solvent-extracted fibre
- ii) washed fibre
- iii) washed, solvent-extracted fibre
- iv) fibre treated with 2% NaOH solution
- v) greige (raw) cotton from the single batch used throughout this investigation

The experimental fabrics made on the pilot laboratory CPNF line using the following synthetic fibres were also evaluated:

- i) Enka fabric
- ii) Absorbent cotton wool BPC fabric
- iii) Rayon fabric
- iv) Rayon fabric made with a longer entangling time of 20 mins

The carded fibre webs of the synthetic fibres that did not form entangled fabric i.e. Grilene and Chisso EA fibres, were also evaluated.

7.3.2 Methods of Analysis

The absorbency properties of the test material were evaluated using sections B & C of the EDANA test method 10.1-72 for nonwovens absorption (see appendix 12). These methods evaluate liquid absorptive capacity (commonly known as absorbency capacity) and liquid wicking rate respectively. The only modification made in the method is that in this investigation, the results are reported as a g/g quantity instead of the percentage used in the method.

Distilled water at 20°C was used as the test liquid.

7.4 RESULTS

TABLE 7.1 - Absorbency capacity tests on standard factory
production of cotton CPNF

Batch No	Absorbency Capacity (g/g)
789	9,8
796	8,3
801	8,6
803	9,
805	11,2
812	9.0
834	10,2
1105	8,7
1109	9,0
Average =	9,3
Std dev =	0,9
COV =	9,7%

Table 7.2 - Absorbency capacity tests on specially prepared cotton CPNF

Fibres used to make fabric	Absorbency Capacity (g/g)
greige (raw) cotton	11,3
washed fibre	12,1
2% NaOH treated fibre	11,8
solvent-extracted fibre	10,1
washed, solvent-extracted fibre	10,5

TABLE 7.3 - Absorbency capacity of pilot laboratory fabrics including
those made with synthetic fibres & cotton fibres

<u>Fibre used to make fabric</u>	<u>Absorbency Capacity (g/g)</u>
Absorbent cotton wool BPC	17,0
Enka	16,5
Rayon	11,9
Rayon (20 minutes)	12,0
Standard cotton CPNF	9,2
Greige (Raw) Cotton	2,3

TABLE 7.4 - Absorbency of synthetic & cotton fibres used in pilot
laboratory trials

<u>Fibre</u>	<u>Absorbency Capacity</u>
Crilene (carded)	40,0
Chisso EA (carded)	25,5
Absorbent cotton wool BPC	21,1
Scoured and bleached cotton fibre	19,7
Enka	19,0
Rayon	13,3
Greige (Raw) Cotton	2,5

TABLE 7.5 - Wicking rate (machine/direction) of standard factory
production of cotton CPNF (scoured & bleached)

Batch No	Height travelled (mm) after time (seconds)		
	10s	30s	60s
789	37	55	69
796	40	57	72
801	40	62	77
803	37	54	69
805	40	57	73
812	41	61	79
815	35	56	66
834	55	60	73
1105	29	38	41
1109	46	67	85
Mean	38,9 ⁺ 4,8	56,7 ⁺ 7,6	70,4 ⁺ 11,7

TABLE 7.6 - Wicking rate (cross direction) of standard factory production
of cotton CPNF (Scoured and bleached)

Batch No	Height travelled (mm) after time (seconds)		
	10s	30s	60s
789	26	38	47
796	32	46	60
801	34	49	60
803	28	40	49
805	31	42	53
812	33	47	58
815	31	45	53
834	28	39	45
1105	31	40	43
1109	35	48	57
Mean	30,9 ⁺ 2,8	43,4 ⁺ 4,1	52,5 ⁺ 6,3

TABLE 7.7 - Wicking rate (machine direction) of experimental factory
produced CPNF

Fibre used to make fabric	Height (mm) travelled in time (seconds)		
	10s	30s	60s
Solvent-extracted fibre	39	61	74
Washed, solvent-extracted fibre	37	59	74
Washed fibre	34	54	62
2% NaOH treated fibre	36	58	69
Greige (Raw) Cotton	34	59	73

7.5 DISCUSSION

The results in Table 7.2 show that the absorbency capacity of the fabrics made with cotton fibre that has been solvent-extracted is not significantly different from that of conventional CPNF. This supports the findings in Chapter 6, that the initial advantage of increasing the frictional properties of cotton by using solvent-extraction to remove the surface waxes is lost once the fabric has been scoured and bleached. The removal of some of the surface waxes from the cotton fibre before the scouring and bleaching process was expected to improve the efficiency of this chemical process, resulting in a more absorbent product.

It appears that although the application of the scouring and bleaching process to the unbleached fabric made with the solvent-extracted fibre resulted in a decrease in tensile properties (see Chapter 8), the effect did not improve the absorbency of the cotton fibre beyond that of the conventional CPNF.

The absorbency results of the other fabrics shown in Table 7.2 also do not differ significantly from that of the conventional CPNF. The results in Table 7.2 are in agreement with a mechanism that has been suggested for the absorbency of cotton fabrics, which proposes that absorption is a function of the spaces in the fabric (62). According to this mechanism, for a given construction, cotton fabrics of the same weight end up with practically identical absorbency properties after laundering (62).

The mechanism offers an explanation as to why the fabrics made with cotton fibres that were different initially, end up with similar absorbency properties. The wicking properties of these fabrics (see Table 7.5, 7.6 and 7.7) are also not significantly different from those of the conventional CPNF. Only the machine direction results are available for the experimental fabrics (Table 7.7) due to limited samples.

The standard CPNF results (Table 7.5 and 7.6) show that the absorption flow rate is faster in the machine direction than in the cross direction. Wicking has been described as capillary adsorption in a porous media (63). It is clear that the fabric spaces are also important in deciding the wicking properties of fabrics. Laundering results in a more uniform distribution of the fibres throughout the space available (62).

The scouring and bleaching process is expected to do the same and effect a uniform distribution of spaces in all the fabrics. Thus the flow rate of the liquid is virtually the same in all the fabrics because of the similar size spaces they have as a result of the re-distribution that takes place during scouring and bleaching.

The absorbency properties of the synthetic fibres (Table 7.3) follow the same trend that was observed when comparing the degree of success of producing CPNF with the synthetic fibres in terms of distinct pattern formation. Absorbent cotton wool BPC and the Enka fibre formed the least successful CPNF and they have the highest absorbency properties. The Enka fibre is hydrophobic in nature, so its apparent absorbency capacity can be attributed to water held in the interfibre spaces in the fabric. Greige (raw) cotton exhibits its hydrophobicity in its very low absorbency capacity.

The differences in absorbency capacities of the pilot laboratory fabrics can also be explained in terms of interfibre spaces in the fabrics. The fabrics with poor pattern definition have smaller interfibre spaces and a bigger surface area to hold the water. The well-defined patterns of the fabrics that form a successful CPNF imply bigger interfibre spaces and thus a smaller surface area for absorption of the water.

The absorbency properties of the synthetic fibres in Table 7.4 includes the results of the Grilene and Chisso EA fibres that were completely unsuccessful in forming CPNF. These two fibres were tested as carded samples to illustrate the maximum absorbency capacity that the fibres are capable of when used in the CPNF process.

The other fibres were not tested as carded samples. The high absorbency capacity values measured for the Grilene and Chisso EA fibres confirm the fact that has already been mentioned in Chapter 6 regarding the role of fibre absorbency in the production of CPNF. A high absorbency capacity or hydrophilicity of the fibre results in water being trapped in the fibre web during the process using water jets to form patterns and in so doing prevents pattern formation. The ability to make a successful CPNF from a "well-defined pattern point of view" increases with decreasing absorbency capacity. The best cotton CPNF must however be absorbent as well.

7.6 CONCLUSIONS

1. A cotton fibre that is too hydrophilic and thus an excellent water absorbent does not form a good CPNF because the water jets cannot "press" the fibre into a fabric or penetrate the fibre well enough to form a pattern. This is demonstrated by the cotton wool which is highly absorbent due to the removal of the surface waxes and could not be made into a successful CPNF.
2. The cotton fibre surface must be partly hydrophilic for a CPNF to be successfully made by water jets, as the water must not be retained by the fibre. This is demonstrated by the greige (raw) cotton with its surface waxes and low absorbency.
3. The high water-holding capacity of the fine synthetic fibres (Grilene and Chisso EA fibres) prevents the water jets from penetrating the fibre web and forming a pattern to create a coherent fabric.

4. The absorbency properties of the rayon fibre appear to represent the upper limit of absorbency capacity that will permit the water jets to penetrate a fibre web and pattern it with a reasonable degree of success. Seeing that a longer patterning time had to be used to achieve a reasonably coherent fabric with the rayon, it is clear that fibres with absorbency capacity values greater than 13 cannot be patterned successfully.

CHAPTER 8 - TENSILE PROPERTIES

8.1 INTRODUCTION

The tensile properties that have been measured in this study are the tensile strength and the tensile energy. The tensile strength was determined as the force required to break a strip of fabric while the tensile energy was determined as area under the load - elongation curve that is plotted during a tensile strength measurement. The tensile energy is also known as the work of rupture. It is the energy needed to break a fibre and is a measure of the ability of the fabric to withstand sudden shocks of given energy (31).

By their definition, tensile properties are mechanical properties because they measure the response to an applied force. Mechanical properties of fabrics are important because they contribute significantly to the properties of the final product. While these properties depend to a certain extent on the fibre properties, the properties are also affected by the structural arrangement in the fabric (31). The fibre properties are a limiting factor of what is possible in a fabric because its strength, for instance, cannot be greater than the sum of the maximum strength of its component fibres (46).

Thus the tensile properties of a fabric can be used to determine the measure of success of the process used to manufacture or produce the fabric, as is the case in this study.

8.2 EXPERIMENTAL

8.2.1 Methods used to produce the experimental fabrics

All the experimental fabrics using both the cotton fibres as well as the synthetic fibres were made using a carded web. 32g (the extra 2g is the allowance made for fibre loss during carding) of each fibre sample was weighed and carded to produce a web with dimensions 1m x 0,5m and a weight of approximately 60g.s.m. (gram per square meter). The webs prepared from the following fibre samples were patterned on a manufacturing line.

1. the greige (raw) cotton (used as a control)
2. the solvent-extracted cotton
3. the washed, solvent-extracted cotton
4. the washed cotton
5. the cotton fibre that had been treated with 2% NaOH solution

The details of fibre preparation were given in Chapter 3. The patterned fabrics were then put through the scouring and bleaching process before being tested.

The fabrics made with the synthetic fibre and the cotton controls were produced on a pilot laboratory line, scaled down to reproduce the patterning process performed on the manufacturing line. The water jets oscillated at a rate of 7 cycles per minute. Water pressure was set at 800 kPa and the fibre webs were patterned for 10 minutes.

Due to limitation of size, a 9cm x 9cm square of the carded fibre web was patterned at a time and this produced a fabric of approximately 7,5cm x 7,5cm. Thus the test results from these fabrics can only be compared among themselves due to non-standard size samples being used.

The following synthetic fibres (see Fig 1.3 in Chapter 1) were tried on the pilot laboratory line.

1. 1,1 dTex*, 38mm Grilene (a polyester fibre)
2. 3,0 den†, 38mm Chisso EA (a polyethylene sheath on a polypropylene core)
3. 3,0 den, 38mm Enka (a polypropylene /polyester bicomponent fibre - identified by means of DSC analysis)
4. 38mm rayon fibre (a regenerated cellulose fibre)
5. greige (raw) cotton
6. Absorbent cotton wool BPC

The attempts to pattern the Grilene and the Chisso EA fibres were completely unsuccessful. The Grilene fibre had a bulky web with a high loft. It retained the water from the jets for some time before allowing it through. Thus at the end of the patterning time the polyester fibre was very heavy with the retained water and the web remained a mass of fibres with no patterning at all. The Chisso EA fibre was not as lofty as the Grilene fibre and it did not retain as much water. It was eventually flattened and the water from the jets passed through the web. There were a lot of air locks in the flattened web and at the end of the patterning time, the Chisso EA web remained a mass of fibres with no patterning at all.

* A tex is the mass in grams of one kilometre of fibres, filaments, slivers and yarns or other linear textile material. A decitex (dTex) is a decigram per kilometre. The tex is a SI unit.

+ Denier is the weight in grams of 9000 metres of a filament or yarn.

The Enka fibre was immediately flattened by the water jets and the water jets penetrated the web. The air locks were not as big as in the Chisso EA fibre but they were gradually dispelled. At the end of the patterning time a faint pattern could be seen on the resultant fabric. The rayon web absorbed and retained the water before the water jets slowly penetrated the web. There were big air locks on the surface of the web but these were gradually dispelled. After 10 minutes a slight pattern formation could be seen. When the patterning time was increased to 20 minutes there was a definite improvement in the pattern formation but it still did not match the distinctness that was found when patterning greige (raw) cotton.

The absorbent cotton wool BPC also retained water for some time before allowing it through. The pattern formed at the end of the patterning time was only slightly better than that seen in the Enka web. Except for the cotton wool all the fibre webs discussed above had a tendency to slide from the nylon belt carrying them. This tendency must also have affected the pattern formation. It is seen as indication of their low friction properties which resulted in a poor adherence to the nylon belt carrying the webs.

In contrast to the experimental fibres, the greige (raw) cotton web stayed in place on the carrier belt. It absorbed the water and was almost immediately penetrated by the water jets. At the end of the patterning period, a coherent fabric with a clear well-defined pattern was obtained.

8.2.2 Test Methods

The tensile properties were tested according to EDANA test method 20.1-72 (see appendix 13) using an Instron Model 1026 Tensile Tester with a load cell type 2512-109 (0-50kg range). The cross-head speed was 100mm/min and the initial jaw separation was 20cm. The sample size was 25cm x 5cm for all fabrics except those produced on the pilot laboratory line. For the latter a sample size of 7,5cm x 2,5cm was used. All the samples were conditioned as specified in the test method.

8.3 RESULTS

TABLE 8.1 - Tensile strength of unbleached standard factory produced cotton
CPNF Results given are an average of 10 values, with std deviations

Type of tensile strength	Tensile strength in Kg per 50mm
Cross direction, dry	0,8 $\pm$ 0,2
Cross direction, wet	2,1 $\pm$ 0,4
Machine direction, dry	3,0 $\pm$ 0,5
Machine direction, wet	5,9 $\pm$ 1,1

TABLE 8.2 - Tensile energy of unbleached standard factory produced cotton CPNF
Results given are an average of 10 values, with std deviations

Type of tensile energy	Tensile energy in Joules
Cross direction, dry	0,8 $\pm$ 0,4
Cross direction, wet	1,8 $\pm$ 0,3
Machine direction, dry	1,6 $\pm$ 0,3
Machine direction, wet	2,8 $\pm$ 0,6

TABLE 8.3 - Tensile strength of bleached standard factory produced cotton CPNF

Results given are an average of 10 values, with std deviations

Type of tensile strength	Tensile strength in Kg per 50mm
Cross direction, dry	2,0 $\pm$ 0,3
Cross direction, wet	1,8 $\pm$ 0,2
Machine direction, dry	7,5 $\pm$ 2,0
Machine direction, wet	6,6 $\pm$ 1,3

TABLE 8.4 - Tensile energy of bleached standard factory produced cotton CPNF

Results given are an average of 10 values, with std deviations

Type of tensile energy	Tensile energy in Joules
Cross direction, dry	1,8 $\pm$ 0,3
Cross direction, wet	1,4 $\pm$ 0,1
Machine direction, dry	1,9 $\pm$ 0,5
Machine direction, wet	2,2 $\pm$ 0,4

TABLE 8.5 - Tensile strength of factory produced CPNF made with solvent-extracted cotton fibre (an experimental product). Results given are an average of 10 tests, with std deviations

Type of tensile property	Result unbleached fabric	Result bleached fabric
Tensile strength, cross direction, dry	$1,7 \pm 0,5 \text{ kg/50mm}$	$1,4 \pm 0,2 \text{ kg/50mm}$
Tensile strength, cross direction, wet	$1,8 \pm 0,4 \text{ kg/50mm}$	$1,8 \pm 0,4 \text{ kg/50mm}$
Tensile strength, machine direction, dry	$4,5 \pm 1,1 \text{ kg/50mm}$	$3,8 \pm 0,6 \text{ kg/50mm}$
Tensile strength, machine direction, wet	$6,0 \pm 1,1 \text{ kg/50mm}$	$5,6 \pm 1,3 \text{ kg/50mm}$

TABLE 8.6 - Tensile energy of factory produced CPNF made with solvent-extracted cotton fibre (an experimental product) Results given are an average of 10 tests, with std deviations

Type of tensile energy	Result for unbleached fabric	Result for bleached fabric
Tensile energy, cross direction, dry	$1,4 \pm 0,3 \text{ J}$	$1,2 \pm 0,2 \text{ J}$
Tensile energy, cross direction, wet	$1,4 \pm 0,3 \text{ J}$	$1,4 \pm 0,4 \text{ J}$
Tensile energy, machine direction, dry	$1,8 \pm 0,4 \text{ J}$	$1,3 \pm 0,2 \text{ J}$
Tensile energy, machine direction, wet	$2,5 \pm 0,6 \text{ J}$	$2,1 \pm 0,4 \text{ J}$

TABLE 8.7 - Tensile energy (cross direction) of bleached factory produced experimental cotton CPNF. Results given are an average of 5 tests

Type of cotton fibre used	Dry tensile energy	Wet tensile energy
Greige (raw) cotton	1,2 J	1,3 J
Washed cotton	1,3 J	1,3 J
2% NaOH treated cotton	1,6 J	1,1 J
Washed, solvent-extracted cotton	1,1 J	1,1 J

TABLE 8.8 - Tensile strength (machine direction) of bleached factory produced experimental cotton CPNF. Results given are an average of 5 tests

Type of cotton fibre used	Dry tensile strength	Wet tensile strength
	kg/50mm	kg/50mm
Greige (raw) cotton	4,4	5,1
Washed cotton	4,9	4,3
2% NaOH treated cotton	4,9	3,5
Washed, solvent-extracted cotton	6,0	5,1

TABLE 8.9 - Tensile energy (machine direction) of bleached, factory produced experimental cotton CPNF. Results given are an average of 5 tests

Type of cotton used	Dry tensile energy	Wet tensile energy
Greige (raw) cotton	1,4 J	1,9 J
Washed cotton	1,8 J	1,5 J
2% NaOH treated cotton	1,6 J	1,3 J
Washed, solvent-extracted cotton	2,1 J	2,0 J

TABLE 8.10 - Tensile properties (machine direction) of experimental CPNF made with synthetic fibres and cotton. Results given are an average of 5 tests

Type of fibre used	Dry tensile strength in	Wet tensile energy in
	g/50mm	Joules
Enka fibre	0,14	0,04
Absorbent cotton wool BPC	0,37	0,08
Rayon (10 minutes)	2,21	0,36
Rayon (20 minutes)	2,63	0,47
Greige (raw) cotton	0,47	0,15

8.4 DISCUSSION

8.4.1 Tensile properties of Standard Cotton CPNF

The tensile properties of the conventional cotton CPNF (see Tables 8.1 to 8.4) can be summarized as follows:

1. The tensile strength of the fabric is stronger in the machine direction (MD) than in the cross direction (CD) in both the unbleached fabric as well as the scoured and bleached fabric.
2. Both the tensile strength and the tensile energy of the unbleached fabric show it is stronger wet than dry.

3. There is no significant change in the tensile strength of the bleached fabric in going from dry to wet.
4. The tensile energy of the bleached fabric is virtually the same in both directions when dry, and it also does not change significantly when the fabric is wet.

The strong intermolecular and intramolecular hydrogen bonding of the hydroxyl group on the anhydroglucose unit of cellulose are responsible for the strength of the natural cellulosic fibres such as cotton (30). The high degree of intermolecular bonding as well as the relatively high degree of crystallinity (70-90%) make it effectively a highly cross-linked polymer system (30). Cross-linking imparts rigidity as well as strength to a polymer (59).

The tensile strength results of the conventional cotton CPNF show that the tensile strength of the fabric is either higher when it is wet (as with the unbleached fabric, Table 8.1) or relatively unaffected (as with the bleached fabric, Table 8.3). This is in keeping with the known behaviour of cotton, which is either unaffected by variations in moisture content or may even increase with increasing moisture (30). The presence of water results in some of the cross-links being broken, but instead of reducing the bond forces, this results in a certain degree of chain mobility and optimum stress distribution (59). These changes ultimately result in a higher fibre strength in the wet state than in the dry state (30).

8.4.2 Tensile properties of factory produced fabrics from specially prepared cottons

In this study, the tensile strength of a CPNF is one of the criteria used to assess the degree of success achieved by a fibre in making the CPNF. The CPNF made with solvent-extracted fibre had potential to make a fabric that improved upon the tensile properties of the conventional cotton CPNF, due to its higher frictional properties (see chapter 6). The dry tensile strength MD (4,5kg 150mm - Dry) of the unbleached fabric made with the solvent-extracted fibre is higher than the equivalent tensile strength of the unbleached conventional cotton CPNF (3,0kg 150m - MD, Dry). However, this superiority is lost when the fabrics are wet (solvent-extracted fibre CPNF = 6,0kg/50mm and cotton CPNF = 5,9kg/50mm).

The solvent-extracted cotton CPNF differs from the conventional CPNF in that it is actually weakened by bleaching (see Table 8.5) and wetness reduces the tensile strength even more. It would seem that the removal of the cotton wax during the solvent extraction process exposes the cotton fibre to a greater degree of degradation by the bleaching process than that experienced by the unbleached cotton fibres in the standard fabric. Bearing in mind that the absorbency capacity of cotton CPFN was also not improved by using the solvent-extracted fibre, it is clear that the only advantage gained by using solvent-extracted fibre is to produce a better pattern definition than is found in standard cotton CPNF.

The tensile properties of the other cotton experimental fabrics (Tables 8.7 to 8.9) are not significantly different from those of the control made with raw cotton and subsequently scoured and bleached. Only the washed, solvent-extracted fibre makes a stronger bleached fabric, but this superiority is lost when the fabric is wet (see Table 8.9). This is because the tensile strength of the wet raw cotton fabric improves, while the other fabric is weaker when wet. It is important to note that unlike the fibre that has only been solvent-extracted, the fibre that has been solvent-extracted then washed actually results in a fabric which is stronger in the bleached state than the standard CPNF. The fabric made with the washed, solvent-extracted fibre also exhibited a better pattern formation than the standard CPNF.

As with the standard cotton CPNF, the tensile energy of the experimental fabrics is virtually the same in both directions. The tensile energy of the CPNF made with solvent-extracted fibre, the only one tested when wet, did not seem to change significantly when the fabric was wet.

8.4.3 Tensile properties of the synthetic fibres

The tensile properties of the synthetic fibres and the cotton fibres (see Table 8.10) show that the Enka fibre made the least coherent CPNF. It was followed closely by the absorbent cotton wool BPC CPNF which was also weak, with poor pattern definition. Both these fabrics were even weaker than the CPNF made with greige (raw) cotton, which still had to be scoured and bleached to make the final product.

Based on the results in Tables 8.1 and 8.3, the raw cotton CPNF can be expected to increase in tensile strength (MD, dry) 2,5 times when scoured and bleached. Thus the tensile strength of this raw cotton CPNF would increase from 0,47kg/25mm to 1,1kg/25mm.

The rayon CPNF made initially (entangling time of 10 minutes) was stronger than the cotton CPNF (see table 8.10). It would still have been stronger even if the greige (raw) cotton CPNF had been scoured and bleached. The strength of the rayon fabric was improved (see Table 8.10) when the entangling time was increased. The absorbency capacity of the rayon CPNF 11,9 g/g (see Table 7.3 in Chapter 7) is for a fabric made using the pilot laboratory process and can thus not be compared directly to the absorbency capacity of the standard CPNF. However, it is enough to indicate that an absorbency capacity that at least matches that of the conventional product can be expected from a factory produced rayon CPNF. Knowledge of the basic properties of the rayon fibre with moisture regain value ranging from 10,7% to 13% support this (59).

The main disadvantage of using the rayon fibre to make a commercial CPNF is that it would take longer to make a reasonably defined pattern. This would raise the manufacturing costs of the product. Apart from costs, the aesthetic appeal of the rayon fabric is definitely much less than that of the standard cotton CPNF because of its poorly defined pattern.

8.5 CONCLUSION

Of the cotton variants (washed fibre, solvent-extracted fibre, 2% NaOH treated fibre and washed, solvent-extracted fibre) only the washed, solvent-extracted fibre made a CPNF which could be considered an improvement on the standard cotton CPNF. The washed, solvent-extracted fibre made a CPNF that had better dry tensile strength and better pattern definition than standard cotton CPNF.

Of the synthetic fibres evaluated, only rayon made a CPNF which was comparable to standard cotton CPNF in its strength and would be commercially viable.

CHAPTER 9 - CONCLUSIONS

The initial objectives (section 1.2.6) have been answered. The process used commercially to produce cotton CPNF resulted in the removal of water-soluble components (first stage), the removal of some of the wax from the surface (scouring and bleaching stage) and a shortening of the cellulose chains (bleaching stage).

The properties of cotton that characterise a successful CPNF are:

1. The cotton fibre must be fairly hydrophobic and have limited absorbency properties. Thus the cotton fibre must have some of its surface waxes in order for the patterning process to work. The reason is that the water jets that produce the pattern must not be absorbed by the fibre and in so doing be unable to penetrate the fibre web.
2. The cotton fibre must have good frictional properties that will enable it to stay on the belt that carries the web during the patterning process as well as good interfibre friction that will promote cohesion in the patterned fabric. The degree of success in making a CPNF improves with increasing friction.

The two paramount properties are inter-related because pilot experiments showed that friction alone is not the decisive factor in the formation of a successful CPNF. If that were the case, the Absorbent Cotton wool BPC which had the best frictional properties ought to have formed the most successful CPNF, which was not the case. It proved to be too hydrophilic.

That these two properties characterise a successful CPNF is further confirmed by the failure of the patterning process to work with all synthetic fibres apart from rayon. The success achieved with the rayon is attributed to:

1. its lower absorbency capacity in comparison to the unsuccessful synthetic fibres.
2. its higher friction properties in comparison to the unsuccessful synthetic fibres.

Apart from these conclusions, it has been shown that the exothermic peak which is so characteristic of greige (raw) unwashed cotton is most likely to be due to the presence of potassium phosphate salts (KH_2PO_4 or K_2HPO_4).

In addition, DSC analysis has shown that the cotton wax extracted from the greige (raw) cotton used in this study has 3 major components. These have been identified by GC-MS analysis as palmitic acid, $\text{C}_{16}\text{H}_{32}\text{O}_2$, octacosane $\text{C}_{28}\text{H}_{58}$ and Cetyl Stearate $\text{C}_{17}\text{H}_{35}\text{CO}_2\text{C}_{16}\text{H}_{33}$.

APPENDIX 1 - A CONVENTIONAL CPNF

APPENDIX 2 - GREIGE (RAW) COTTON FIBRE



APPENDIX 3 - UNBLEACHED CPNF



APPENDIX 4 - GRILENE FIBRE

APPENDIX 5 - CHISSO EA FIBRE

APPENDIX 6 - ENKA BICO FIBRE

APPENDIX 7 - VISCOSE RAYON FIBRE

APPENDIX 8 - ABSORBENT COTTON WOOL BPC

SOUTH AFRICAN BUREAU OF STANDARDS
STANDARD TEST METHODS FOR TEXTILES
FLUIDITY OF COTTON, RAYON AND
ACETATE FIBRE

SECTION 1. APPARATUS

- (a) *Capillary viscometer.* X-type, B.C.I.R.A.
- (b) *Glass jackets*
- (c) *Weighing tubes or bottles*
- (d) *Glass funnel.* A short-stemmed glass funnel, 3.5 cm. in diameter.
- (e) *Camel-hair brush or tweezers*
- (f) *Steel or glass rod.* A steel or glass rod 30 cm. in length $\times$ 1.6 mm. in diameter.
- (g) *Blackened glass container*
- (h) *Short burette.* A short burette, graduated at 0.7-ml. intervals.
- (i) *Revolving wheel.* A revolving wheel capable of revolving at 4 r.p.m.
- (j) *Electrically-heated glass thermostat.* An electrically-heated glass thermostat ($20 \pm 0.2^\circ\text{C}$) and stirrer.
- (k) *Stop-watches.* Two stop-watches, each graduated in seconds and 0.2 seconds.

SECTION 2. REAGENTS

- (a) *Standard cuprammonium solvent.* A solvent consisting of 15 ± 0.1 g. copper per litre, 200 ± 5 g. ammonia per litre, and less than 0.5 g. nitrous acid per litre.
- (b) *Mercury*

SECTION 3. PREPARATION OF TEST SPECIMEN

From each test sample cut a test specimen weighing not less than 5 g., and desize it as described in *S.A.B.S. Method 114* using petroleum ether (B.P. 60 to 120°C) in place of the methylene chloride. After drying and conditioning of the specimens, proceed as follows:

In the case of fabric, cut the strips diagonally into short fragments; in the case of fibres and yarn, cut them into very short lengths.

SECTION 4. PROCEDURE

4.1 Accurately weigh the fabric fragments and the short lengths of yarn and fibre required by a viscometer calibration to yield a 0.5 per cent solution of dry cotton or, in the case of rayon, 2 per cent.¹⁾

4.2 Clamp the viscometer in a vertical position and approximately half-fill with cuprammonium solvent. Open the lower clip momentarily to fill the capillary. Add 0.7 ml. of mercury. Using the funnel and brush, introduce the weighed specimen into the viscometer and mix rapidly by means of the rod. Fill the viscometer completely and insert the stopper so that the excess of liquid overflows, displacing all air. Close the outlet immediately, wrap the viscometer in black cloth, to exclude any light, attach it to the revolving wheel and rotate overnight.

4.3 Remove the viscometer and carefully drain the mercury. Put the viscometer into a closed jacket (excluding all light) supported in the thermostat at $20 \pm 0.2^\circ\text{C}$ and leave for 15 minutes. Transfer to the open jacket fixed vertically in the thermostat, remove the stopper and allow the solution to flow freely through the capillary tube. Measure the time in seconds required for the meniscus to fall from the top to the intermediate and bottom marks.

¹⁾ The weight of acetate fibre required to give a 2 per cent solution of cellulose may be obtained from the formula:

$$W = 2V \left[\frac{1}{100 - m} \times \frac{142.9}{142.9 - A} \right]$$

where

W = required weight of cellulose acetate, in grams,

V = volume of the viscometer, in millilitres,

m = per cent moisture content (grams water per 100 g. air-dry cellulose acetate), and

A = per cent acetic acid yield of the cellulose acetate.

SECTION 5. CALCULATION

$$\text{Fluidity, rhes} = \frac{C}{(t - \frac{K}{t})}$$

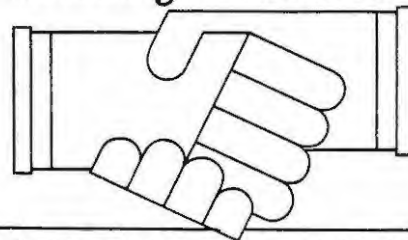
where

- t = time in seconds, for the meniscus of the liquid to travel from the top to the bottom mark,
- C = viscometer constant including the density value of the cuprammonium solution, and
- K = kinetic energy correction constant of the viscometer.

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September 14, 1987.

Dear Professor Letcher,

Please find enclosed the results of the fibre/metal friction tests carried out on your 5 samples of cotton.

The method used is similar to that described in the Shirley Institute Bulletin Volume 53 No. 2 p. 33. However, the apparatus has been completely redesigned and rebuilt incorporating a micro-processor.

Our invoice for the work will be forwarded shortly.

Yours sincerely,

A handwritten signature in cursive script, appearing to read 'R. W. Rennell'.

Dr R W Rennell
Instrumentation

Encl



Procedure for Coefficient of Friction Measurements

The SP-101A and its standard accessories have been designed to conform to ASTM Standard D-1894. The user should be completely familiar with the requirements of this spec. While originally written to cover COF measurements on plastic films, this spec. may be used as a guide for other materials as well. Of course the application of the SP-101A is by no means limited to that method. Other sled configurations and weights may be substituted.

Calibration - COF

Establish the following controls conditions:

- Main Power ON
- Averaging Time Selector on 10
- Meter Select Switch on Instantaneous
- Platen Speed Selector on Low (6"/min.)
- Zero Check Switch in Test (down)

Remove the Load Cell and Mount Assembly by removing the two screws indicated in figures 3 and 4. Leave the electrical connection from the Load Cell plugged into the console. Attach the Friction Sled Tether to the ball end on the Load Cell by snapping on the bead chain connector. Orient the Cell and Mount Assembly as shown in figure 5, with the ball-end pointing down and the Tether hanging vertically. While it is best to use a ring stand or some other mechanical support for this purpose, a reasonably satisfactory job can be done by hand-supporting the unit. In either case make sure that fingers or clamps do not touch the Load Cell itself. At this time, the weight of the Bead Chain and Tether should be carried by the Load Cell, but the Sled weight should not, resting on the platen or table as shown in figure 5. Bring the Meter reading to 000.0 with the front panel Fine Balance Control. If the mass of the material to be applied to the sled is appreciable, it should be applied now. Most commercial plastic films and papers are so light that their contribution to sled weight may be ignored.

Now, also suspend the Sled weight by raising the cell as shown in figure 6. Adjust the front panel Cal control, using a small screwdriver, to make the Meter read 100.0. Try applying & removing the Sled weight only several times, observing that the zero and full scale indications are correct and repeatable.

Since the Coefficient of Friction is equal to the tractive force divided by the normal force (weight pressing the two surfaces together), the meter will now read COF directly. The calibration procedure using the sled weight to establish force span, effectively normalizes the calibration, by making the denominator of the fraction 1 or 100.0%. For example a Meter reading of 25.0 would indicate a COF value of .250.

Reassemble the Load Cell and Mount Assembly. With no tension applied to the Tether, rebalance the Meter to 000.0 to correct for the change in cell tare weight caused by the new orientation of the cell. This is a valid manipulation since the Balance (Tare) and Calibration (Span) act independently of one another.

Specimen Treatment - COF

Place the two test surface material specimens on the Sled and on the Test Platen, following ASTM D-1894, or according to your company's established procedure. If the material is thicker than 10 or 15 mils, it may be necessary to vertically reposition the Load Cell to keep the Tether level. Loosen the two Phillips Head screws which secure the cell to its mount and slide it to the correct alignment. Do not apply any manual force or torque to the Cell! Reposition it carefully only when the mounting screws are completely free, and do not hold the Load Cell when retightening the screws. Otherwise, the cell may be seriously damaged.

Because this measurement concerns surfaces, their condition is critical to the values obtained. Be sure to keep all test surfaces clean and free from all contaminants, including fingerprints.

Some materials have COF values that are strongly dependent on the time of repose of the sled. For such materials, try to minimize, or at least be consistent, in the time elapsing from application of the normal force and the start of the platen motion.

Running a Test - COF

Select an Averaging Time with the appropriate switch. The 10, 20 and 50 seconds correspond to 1, 2 and 5 inches of platen travel, respectively.

Place the Sled far enough out to take up most, but not all of the slack in the Tether. *

Depress the Start/Reset Button. The Platen should start moving and the Platen Moving Light should come on. After approximately 4 seconds, the LED Indicator will come on, indicating that the force signal is being integrated with respect to time (or distance). The LED Indicator should remain on for the time selected. When the LED goes off, move the Meter Select Switch successively to Peak and to Average, making note of the steady meter readings obtained.

The Peak value of COF is also known as the Static value. Average value of COF is also known as the Kinetic value. Remember that if either value exceeds 199.9 the display will blank. The analog memories for Peak and Average will retain their values accurate to 1% for 2 minutes or more.

The Platen Drive may be allowed to run to its normal limit of travel or it may be stopped instantly by depressing the Platen Stop switch.

After installation of a new set of specimens, operate the Start/Reset Button, which will reset the memories and start the Platen Drive for the next test.

The Meter Select Switch may be moved at any time, without affecting the data, and may be left in any position during a test..

*See Supplemental Notes SW-1

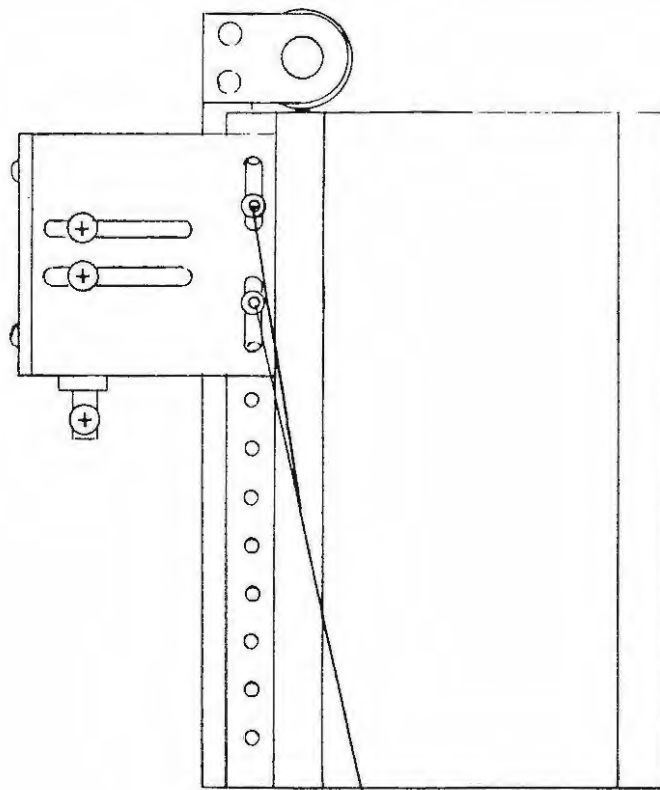


FIG 4

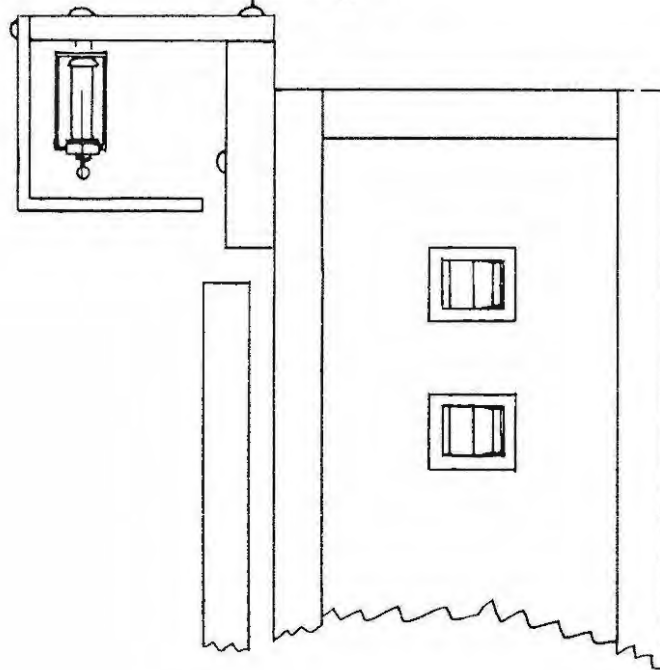


FIG 3

REMOVE TO
DISMOUNT FOR
CALIBRATION
(2 SCREWS
8-32 ALLEN)

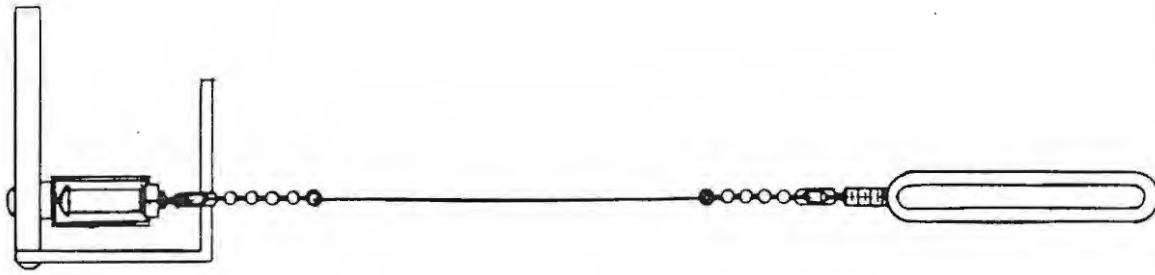


FIG 6

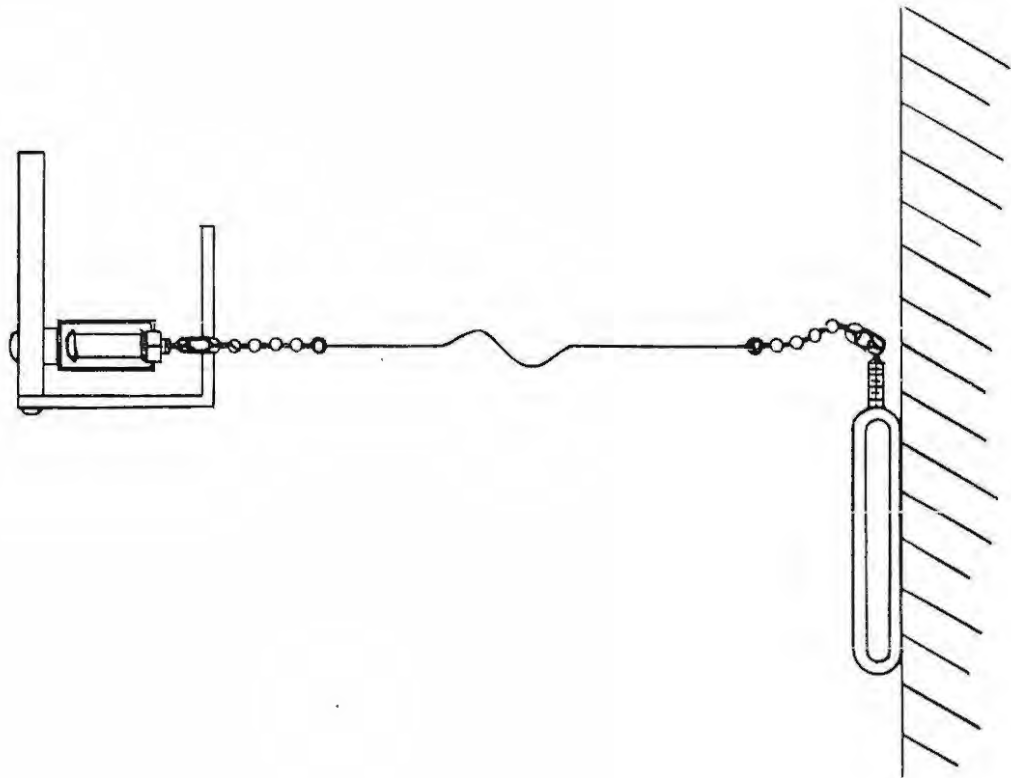


FIG 5



RECOMMENDED TEST : NONWOVENS ABSORPTION

A. SCOPE

This test method covers the evaluation of the behaviour of nonwoven fabrics in the presence of liquids. This can be done by testing for :

- liquid absorbency time
- liquid absorptive capacity
- liquid wicking rate (capillarity)

It should be noted that these different aspects of absorbency relate to various end uses of the tested products.

- **Liquid absorbency time** is the time required for a sample of absorbent material to become completely wetted by the test liquid. It is the time required for a material to imbibe a liquid into its interior structure.
- **Liquid absorptive capacity** measures the weight of liquid that is absorbed per unit weight of the test absorbent or by percentage, after a standard time or after the time needed to wet completely the material.
- **Liquid wicking rate** measures the capillarity of the test material, i.e., the speed at which the liquid is transported into the fabric.

B. TESTING CONDITIONS

Condition the sample for 24 h and test at 65 ± 2 % relative humidity and $20 \pm 2^\circ\text{C}$. (ERT 60.1-75).

A. LIQUID ABSORBENCY TIME

C. RECOMMENDED METHOD

ASTM D 1117-80

«Testing nonwoven fabrics».

The liquid absorbency time test measures the time required for the complete wetting of a specimen strip (5 g) loosely rolled into a cylindrical wire basket (3 g) and dropped on to the surface of the liquid from a height of 25 mm.

In this method the liquid comes into contact with all surfaces of the sample.

N.B. Specifications for gauge and dimensions of the basket given in ASTM D 1117-80 § 5.2, make it impossible to achieve the necessary weight. The weight of the basket is, however, all important. The gauge should therefore be adapted accordingly.

D. EQUIVALENT METHODS

None

E. AMENDMENTS

Where the word «water» is used, «liquid» should be inserted.

F. EQUIPMENT

1. Cylindrical Wire basket open at one end. Height 8 cm, diameter 5 cm, weight 3 g (constructed of suitable gauge wire to achieve the weight of 3 g, e.g. 0,5 mm diameter stainless steel wire). The mesh should be approx. 2 cm square.
2. Container for liquid.
3. Stop-watch.

10.1-72

G. PROCEDURE AND WORKED EXAMPLE

PROCEDURE	WORKED EXAMPLE
1. Cut 5 samples in machine direction 76 mm wide and of sufficient length such that they each weigh $5 \pm 0,1$ g. These strips should be equally spaced across the sheet.	76 mm x 165 cm
2. Condition to 65 ± 2 % R.H. and $20 \pm 2^{\circ}\text{C}$. for 24 h (ERT 60.1-75).	
3. Loosely roll a sample and place in the wire basket.	
4. Drop basket, on its side, from a height of 25 mm from the liquid surface into a container of liquid at room temperature.	
5. Record time taken for sample to wet completely by watching the progress of absorption.	6,3 s
6. Repeat with the other 4 samples.	6,0 s 6,7 s 6,4 s 6,2 s
7. Calculate average liquid absorbency time	6,3 s

H. REPORT

1. Type of material .
2. Conditions of the sample (ERT 60.1-75) .
3. Dimensions of sample .
4. Test method used and any deviations from the standard method .
5. Surface tension and temperature of liquid .
6. Average and liquid absorbency time and standard deviation .

B. LIQUID ABSORPTIVE CAPACITY

C. RECOMMENDED METHOD

DIN 53923. «Determination of water absorption of textile fabrics».

The absorptive capacity method measures the amount of liquid stored within the test specimen during a certain time.

This method effectively measures the liquid stored within the test specimen itself as the drainage has occurred vertically. The drainage time is quite short, which is practical as well as especially important when very volatile liquids are used.

N.B. ASTM D 1117-80, § 5.4 differs mainly in the drainage system (horizontal draining of various layers). It therefore serves slightly different purposes.

Drainage time in this method should be shortened and brought back to two minutes.

D. EQUIVALENT METHODS

None

E. AMENDMENTS

Where the word «water» is used, «liquid» should be inserted.

F. EQUIPMENT

1. Wire gauze made from stainless steel of at least 120 mm x 120 mm with a metal frame. Gauze made from test sieve netting 2 DIN 4188.
2. Dish with test liquid for laying in the wire gauze with the sample attached.
3. Suitable weighing glass with cover.
4. Stop-watch.

G. PROCEDURE AND WORKED EXAMPLE

PROCEDURE	WORKED EXAMPLE														
1. Cut 5 samples 100 mm x 100 mm from the sheet. If an individual sample weighs less than 1 g, several samples are laid on top of each other until the pile weighs at least 1 g.	Sample 100 mm x 100 mm = approx. 0,40 g (Gram- mage = 40 g/m ²) so piles of 3 used														
2. Condition according to ERT 60.1-75.															
3. Weigh the sample (or pile) to 0,01 g, using weighing glass with cover.	Weight sample = 1,21 g														
4. Place sample or sample pile on the stainless steel gauze, fastening it at the edges with clips.															
5. The gauze with sample is placed 20 mm below liquid surface in a suitable dish at room temperature. It is best to introduce it obliquely in order to avoid trapping air bubbles.															
6. After 60s remove sample and gauze.															
7. Remove all clips but one at one corner.															
8. Hang freely to drain vertically for 120 ± 3s.															
9. Take sample off gauze and place in weighing glass.	Wt wet sample = 7,72 g														
10. Repeat for the other 4 samples.															
	<table> <tr> <th>Dry Wts</th><th>Wet Wts</th></tr> <tr> <td>1,09 g</td><td>6,98 g</td></tr> <tr> <td>1,22 g</td><td>7,32 g</td></tr> <tr> <td>1,31 g</td><td>8,65 g</td></tr> <tr> <td>1,28 g</td><td>8,32 g</td></tr> <tr> <td>1,222 g</td><td></td></tr> <tr> <td>7,798 g</td><td></td></tr> </table>	Dry Wts	Wet Wts	1,09 g	6,98 g	1,22 g	7,32 g	1,31 g	8,65 g	1,28 g	8,32 g	1,222 g		7,798 g	
Dry Wts	Wet Wts														
1,09 g	6,98 g														
1,22 g	7,32 g														
1,31 g	8,65 g														
1,28 g	8,32 g														
1,222 g															
7,798 g															
11. Calculations and results															
a. Average weight of 5 original samples (M_k)															
b. Average weight at end of test (M_n)															
c. Liquid absorptive capacity (W_A)															
$W_A = \frac{M_n - M_k}{M_k} \times 100$	$\frac{(7,798 - 1,222) \times 100}{1,222}$														
	= 538 %														

10.1-72

H. REPORT

1. Type of material .
2. Conditions of sample (ERT 60.1-75) .
3. Dimensions of sample .
4. Test method used and any deviations from the standard method .
5. Surface tension and temperature of liquid .
6. Average liquid absorbency time and standard deviation .

C. LIQUID WICKING RATE

C. RECOMMENDED METHOD

DIN 53924. «Velocity of soaking water of textile fabrics».

The capillarity method measures the rate of vertical capillary rise in test specimen strips suspended in the test liquid.

N.B. This test method, however, basically measures the rate of absorption from the edges of the specimen and difficulties could be encountered when judging and comparing the results obtained with **anisotropic** materials.

Capillarity should then be measured in both directions.

D. EQUIVALENT METHOD

None

E. AMENDMENTS

Where the word «water» is used, «liquid» should be inserted.

F. EQUIPMENT

1. Base plate (with levelling screws).
2. Dish for the test liquid.
3. Horizontal support which can be adjusted along a vertical support.
4. Clamps on the above horizontal support for fastening the samples.
5. Measuring rods with a millimetre scale fixed vertically on said horizontal support.
6. Stop-watch.
7. Glass rods 4-5 mm diameter and 30 mm length.

G. PROCEDURE AND WORKED EXAMPLE

PROCEDURE	WORKED EXAMPLE
1. At least 5 samples are cut in the machine direction and the cross direction respectively. Size 30 mm wide x 250 mm long.	5 MD samples 5 CD samples Each 30 mm x 250 mm
2. Two holes, about 5 mm diameter, are punched out of the narrow end of each strip 5 mm from narrow and longitudinal sides.	
3. Condition according to ERT 60.1-75.	
4. Clamp vertically to the horizontal support with holes downwards.	

5. Slot a glass rod through the two holes to weigh the samples and keep them vertical.																													
6. Place sample parallel to the mm measuring rod and projecting 15 mm below the lower end of the rod.	1 sample from MD																												
7. Lower horizontal support until zero point of measuring rod touches the liquid surface (the lower sample edge is then 15 mm below the surface).																													
8. At this moment start stop-watch.																													
9. Record height of capillary rise after 10, 30, 60 (and 300 s if required).	<table><tr><th>Time s</th><th>Capillary Rise, mm</th></tr><tr><td>10</td><td>33</td></tr><tr><td>30</td><td>48</td></tr><tr><td>60</td><td>57</td></tr></table>	Time s	Capillary Rise, mm	10	33	30	48	60	57																				
Time s	Capillary Rise, mm																												
10	33																												
30	48																												
60	57																												
10. Repeat with the other 9 samples.																													
	<table><tr><th colspan="4">Sample MD, mm</th></tr><tr><th></th><th>10 s</th><th>30 s</th><th>60 s</th></tr><tr><td>2</td><td>30</td><td>45</td><td>55</td></tr><tr><td>3</td><td>29</td><td>50</td><td>50</td></tr><tr><td>4</td><td>32</td><td>51</td><td>59</td></tr><tr><td>5</td><td>37</td><td>46</td><td>60</td></tr></table>	Sample MD, mm					10 s	30 s	60 s	2	30	45	55	3	29	50	50	4	32	51	59	5	37	46	60				
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11. Calculations and results																													
a. average liquid wicking rate — machine direction	<table><tr><th>Time s</th><th>Capillary Rise, mm</th></tr><tr><td>10</td><td>32</td></tr><tr><td>30</td><td>48</td></tr><tr><td>60</td><td>56</td></tr></table>	Time s	Capillary Rise, mm	10	32	30	48	60	56																				
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b. average liquid wicking rate — cross direction	<table><tr><th>Time s</th><th>Capillary Rise, mm</th></tr><tr><td>10</td><td>25</td></tr><tr><td>30</td><td>33</td></tr><tr><td>60</td><td>43</td></tr></table>	Time s	Capillary Rise, mm	10	25	30	33	60	43																				
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10	25																												
30	33																												
60	43																												

10.1-72

H. REPORT

1. Type of material .
 2. Conditions of the sample (ERT 60.1-75) .
 3. Dimensions of sample .
 4. Test method used and any deviation from standard .
 5. Liquid specifications .
 6. Average liquid absorbency time and standard deviation machine direction .
 7. Average liquid absorbency time and standard deviation cross direction .
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TENSILE STRENGTH 20.1-73

RECOMMENDED TEST : NONWOVENS TENSILE STRENGTH

A. SCOPE

This test method examines the behaviour of nonwoven fabrics when subjected to tensile stress.

Values for the tensile strength and elongation of a specified width and length of material are determined by applying a force longitudinally at a specified constant rate of extension.

The recorded curves have one or other of the following configuration :

(Curve : mirror image)

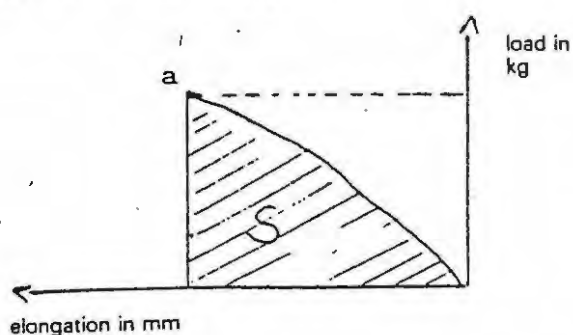


Figure 1

«a» represents the point of maximum load and the corresponding elongation.

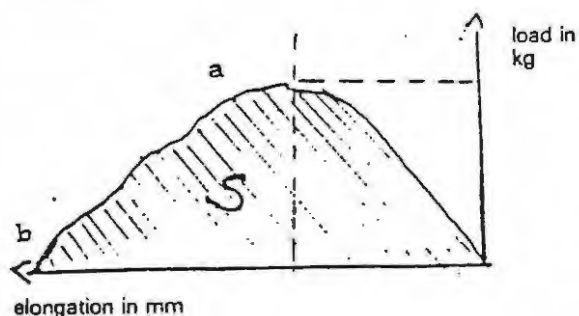


Figure 2

«a» represents the point of maximum load and the corresponding elongation.

«b» represents the total elongation of the product at break.

Tensile strength : this is the maximum load, i.e. the load recorded at point «a».

Elongation at maximum load : this is the elongation recorded at point «a» (In the case of Figure 1, it corresponds to the elongation at break.)

Total elongation of the product at break : in the case of Figure 2 this is the elongation at point «b».

N.B. The method may, if required, be extended to include the determination of the rupture energy of the fabric under test.

Breaking length : this is the length at which a strip of nonwoven of any uniform length, assumed to be suspended by one of its ends, breaks under its own weight.

Total rupture energy : this is the energy needed to bring that strip to break; it is represented by the area comprised under the whole tensile curve as in Figure 2.

This data is particularly useful in research and development work and also for characterising some products.

20.1-73

B. TESTING CONDITIONS

Condition the sample for 24 h and test at 65 ± 2 % relative humidity and $20 \pm 2^\circ\text{C}$. (ERT 60.1-75).

N.B. In the case of wet tensile strength testing, samples should be in a completely saturated state after immersion, normally in distilled water. If any other agent is used, this should be specified.

C. RECOMMENDED METHOD

DIN 53857. «Tensile strength testing on a strip of textile fabric».
(as amended in E below).

D. EQUIVALENT METHODS

ASTM D 1682-64 (Reapproved 1975)

AFNOR NF G 07-001 (§ 2 : 2,8, 2,6) (January 1973) } (as amended in E below)

BS 2576 : 1977

E. AMENDMENTS

a) The fabric samples should be 50 mm wide and of sufficient length to allow a tensiometer jaw separation of 200 mm. By these means, risks due to the local heterogeneity of nonwovens, or to undue cutting of long-fibre nonwoven fabrics, are avoided.

b) A constant rate of extension should be applied. This should be 100 mm/min.

N.B. Anomalies can occur when testing some spunlaid and glass fibre nonwovens, which can require deviations from the said test procedures.

c) Pretension is not necessary, provided the elongation measurement is taken from the point where the load curve leaves the zero line.

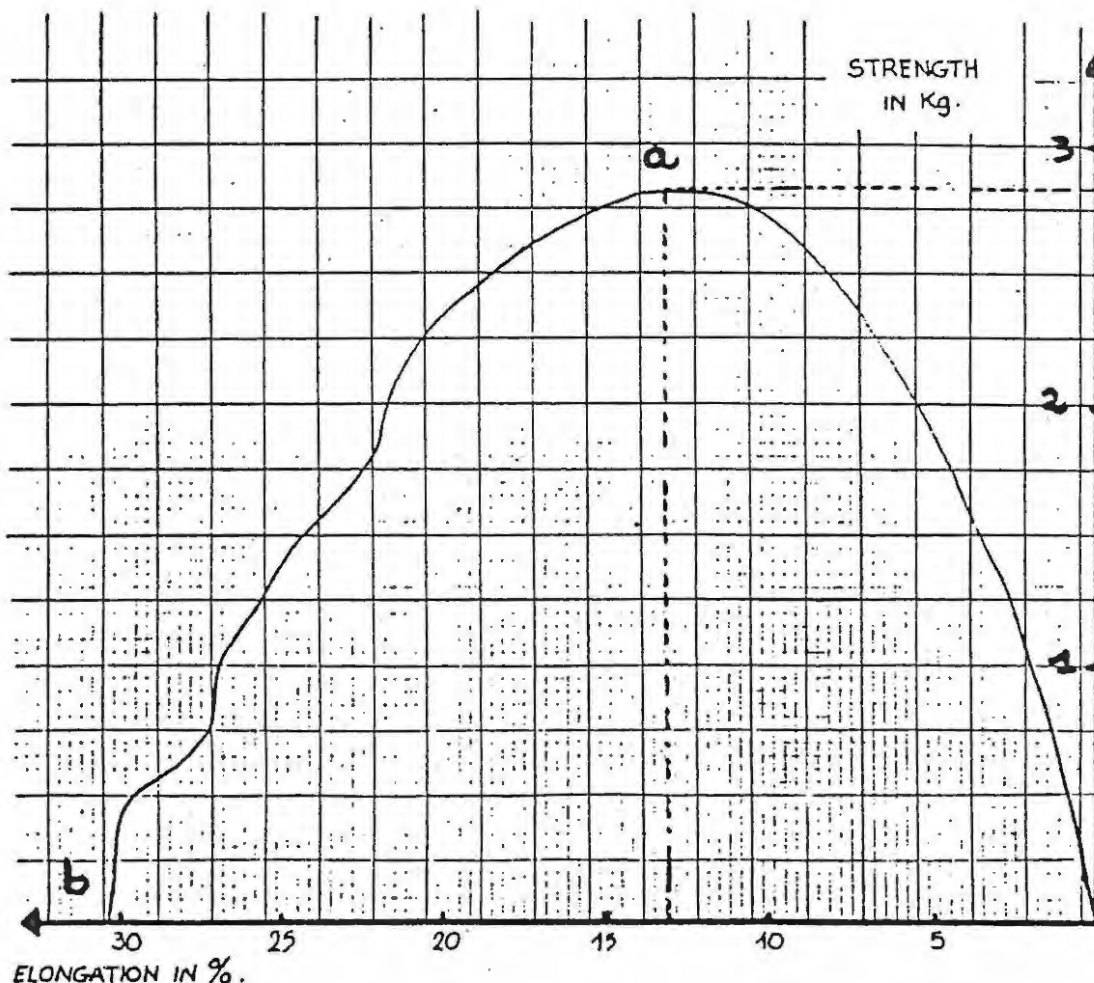
F. EQUIPMENT

- A laboratory paper cutter, not scissors, as samples must be cut out absolutely cleanly.
- An electronic dynamometer of the constant rate of extension type, equipped with jaws 50 mm wide and fitted with a system for recording load-elongation curves.

G. PROCEDURE AND WORKED EXAMPLE

PROCEDURE	WORKED EXAMPLE
1. Cut out 10 samples for each direction (machine direction and cross direction), according to ERT 130.1-80. They must be cut out cleanly, 50 mm wide, and more than 200 mm long so that a distance of 200 mm can be obtained between the jaws of the dynamometer.	250 mm x 50 mm
2. Condition samples according to ERT 60.1-75.	Weight 0,60 g
3. Weigh and calculate weight per m^2 according to ERT 40.2-77.	Weight per m^2 : $\frac{0,60 \times 10^6}{250 \times 50} = 48 \text{ g/m}^2$
4. Place the sample between the jaws of the apparatus, these being 200 mm apart.	

5. Apply traction at 100 mm/min. and record the load-elongation curve, as shown below. (Curve : mirror image)



6. Establish the scale of the load-elongation curve.
7. Read off on the curve the breaking strength (point «a»).
8. Read off on the curve the elongation at the maximum load (point «a»).
9. Read off on the curve the total elongation at break (point «b»).
10. Calculate the breaking length.

Breaking length is calculated by the following formula :

$$L = \frac{F \times 10^6}{50 \times 9,8 \times g}$$

where :

L = breaking length in metres
 F = breaking resistance in N of a 50 mm wide strip
 g = weight per unit area, expressed in g/m², of the nonwoven being tested.

Load : 1 cm = 0,29225 N
 Elongation : 1 cm
 = 2,345 %
 = 4,69 mm
 deformation of the sample

$$F = 27,7 \text{ N}$$

$$a = 13,1 \%$$

$$b = 30,3 \%$$

$$L = \frac{27,7 \times 10^6}{50 \times 9,8 \times 48} = 1176 \text{ m}$$

$$L = \frac{27,7 \times 10^6}{50 \times 48 \times 9,8} = 1178 \text{ m}$$

20.1-73

11. Calculate the total rupture energy (energy at break). The determination is made from the total load-elongation curves, i.e. the area under the curves.
This area (A) has to be measured and the scale of the graph (a) determined.

The formula for the total rupture energy (E) is therefore (expressed in joules) :

$$E = A \times a \times 9,81 \times 10^{-5} \text{ J}$$

12. Calculate the index of energy at break :

$$I = \frac{E}{g \times l \times w}$$

E = rupture energy in J.

g = weight per unit area of nonwoven, expressed in g/m² of nonwoven tested

l = length of sample tested (200 mm)

w = width of sample tested (50 mm)

13. Carry out the operation 10 times for each direction (machine direction and cross direction) and give the average of each characteristic in each direction.

$$A = 85,19 \text{ cm}^2$$

$$a = 1 \text{ cm}^2 = 292,25 \times 0,469 \\ = 137,07 \text{ g} \times \text{cm}$$

$$E = 85,19 \times 137,07 \times 9,81 \\ \times 10^{-5}$$

$$E = 1,145 \text{ J}$$

$$I = 1,145$$

$$48 \times 0,2 \times 0,05$$

$$I = 2,39 \text{ J/g}$$

H. REPORT

1. Type of the material tested .
2. Dimensions of the sample .
3. Condition of the sample (wet, dry) .
4. Test method used, and any deviations from standard .
5. Indicate whether or not **pretension** was used .
6. Average and standard deviation tensile strength (in N), specifying direction of test .
7. Average and standard deviation percentage elongation, at maximum load, specifying direction of test .
8. If necessary, specify rupture energy and index of rupture energy.

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