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THE MANUFACTURE PROPERTIES AND USES OF INFLATED VISCOSE RAYON FIBRES

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ABSTRACT

Methods of creating inflated fibres are reviewed, and processes which have been operated commercially are highlighted. The development work on the sodium carbonate route which lead to the commercial large scale production of hollow rayon and the so-called super-inflated rayon is described. The lesser known members of the inflated fibre family are mentioned, and their position in the inflation heirarchy is discussed.

The actual and potential applications of the whole family of products in knitted, woven, per-making, and surgical products are reviewed.

A REVIEW OF INFLATION PROCESSES

The early commercial rayons were intended as substitutes for natural silk, although most suffered from the properties of harsh handle, hard metallic-type lustre and lack of cover or insulation, all these properties being uncharacteristic of the true product. To help eliminate some of these problems attempts were made to produce hollow rayons.

Although hollow rayon fibres ("straws" etc) were produced as early as 1910 by a "hot-pin" process (1), the idea of incorporating an inert gas into the viscose solution to generate inflated fibres was not patented until 1920 by L. Drut (2). J. Rousset developed this idea to include the use of gas generating additives such as metal bonates or volatile liquids, which elop gas or vapour within the spinning fibres by the action of the acid or the heat from the spin bath (3). Courtaulds later patented the idea of producing inflated fibres without added agents, but using a low soda viscose (4).

These early ideas were improved and developed (5-8) but most of the subsequent ideas were simply refinements of the two basic processes of emulsification/dissolution and gas generation. One notable exception was the use of electrolysis of the viscose just prior to spinning to controllably generate oxygen bubbles within the fibres (9). However, this process never became commercial! Other notable developments are listed below:

1925 - Two bath process. Coagulation and carbonate impregnation followed by regeneration and inflation (10).

1926 - Twin gear pumps, the second running faster than the first, to introduce gas bubbles (11).

1926 - Use of magnesium sulphate in spin bath to promote more uniform inflation and more circular cross-section (12).

1926 - Vacuum evaporation of a volatile fluid (13).

The first commercial inflated fibres evolved in the 1920s under the "Celta" brand-name, largely by the Alsa company. Other types were also available, known by the names of "Luft-seidi" and "Soie Nouvelle". These early fibres were used to create light yarns with high covering power. The early processes suffered from difficult spinning and from lack of inflation control (a lot of the fibres ended up as flattened tubes and some were not inflated at all).

In 1942 "Bubblfil" was produced by du Pont de Nemours. This process used air injection just prior to spinning the viscose through very large jet holes to create filaments with spaced bubbles. The fibre was popular as a bouyancy aid in life jackets, pontoons and rafts during the war. Also it was considered to have good insulating properties and was used for this purpose in aviators' uniforms and in sleeping bags. Production stopped in 1943.

Development slowed dramatically after the early work. In the 1940s several patents emerged from America (14,15). They were essentially similar to the earlier reports and were not commercialised. It was not until 1960 that further patents, originating in America, were issued relating to the production of flat inflated fibres which were particularly useful for paper-making (16). The processes described used the gas emulsion technique, the emulsions being stabilised by the use of surfactants in the viscose. These developments led to the commercial production of "RD101" in America by the American Viscose Corporation. Soon after Courtaulds introduced "PM1" and "PM2" fibres made by a sodium carbonate route (17). PM1 fibre was designed for use in high quality papers. PM2, a slightly less inflated version of PM1, was developed for medical nonwovens, particularly nappies and sanitary towels. At about the same time several patents by Japanese workers were issued for similar paper-making fibres made by sodium carbonate routes, but also incorporating "water swellable chemicals" within the fibres (18). A lot of work was carried out to determine the important factors for the production of good paper-making fibres - notably Treiber, Dumbleton and Ehrengard (19,20). However, substantial markets in these areas never materialised and production of all of these fibre developments has now stopped.

Courtaulds continued work on the sodium carbonate route with particular emphasis on controlling the inflation process to produce distinctly different fibre types in 100% form. A permanently hollow fibre was developed by using modified viscose and high salt figure (21 "Viloft") and was commercialised in 1976. Later two types of highly inflated and collapsed fibre types were produced using unmodified viscose at low salt figure and spinning into a high temperature, high acid spin bath (22,23 "SI Fibres"). One of these was commercialised in 1979. In 1984 a product derived from Viloft ("Courcel") was introduced in America.

During this same period other companies have concentrated effort into improving the control aspects by thoroughly investigating the various effects of spin bath and viscose composition, notably Mitsubishi, using a high carbon disulphide, no carbonate route (24) and International Paper Co. using a carbonated viscose, high carbon disulphide route (25).

DEVELOPMENT OF MANUFACTURING METHODS

The Various Fibre Types Isolated

Figure 1 illustrates the fibre cross-sections of the various inflated fibres isolated to date. Essentially the fibre types differ only in the extent to which the initially formed filaments have been inflated by the spinning gases. Thus in progressing from standard rayon through to super-inflated fibre the uncollapsed tube diameter gradually increases and the cellulose wall thickness decreases. The effects can be seen more clearly in the micrographs in Appendix 1.

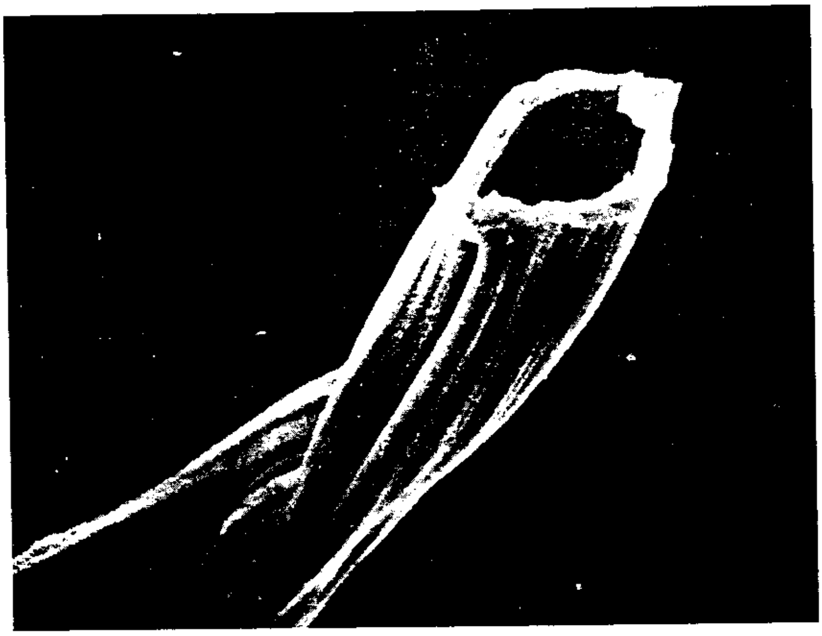
FIGURE 1

INFLATED VISCOSE RAYONS AND THEIR PROPERTIES

CROSS SECTION	NAME	WATER IMBIBITION %	DRY TENACITY gms./den.	DRY EXTENSION %	COMMENTS
	STANDARD RAYON.	90 – 100	1·9-2·5	18–30	Uninflated rayon for comparison.
	–	110	–	–	Not commercial.
	VILOFT	120–140	2·2-2·5	13-15	High bulk cotton-like textile fibre.
	"PM2 FIBRE"	150–160	1·8 - 2·0	20-25	Self bonding fibre for wet laid non-wovens. Not commercial.
	"PM1 FIBRE"	160–180	1·4- 1·8	20-25	Self bonding fibre for quality papers. Not commercial.
	SUPER INFLATED FIBRE.	190-350	1·0-1·4	30-50	Highly absorbent, opaque and bulky fibre for non wovens.

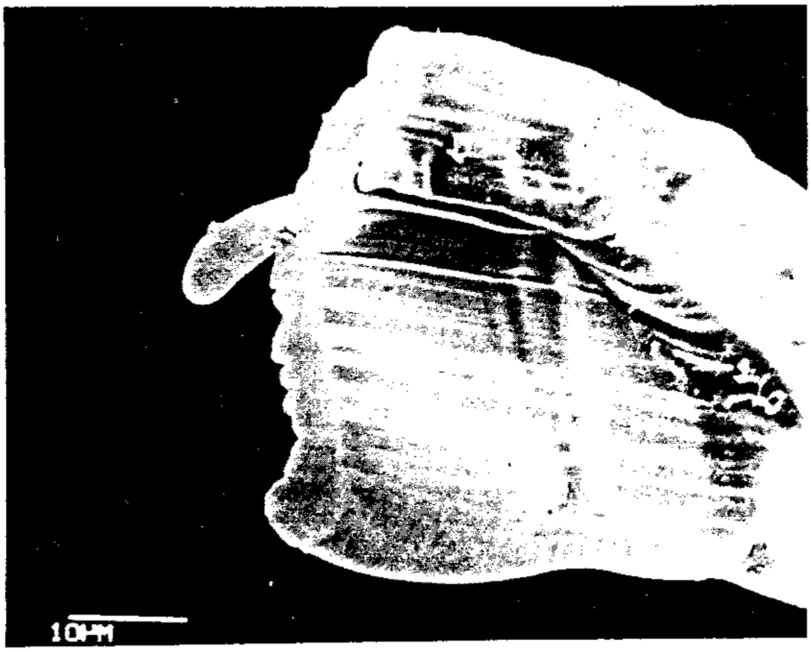
APPENDIX 1

STANDARD RAYON

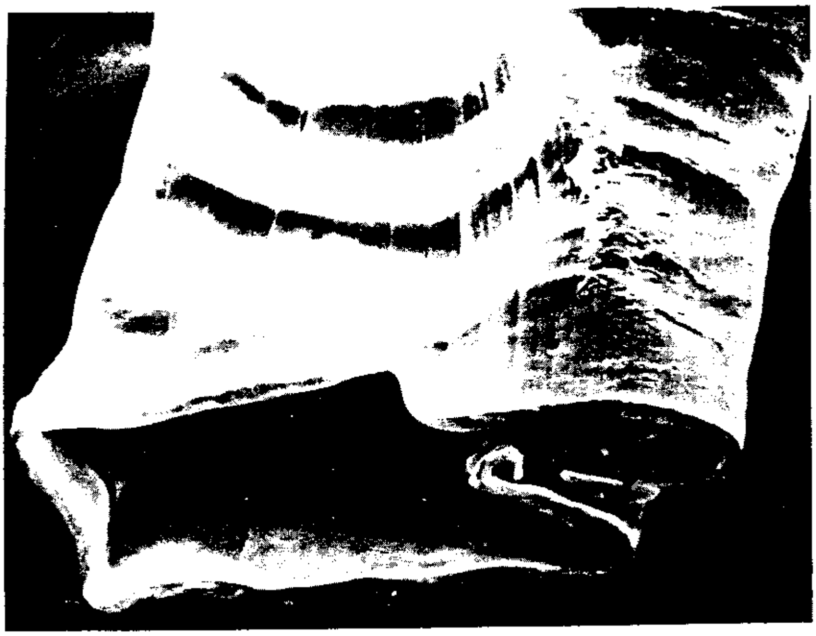


VILOFT

APPENDIX 1 (continued)



SI (DRY)



SI (WET)

The Mechanism of Inflation

To understand how these various forms are produced and how the inflation process comes about, it is probably worth asking in the first instance why <u>all</u> rayon fibres are not inflated. The viscose process generates sodium carbonate and sodium trithiocarbonate by carbon disulphide by-product reactions:-

$$3CS_2 + 6NaOH \rightarrow Na_2CO_3 + 2Na_2CS_3 + 3H_2O$$

These by-products will decompose in the spin bath to give carbon dioxide and hydrogen sulphide. By performing calculations including viscose γ number, carbon disulphide and cellulose content it is possible to estimate the amount of these gases which will be liberated in acid. The answer is approximately 3→4L for every 1L of viscose (at NTP). This then is more than enough gas to inflate all of the fibres produced, and so why do standard rayon fibres not contain bubbles? The answer to this question is not entirely certain but it is thought that bubbles will only form and grow within a filament when certain conditions are satisfied:-

1) The concentration of the gases within the filament must be greater than the saturation concentration.

2) A nucleus for formation must be present to avoid super-saturation (often thought to be a gas bubble itself or a small particle).

3) The formed bubble must develop sufficient "pressure of formation" (from the transport of gas into it) to overcome both the osmotic pressure within the forming filament and the Laplace pressure created by the bubble surface.

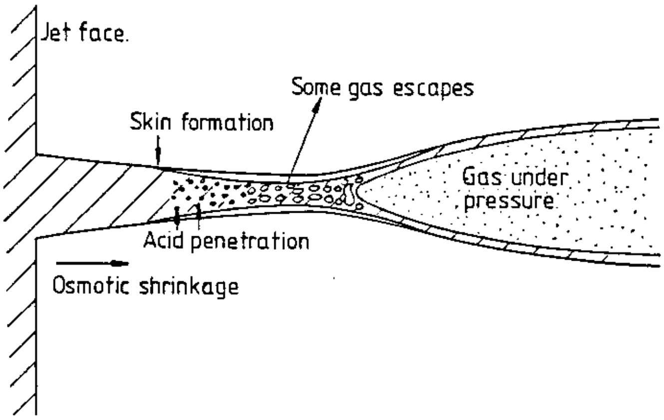
4) Finally the bubble must be trapped within the fibre by the outer membrane or skin. This item incorporates a time element - it is no good producing the right bubbles before the fibre can "hold" them.

The theory is then that under normal circumstances all of these conditions are not satisfied (quite fortuitously!) and that the extra pressure and volume of carbon dioxide from added sodium carbonate is required to overwhelm conditions 1 and 3. Link this with suitable conditions of skin formation, coupled with <u>continued</u> gas generation (helped by the extra sodium carbonate) to satisfy condition 4 and an inflated fibre results. It is worth noting that inflated fibres can be generated without added sodium carbonate by using extreme viscose and spinning conditions - high carbon disulphide charges, low viscose soda etc., and this observation fits with the theory that we are indeed dealing with a simple balance of conditions and not with a unique effect resulting from the use of extra sodium carbonate.

Thus, to summarise, when the viscose thread enters the spin bath, the normal processes of coagulation and regeneration commence. Acid penetrates the outer skin to generate carbon dioxide and hydrogen sulphide within the forming filament, and via the process described above bubbles begin to form. Some gas escapes but further bubbles are generated. Eventually as the skin thickens bubbles begin to coalesce and become trapped to form a continuous "stationary" bubble at a certain distance from the jet face. The pressure within the bubble will depend upon the rate of evolution of the gas, the permeability of the fibre wall, the spin bath temperature and so on, and this pressure will in turn alter the extent to which the filament becomes inflated. Figure 2 shows this process in a diagrammatic form.

<u>FIGURE 2</u>

<u>STATIONARY BUBBLE THEORY OF FILAMENT INFLATION</u>

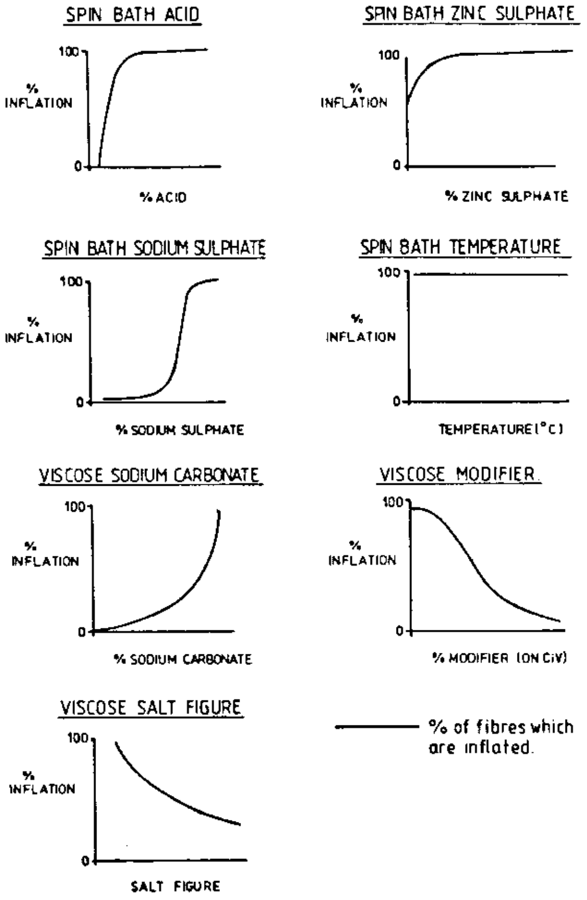


Factors Affecting Inflation and Hence Fibre Shape

Having dealt with the basic inflation mechanism it is now possible to discuss in more detail the effects on this process of the various viscose and spinning parameters. Evidently, as the process is dependent on so many variables which are inter-related, it is not possible to give a universally applicable description of the effects involved. For the purpose of this illustration, therefore, a simple examination will be made of the <u>trends</u> only.

Figure 3 shows the approximate effects of the parameters considered, on the level of inflation within the fibres produced. The level of inflation is considered simply here as the percentage of fibres which have been inflated in a fibre cross-section assessment, i.e. all of those fibres which no longer possess a standard rayon type cross-section, and have been inflated and remain hollow or have subsequently collapsed. Generally speaking, as the "% inflation" increases we move into production conditions suitable for fibres further down the inflation hierarchy as illustrated in Figure 1.

FIGURE 3
FACTORS AFFECTING THE INFLATION PROCESS



Thus, dealing with the various factors individually, it is found that percent inflation increases rapidly as the acid strength rises beyond a certain critical level. The acid in the spin bath has a triple effect - in coagulation, regeneration and rate of carbon dioxide evolution. Increasing acid strength therefore promotes faster and less permeable skin formation, coupled with more rapid and extensive carbon dioxide inflation.

The zinc sulphate content of the spin bath affects the extent of coagulation and zinc cellulose xanthate formation in the skin. The formation of a tougher, denser and hence less permeable skin at high zinc levels gives rise to increased inflation. However, the effect which zinc is thought to have on slowing down the penetration of acid into the filament core must not be forgotten. This feature may explain why the zinc effect is not as marked as might be expected.

The sodium sulphate level affects the degree of coagulation and shrinkage, and hence as the sodium sulphate level increases a denser barrier to gas escape is produced. Further, this action probably takes place prior to any gas generation, as initially most acid is used up simply in the neutralisation of the viscose soda. It is found in fact that inflation does not really commence until a relatively high level of sodium sulphate is used - approximately 18 - 20%.

The spin bath temperature has little effect on the level of inflation, although it is evident that this parameter will influence the rate of the reactions taking place and the extent of expansion of the gases evolved. It is found that, although the percentage of inflated fibres remain unchanged, the extent to which these fibres inflate does increase with increasing temperature.

The influence of sodium carbonate on the inflation process has already been discussed. It is found in practice that small levels of additional sodium carbonate do not affect the process markedly, and levels as high as 3 or 4% are required to readily inflate the fibres produced.

The level of modifier in the viscose and the viscose salt figure both influence inflation in similar ways. High modifier or salt figure gives reduced inflation. Modifier, in the presence of zinc, slows down the ingress of acid and hence the regeneration process - in a similar way, high salt figure viscose requires greater salt levels for coagulation and more acid for regeneration.

These then are the major factors which influence the inflation process. Other factors which have not been mentioned either have little influence on the process or the influence can be predicted from the explanations already given. Thus by altering the conditions to those for high or low inflation, and by selecting appropriate conditions for stability, it is possible to produce all of the fibres illustrated in Figure 1.

PROPERTIES AND USES OF INFLATED FIBRES

As has been discussed already, the essential difference between the various inflated fibres is in the extent of inflation which has taken place during spinning. Most of the special properties of these fibres, and the changes in properties on moving through the inflated fibre hierarchy, can be explained by the changes in inflation level. However, some properties are unique to a particular fibre. This can simply be a reflection of the special production conditions necessary for an individual type, or it can be a result of the peculiar physical or engineering properties of a certain cross-sectional shape. In general though, increased inflation results in increased fibre surface area and hence increased bulk and moisture absorbency.

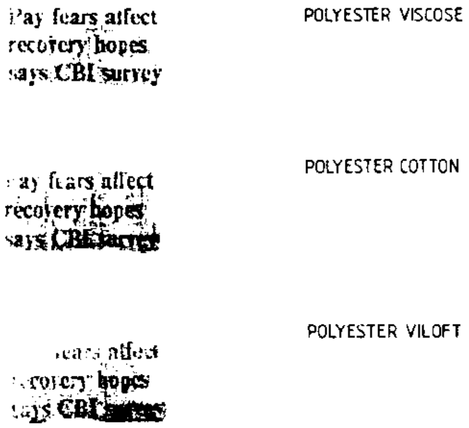
Before discussing the various fibres individually it would be useful to refer back to Figure 1, firstly as a reminder of the cross-sections available and secondly to examine some of the basic fibre properties and how they change with cross-sectional shape. It can be seen that, in terms of water imbibition (that water which is absorbed within the fibre structure and on the fibre surface), there is a steady trend of increasing absorbency as we move down the list. The inflation process tends to be detrimental to fibre tensile properties, partly because of the nature of the process and partly because of the production conditions necessary for generating the desired fibre properties. For example, SI Fibre has very low tenacity but high extensibility partly because of the low stretch nature of the recipe. On the other hand, Courcel is tailored to have a relatively high tenacity - putting it somewhat "out of line" in the inflated fibre trends.

Courcel

Courcel was developed to fill a market need for a fibre with high bulk or cover, full handle and good absorbency, for use particularly with polyester. Despite the good properties of polyester, consumers react unfavourably towards its "synthetic" handle and wear properties and the generally limp and lean nature of its fabrics, even when blended with cotton. Table 1 and Figure 4 illustrate some of the special Courcel properties. For example, the permanent tubular structure gives an unusually high torsional rigidity - higher than that of cotton. This, coupled with the high bending rigidity characteristic of a "tube" gives rise to a desirably stiff, full handle. The bulk and cover properties, coupled with a light weight, are brought about by the effectively low density of Courcel. In fact Courcel at 1.5 denier behaves just like a 3 denier fibre but without the extra weight.

FIGURE 4

THE BENEFIT OF VILOFT ON FABRIC COVER



	POLYESTER/ VILOFT.	POLYESTER/ COTTON.	POLYESTER/ STANDARD RAYON.
LIGHT TRANSMISSION (%)	5·2	5·7	6·7

TABLE 1

SPECIAL VILOFT CHARACTERISTICS

	Viloft	Standard Rayon	Cotton
Torsional Rigidity x 10^{-9} ($Nm^3\ tex^{-1}$)	3·7	1·5	3·2
Air Permeability * ($m^3\ minute^{-1}$) x 10^{-3}	2·2	6·5	4·0
Effective Density ($g\ cm^{-3}$)	1·1	1·5	1·5

* Using Micronair Apparatus

On aspects of comfort there are many factors which play a part, but the popularity of Courcel is very clear. Table 2 shows the preferred handle of Courcel in woven fabrics of a given yarn count, both before and after washing. Moisture absorbency and moisture transport or wicking are regarded as being significant in the total "comfort measure" of a fabric, and here again Courcel scores very highly - see Figures 1, 5 and 6, plus Table 3.

TABLE 2

HANDLE ASSESSMENT OF VARIOUS WOVEN FABRICS

Judges; all fabrics with 50% polyester; showing the percentage of judges who put the indicated fabrics as "1st choice."

	Standard Rayon	Cotton	Viloft
Original samples	25	25	50
Washed samples	29	7	64

TABLE 3

TOTAL FREE ABSORBENCY OF VARIOUS FIBRES IN CARDED FORM.

	100% Standard Rayon	100% Cotton	100% Viloft	100% SI
Total free absorbency $cm^3 g^{-1}$	21·2	27·5	25·3	25·6

FIGURE 5

WICKING RATES

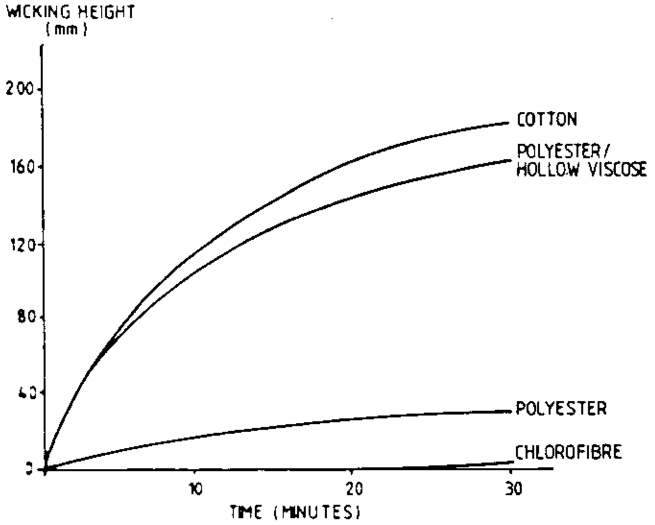
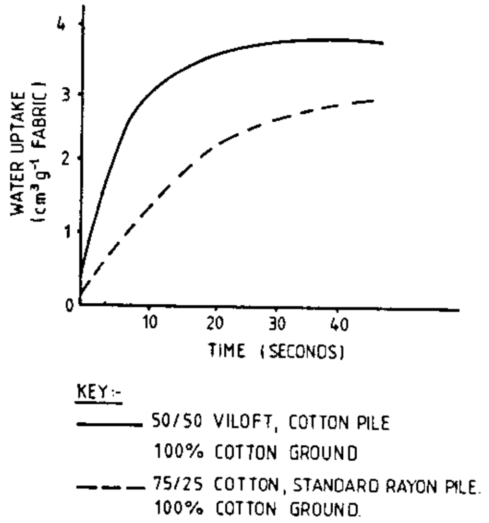


FIGURE 6

RATE OF MOISTURE UPTAKE FOR DIAPERS WITH VILOFT AND COTTON.



Finally, the permanently hollow nature of Courcel confers one other very desirable property - that of insulation. The encapsulation of tiny air pockets within a fabric structure leads to very low thermal conductivity. User trials of a variety of "thermal" fabrics under different conditions have shown that Courcel/polyester rates very highly, particularly in sleepwear - see Table 4.

TABLE 4

WEAR TRIALS ON A VARIETY OF FABRICS FOR DIFFERENT ACTIVITIES.

Garment code	A	B	C	D
ACTIVITY :-				
ROCK CLIMBING	2	4	4	4
FELL WALKING	4	4	4	4
FELL RUNNING	2	2	8	2
CANOE/SAILING	0	4	2	4
MANUAL LABOUR	2	2	4	2
NORMAL WEAR	2	4	2	4
SLEEP	2	4	2	8
TOTAL RATING	14	24	26	28

CODE A IS 85% CHLOROFIBRE
CODE B IS 50% WOOL / 24% ACRYLIC / 12% COTTON
CODE C IS 100% POLYPROPYLENE
CODE D IS POLYESTER/ VILOFT

SCALE :- 0 - POOR
2 - FAIR
4 - GOOD
8 - VERY GOOD

Of all Courtaulds' inflated fibres, the hollow products, Viloft and Courcel, are produced in the largest quantities for use largely in conventional textile applications. The vast majority of this usage is in knitted garments in blend with polyester, normally at a 50:50 blend ratio. A small but increasing amount is also used in blend with cotton. The very desirable comfort and thermal properties of the fabrics lead to their use in underwear (particularly mens and boys), sleepwear, sportswear (including track suits, vests, aerobic dancewear, socks etc.) boot and shoe linings and in flannelette sheets and underblankets. There is also some use in domestic textiles - towels and nappies etc. where soft handle and fast moisture absorption make the products very popular.

The woven textile usage extends mainly into fashion dresses, blouses [illegible] where the good cover and firm handle of the fabrics, coupled with a light weight, make them very suitable for this trade.

PM

The PM fibres were first developed by Courtaulds in the 1960s. Basically they are fibres which have been inflated to such an extent that the wall thickness of the tubular fibres produced is insufficient to support a permanently hollow structure, and the tubes collapse to give thin tape-like fibres. The wall stretching which takes place during inflation virtually removes the normal longitudinal crenulations which can be observed along standard rayon fibres and so a very smooth fibre surface results. This in turn results in a very high level of surface to surface contact between adjacent fibres, and the hydrogen bonding forces involved are sufficient to effectively "glue" the fibres together - they are in fact self-bonding in character. PM1 and PM2, the two versions so far developed, are similar in character but PM2 is slightly less inflated than PM1 and therefore exhibits the typical PM characteristics at a somewhat reduced level. The self-bonding characteristics of PM fibres led to their evaluation in paper-making, with PM1 being the preferred version for speciality papers hitherto made from cotton, linen or manila pulps.

Table 5 shows how the properties of PM fibres are utilised in paper-making to control strength, drainage and porosity.

SI Fibre

SI fibre takes the inflation process to its practical limits, such that the fibre wall of the initially spun tube is only about 2μ thick. The deflation tendency is so great that the wall collapses irregularly to form a multi-limbed cross-section. The resulting fibres have such a high surface area and such an amorphous structure (by virtue of the high acid, low zinc, low stretch conditions) that the water imbibition of the fibre can reach levels as high as 350%.

PM2 fibre, although possessing self-bonding properties, is much softer and generally more conventional in handle than PM1. It is found that PM2 can be made into wet laid nonwoven fabrics with little or no binder. The resulting fabrics tend to break up in flowing water - a property which was exploited in coverstock for "flushable" nappies and sanitary towels in the early 1970s.

TABLE 5

TESTS ON PAPERS MADE FROM PM FIBRES AND STANDARD RAYON

Paper Property:	PM Sample 1	PM Sample 2	Standard Rayon
DRAINAGE TIME / SEC.	8·5	4·8	3·0
FREENESS C.S.F.	684	717	795
BASIS WEIGHT G.S.M.	61·4	60·6	60·0
BULK $cm^3 G^{-1}$	1·3	1·6	IMPOSSIBLE TO TEST. SHEETS TOO FRAGILE.
TEAR FACTOR	346	425	
BURST FACTOR	58	57	
BREAKING LENGTH, METRES	3981	3051	
EXTENSION, %	3·2	3·3	
GURLEY POROSITY, SEC./100 cm^3	20·8	2·1	
OPACITY	66·3% ± 1·3	80·7% ± 0·8	

It is largely this capacity for absorbing water which puts SI in a field of its own and makes it suitable for several very specialised end uses. Table 6 gives the tampon plug absorbencies for a variety of fibres, and also the total free absorbencies. It can be seen that SI performs very well in both tests, but particularly in the tampon absorbency.

In the medical field, purity is of major importance and SI meets all Pharmacopoeia requirements with ease. Table 7 shows the standard purity requirements and the measured values for SI and Alloy fibre.

Finally, in nonwoven end uses, SI can affect both the absorbency of the final fabric and its bulk, cover and handle properties to a greater extent than Courcel. Further, the high surface area of the fibre often means that binder levels can be reduced to give the fabric a desirable softness. Figure 7 shows how fabric drape and bulk alter with increasing percentage of SI in a standard rayon/SI blend.

SI is a very young fibre and was first produced at a commercial level only 6 years ago in 1979. In its current form it is intended to be used mainly in tampon manufactured where its purity, high absorbent capacity and cotton-like stability make it ideal for the modern tampon. This, in addition to the medical and psychological acceptability of 100% cellulosic products, makes SI highly suitable for this market.

CONCLUSIONS

It has been possible, using the inflation process, to produce a range of viscose fibres with increasingly high surface area, and the attendant increases in absorbency. Up to a certain hole size, stable hollow fibres with physical properties not too far removed from conventional staple fibres can be made, and these fibres can be processed without undue difficulty on traditional

TABLE 6

ABSORBENCY DATA FOR SI AND OTHER FIBRES.

	CRIMPED RAYON	VILOFT	ALLOY FIBRE	SI	COTTON
SYNGINA ABSORBENCY. 170mm. Head ~0·35 g cm^{-3} Plug Density Liquid – Water	5·6	6·0	6·9 (Plug density 0·48)	7·3	—
TOTAL FREE ABSORBENCY (cm^3 g^{-1})	21·2	25·3	—	25·6	27·5

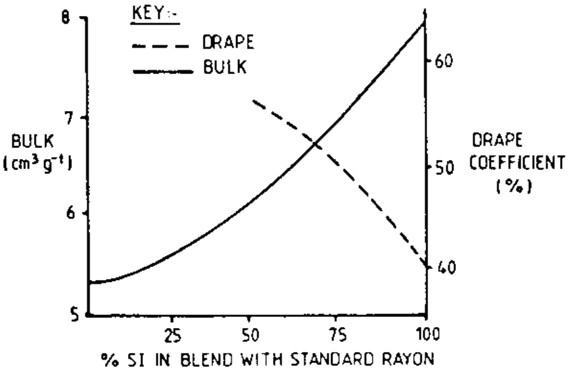
TABLE 7

PHARMACOPOEIA PURITY REQUIREMENTS

Test	Standard Requirement	SI Fibre	Alloy Fibre
MOISTURE CONTENT (%)	13MAX	10·0	9·8
WATER EXTRACT (%)	0·7 MAX	0·3	2·5
SULPHATED ASH (%)	0·45 MAX	0·3	7·7
DIETHYL ETHER EXTRACT (%)	0·3 MAX	0·02	0·04
EXTRACT COLOUR - WATER ALCOHOL	COLOURLESS FAINT YELLOW	COLOURLESS COLOURLESS	COLOURLESS COLOURLESS
ACID / ALKALI TEST	NEUTRAL	NEUTRAL	NEUTRAL
H_2S TEST	NONE	NONE	NONE
SURFACE ACTIVITY (mm)	2 MAX	0	27

FIGURE 7

THE EFFECT OF SUPERINFLATED FIBRE ON FABRIC BULK AND DRAPE.



textile machinery. As inflation proceeds above this level, collapse across a diameter occurs to give smooth surfaced flat fibres which, when cut to short lengths, can be used in paper-making and wet laid nonwoven manufacture. Finally, further increases in inflation cause irregular collapse, and the resultant super-inflated fibres are not easy to categorise. We are nevertheless learning how to use them in conventional textiles, in nonwovens both wet and dry, and in surgical waddings.

Compared with the ordinary viscose fibres, the inflated products are still made on a relatively small scale and command relatively high prices for more specialised applications. Process improvements continue to be made and these, coupled with continued growth in sales, can be expected progressively to improve their competitive position.

ACKNOWLEDGEMENT

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