INHALT/*CONTENT*

Viscose Fibres

Lyocell Fibres

INVESTIGATIONS ON THE STATE OF SOLUTION OF VISCOSE

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Viscose solutions contain different amounts of micro-scopically visible macro gel particles and submicroscopic micro gel particles, depending on the preparation condi-tions. These particles influence the processing properties of viscose, e.g. filterability and viscosity, and may have negative consequences on the fibre properties. The content of macro gel particles correlates with the filter clogging value, and the content of micro gel particles may be determined by rheological investigations. Varia-tion of the viscose preparation conditions will influence not only the total number of particles but also the ratio of the two particle types. Whereas differences in the quality of the pulp and alkali cellulose or an electron beam treatment have a preferential influence on the macro gel particle content, the micro gel particle content is influenced more strongly by the viscose dissolving temperature. The extent of changes in the state of solution of viscose caused by reducing the CS_2 level or by additives depends on the pulp quality and other para-meters of the viscose preparation.

Introduction

It is well known that in viscose and other cellulose solutions a part of the cellulose is not dissolved molecular disperse but exists in the form of aggregates and particles. Besides undis-solved fibre fragments, different particles and aggregates exist in the microscopic and submicroscopic region. About 40 years ago Treiber [I] discussed the existence of a continuous particle spectrum and mentioned the different views in the literature on the state of solution of viscose. We agree principally with a continuous particle spectrum but classify the particles under the limit of detection in different size ranges according table 1. Sometimes these particles are viewed as a pollution in the viscose. Particles and aggregates are, however, an inherent part of viscoses contributing to the properties of the dope and ultimately of the fibres. But the amount and the size distribution of the particles and aggregates is variable and depend as well on the quality of the pulp used for the production of viscose as on the conditions of the viscose preparation.

Particles and aggregates in viscose

Type of particle	Analytical methods
macro gel particles (microscopic region)	clogging value, particle counter
micro gel particles (submicroscopic region)	turbidimetry, rheology
chain aggregates	light scattering

Table 1

This paper deals in the first part with some questions of the ana-lytical determination of particles and aggregates in viscoses, in the second part results will be demonstrated on the influence of some conditions of the viscose preparation on the state of solution and in a third part some examples are given how the properties of viscose are influenced by different types of particies.

Analytical methods for the detection of particles and aggregates

The oldest method for the investigation of the purity of viscose is the determination of a filter clogging value either by measure-ment of the discharge quantity of viscose under constant pressure or by measurement of the increasing pressure by filtration under constant discharge quantity. We are working under constant pressure and use filter paper as the filter medium. Because the viscosity of the viscoses influences the clogging value, we correct the determined k_w value by the viscosity according $k_r = k_w / \eta^{0.4}$. The clogging value gives summary Information about the content of particles above a size of about 5 μm whereas this size borderline of the particle size depends on the filter type. We refer to the cellulosic particles influencing the filterability as macro gel particles. But viscoses may also contain inorganic particles which also contribute to the clogging of the filters. If the viscose preparation is made under normalized conditions, then the clogging value is a quality para-meter of the pulp.

Additional information about number and size of macro gel particles plus other particles of similar size may be obtained by particle counting in viscoses. The various commercial devices for the particle counting are working on different principles, e. g. the difference in electrical conductivity or optical density between particle and solution. Each method has its advantages and disadvantages. If different types of particle counters are used for the investigation of viscoses, sometimes different results or changed relations between the results are obtained as it is demonstrated by tabie 2. Changes of the correlation between the particle number and the clogging value of viscoses may be observed too if various types of viscoses are investiga-ted. These differences may be caused by changed composition or the swelling state of the particles and by changed particle size distribution depending on the used pulp and the prepara-tion conditions of the viscoses. The content of the macro gel particles in unfiltered viscoses amounts about 0.1 % or less of the cellulose.

Comparing particle counting in viscoses by Coulter Counter (electrical conductivity) and PAMAS-Sensor (optical density)

Viscose	Number of particles/g viscose recorded by							
	Coulter Counter				PAMAS-Sensor			
	>5	>10	>15	>25	>5	>10	>15	>25 [μm]
unfiltered	11883	954	235	28	16780	1341	244	42
filtered	3304	251	60	10	5957	196	44	11
start of spinning	3625	579	177	I0	2665	114	31	7
end of spinning	2976	305	40	8	1640	55	12	3

Table 2

While a large number of investigations have been published on macro gel particles in viscoses and in solutions of other cellulose derivatives only a limited number of publications deal with micro gel particles. One reason is that no easy methods were available for the determination of these particles. Electron microscopy, light scattering and turbimetry have been used for some investigations [2, 3, 4].

It is known that the rheological behaviour of polymer solutions may be influenced by their particle content [5]. In the region of small values of the shear stress, the flow curve of polymer solutions is changed by gel particles, especially micro gel particles. This effect was applied by us for the characterization of the micro gel particle content of viscoses. We used a rotation viscosimeter for the rheological measurements and plotted, according to Linsert [6], the logarithm of the viscosities versus the linear values of the shear stress. As it is demonstrated by figure 1, we obtained a linear course of the curves in the region of high and medium values of the shear stress. But at low shear stress the curve is not linear, the viscosity increases strongly. For the numerical evaluation of such rheograms the linear part of the curves was extrapolated to the ordinate. The logarithm of the viscosity of this intersection with the ordinate was subtracted from the logarithm of the measured static viscosity, e. g. the ball falling time. We call this term „rheo value" in accordance with the term filter value. It is approximately a size for the micro gel particle content of viscose. This value depends on the pulp quality and the conditions of the viscose preparation, but there is no general relationship between the clogging value and the rheo value. Specific parameters of the viscose preparation have strong influences, either the filter value or the rheo value can be strongly influenced as it is demonstrated in the next section. The rheo value is normally not influenced by a filtration of the viscose, but we found in some cases an increased rheo value after the filtration of the viscose. We assume it is caused by a destruction of some larger particles to smaller one by shear stress during the filtration.

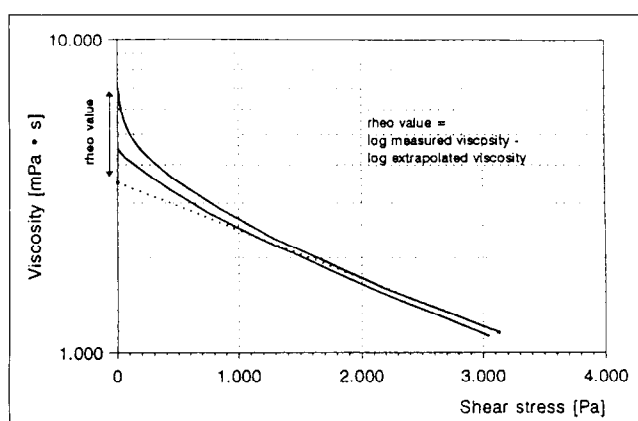### Rheograms of viscoses with different CS₂ input

Fig. 1

Another method to characterize the state of solution by rheological behaviour is to detect the gradient of the loss angle at different shear frequencies via a cone plate rheometer. The resultant gradient of the loss angle will be inbetween „0" for an ideal gel and „-1" for an ideal viscoelastic fluid. As it will be shown in the next chapter, electron-beam pulp treatment can influence this rheological behaviour of viscose solutions.

Light scattering investigations of Burchard et al. in Freiburg [7], Fischer et al. in Tharandt [8] and first trials in our labs have

shown that viscoses contain in addition to gel particles, aggregates of chains, whereas the average number of the chains in the aggregates and the density of the aggregates depend on the conditions of the viscose preparation.

Influence of the conditions of the viscose preparation on the state of solution

Though in the past the processibility of the viscose pulps was improved, there are still essential differences in the quality between different pulps visible in the purity of viscoses prepared under standard conditions by use of these pulps. Comparing commercial pulps with high reactivity and with bad reactivity, the filter values of the viscoses prepared from these pulps may differ in the ratio of about 1 to 10. Not only the amount of macrogel particles, but also the micro gel particles and the chain aggregates may be influenced by the type of the pulp. If different pulps are used for the preparation of viscoses, the differences in the state of solution depend also on the conditions of the viscose preparation. This influence of the conditions of the viscose preparation increases with decreasing reactivity of the pulps.

It is known that the properties of the pulps and the conditions for the preparation of viscose may be changed by various activating treatments of the pulps. One way tested for technical use is electron beam treatment of pulps. Using such pretreated pulp for the viscose preparation the content of macro gel particles is reduced and the filterability of viscose is improved. In this way it is possible to reduce the CS₂ content of viscose without the filterability changes for the worse. The improvement of the filterability of viscose depends on the dose of the electron beam treatment of the pulp as it is demonstrated by figure 2. With increasing radiation levels the filterability initially improves, runs through a maximum and then worsens. We found that the content of micro gel particles in viscose is also reduced by an electron beam treatment of the used pulp. But the relative reduction of the rheo value is lower than the relative reduction of the clogging value as it is shown in figure 3. This is an indication for a changed particle size distribution in the viscose caused by the electron beam pulp treatment.

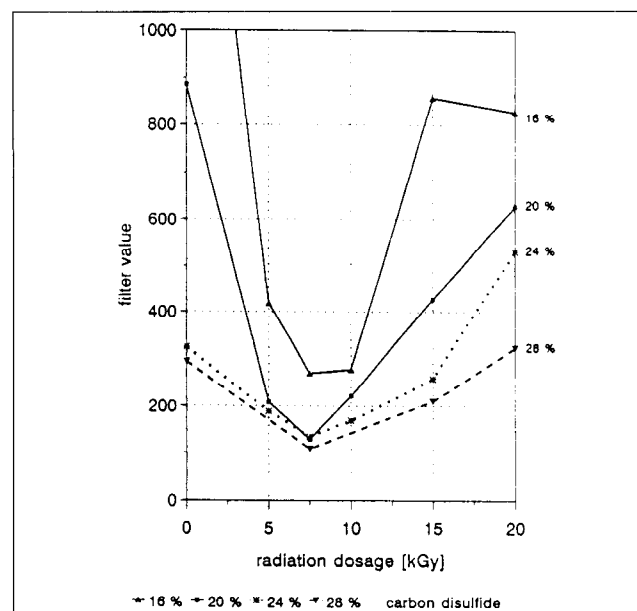### Influence of electron beam treatment of pulp on filter values of viscoses at different CS₂ levels

Fig. 2

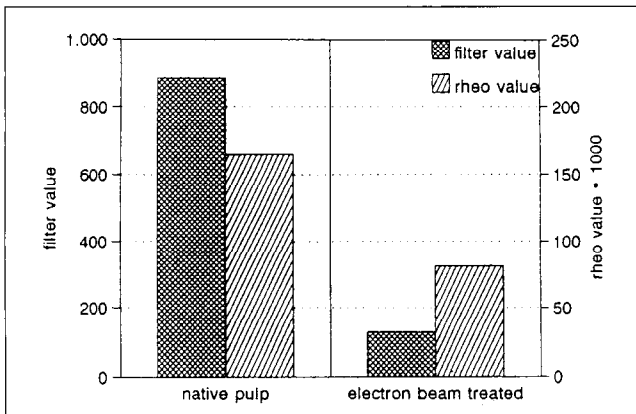### Changes of filter value and rheo value of viscose by an electron beam treatment of pulp

Fig. 3

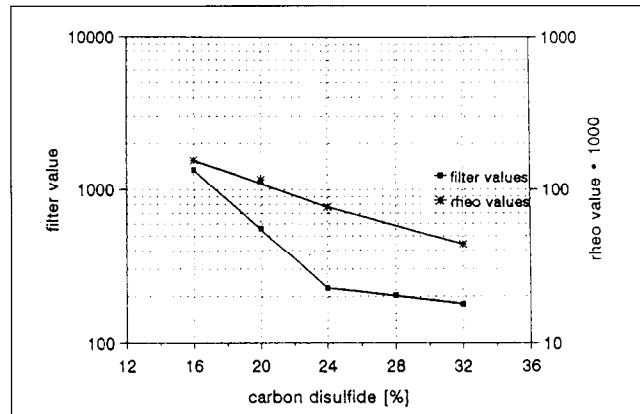### Filter values and rheo values versus CS₂ input for the same series of viscoses

Fig. 5

Looking on the process parameters of the viscose preparation, the amount of CS$_2$ is of special interest because there is a direct relationship to environmental problems. The state of solution of viscose may be changed by a reduction of the amount of CS$_2$ used for the production of viscose. If a good pulp is used and viscoses with 35 and 30% CS$_2$ are compared, no essential differences were found, either in the gel particie content or in the chain aggregates. Reducing the CS$_2$ content of the viscoses still more, the filter values increases at first for some types of viscoses only slightly but for other series of viscoses the increase is stronger till an inflection point is reached. For CS$_2$ contents below this inflection point the curves rises essential stronger, as it is demonstrated by figure 4. The CS$_2$ level at the inflection point and the course of the curves depend on the quality of the pulp and the conditions of the viscose preparation. Using pulp with high reactivity and advantageous conditions for the viscose preparation, the inflection point may be situated even at a CS$_2$ level below 20%. The different change of the content of micro gel particles caused by decreasing CS$_2$ of the viscoses compared with the macro gel particles is shown in figure 5. The rheo value increases with decreasing CS$_2$ content also for such viscoses where the filter value is changed only slightly. But below the inflection point the rheo values rise to a smaller extent than the filter values.

Below a definite CS$_2$ level, the chain aggregates are also changed by a decreasing CS$_2$ content in the viscoses. The average number of chains in the aggregates and their density increases with decreasing CS$_2$ content.

It is known that besides the CS$_2$ level other parameters of the viscose preparation, e. g. the sodium hydroxide content and the dissolving temperature, have an influence on the quality of the viscose. We found that both parameters, besides their effect on the filterability, have a strong influence on the micro gel particle content. Figure 6 shows that as a result of a decreased dissolution temperature of viscose, the relative reduction is somewhat larger for the rheo value than for the filter value. The relative decrease of the particle content caused by diminished dissolving temperature increases with decreasing CS$_2$ content of the viscoses. So we were able to prepare viscoses with acceptable filterability at rather low CS$_2$ levels using high quality commercial pulps without electron beam treatment but low dissolving temperature.

Influence of dissolution temperature of viscose on filter value and rheo value
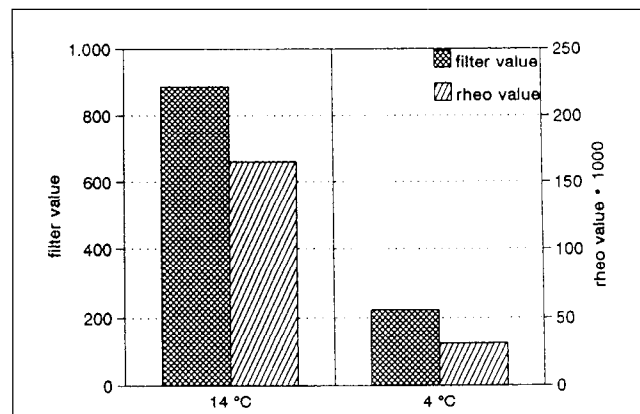
Fig. 6

Filter values versus CS₂ for different types of viscoses
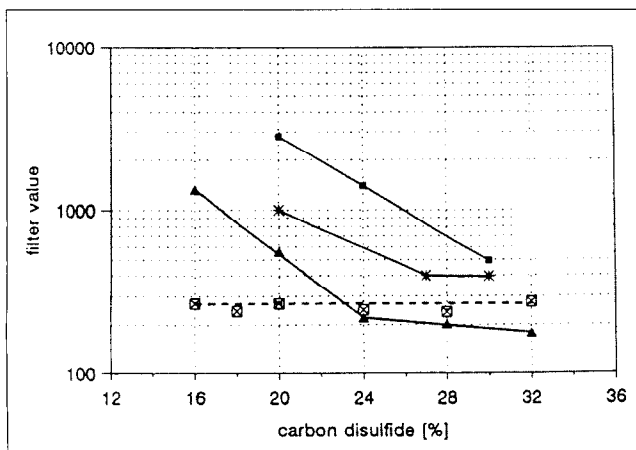
Fig. 4

If we compare the rheological behaviour of viscose solutions made from electron beam treated and nontreated pulps at low dissolving temperature, we see in particular at low CS$_2$ levels a significant shift of the gradient of the loss angle of the electron beam treated sample to „-1". This means that the state of solution of such viscoses is similar to an ideal viscoelastic fluid as compared with viscose solutions from the nontreated pulp (figure 7). Comparing furthermore the post-ripening behaviour of viscose solutions made from nontreated pulp versus viscose solutions from electron beam treated pulps of the same type, we see, as it is shown in figure 8, a real difference in the ripening behaviour. Our previous results of the postripening behaviour of viscoses prepared with different CS$_2$ levels but with the same alkali

Gradient of the loss angle "tan delta" at different shear frequencies
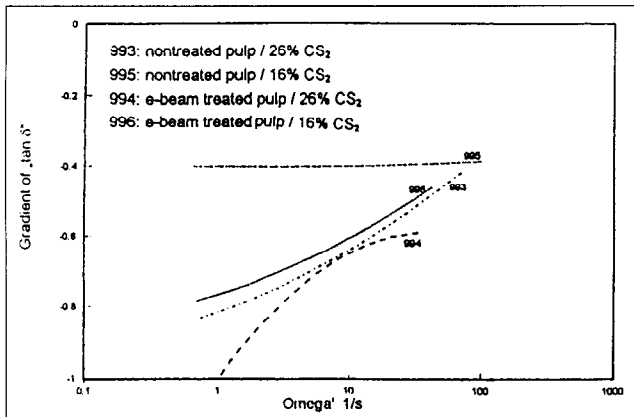
Fig. 7

Postripening behaviour of viscoses from e-beamed and nontreated pulps with different CS_2 levels

Fig. 8

addition of an additive is often necessary. For sulphite pulps the necessity depends on the type of pulp and the conditions of the viscose preparation. But an additive may equalize also deviations in the viscose preparation and especially if the reduction of CS_2 content of the viscose is desired, an additive may be useful.

Influence of addition of Visco 388 on clogging values

| Addition of the additive	Clogging value k_r
without	935
0.1 % to pulp	741
0.2 % to pulp	574
0.2 % (refering to cellulose) to the dissolving lye	758

Table 3

Impact of viscose particles on the production of filament yarn

It is known that besides the dependence of the filterability of viscoses on their particle content the particles may influence process stability, spinnability and the fibre properties too. The limit of the size and the amount of particles influencing the fibre properties depend on the spinning conditions and the produced fibre type. Not only the properties of high tenacity fibres may be influenced by the gel particle content of viscoses but also filament yarn for textile purposes. Table 4 shows the influence of a different particle content changed by different conditions of preparation and filtration of viscose on the yarn properties. We found for these trials the best correlation if the fiber properties are correlated with a numerical value formed by addition of filter value and rheo value of the filtered viscose.

Influence of particle content of spinning viscose on tenacity of filament yarn

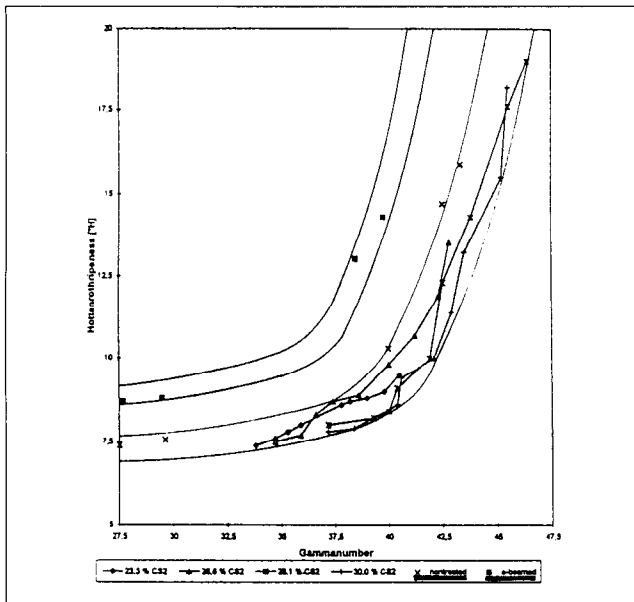| Particle content [k_r value + rheo value - 1000]	Tenacity of the filament yarn [cN/tex]
89	18.2
130	17.7
211	17.0

Table 4

content and with low dissolving temperature show a small ribbon like zone in the "gamma number versus Hottenroth number" plot. The actual results of the nontreated pulp sample lie in this zone but the results of the electron beam treated pulp show a zone with a similar course left by the previous zone. That means that the readiness of coagulation of the electron beamed pulp viscoses is significantly lower than from the nontreated pulp at the same degree of xanthation. As it will be shown later, this difference has no impact on filament yarn data.

It is known that by an addition of additives the quality of viscoses may be influenced. Normally the additives are added to the pulp. In the older literature it was emphasised in agreement with our previous results that an improvement of the viscose quality is only obtained if the additive is added before the xanthogenation. But with the modern additives it is necessary to modify this statement. Using the additive Visco 388 we found the largest effect, if this additive is added to the pulp or to the alkali cellulose. But the filterability of viscose was also improved, however to a lower extent, if Visco 388 was added during the dissolution of viscose (table 3). The relative improvement of the clogging values caused by additives is different, if different pulps are used for the viscose preparation. For prehydrolysed kraft pulps the

Spinning of the above mentioned viscose solutions of electron beamed and nontreated celluloses under identical lab conditions results, particularly for those viscoses with a low CS_2 content, in no improvement of the spinning behaviour for the electron beamed cellulose, as it should have been expected according to the changed coagulation behaviour, neither significant improvements of the textile-physical properties of the filament yarns, spun from electron beamed cellulose, could be found. The level of the textile data obtained by spinning of viscoses with a CS_2 content of 16% is 40% lower than with a CS_2 content of 26%, and thus unacceptable (table 5). Use of viscose from electron beamed pulp did not improve this situation.

The micro gel particles contribute to the viscosity of viscose or other cellulose solutions. Already Gray, More and Yorke [4] had mentioned a loose correlation between the measured turbidity and the viscosities of viscoses. We found a linear correlation between the rheo values and the viscosities of viscoses prepared with different amounts of CS_2, as it is demonstrated by figure 9. The intersection of the regression line with the ordinate would correspond to the viscosity of a viscose without micro gel particles.

Influence of particle content of spinning viscose on tenacity of filament yarn

Pulp treatment	CS$_2$ input [%]	Tenacity [cN/tex]	Elongation [%]	Working capacity [mJ/tex · m]
e-beamed	26	21.5	21.4	32.2
e-beamed	26	23.0	22.6	36.2
e-beamed	16	18.9	16.1	23.4
e-beamed	16	20.6	13.7	20.7
nontreated	26	20.6	20.9	30.1
nontreated	26	20.9	21.0	30.8
nontreated	16	17.6	16.3	22.1
nontreated	16	18.6	14.1	19.7

Table 5

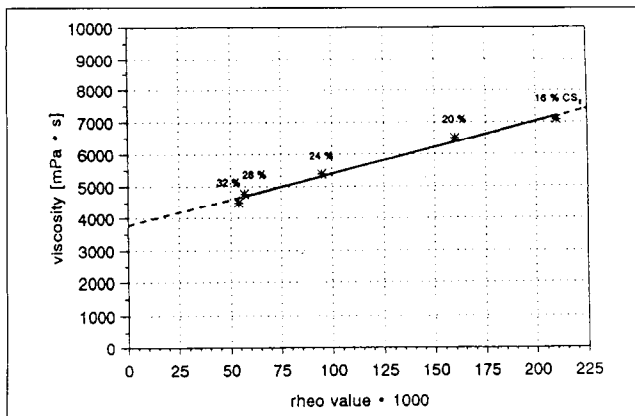## Rheo value versus viscosity for a series of viscoses

Fig. 9

Nothing is known today about the influence of the chain aggregates on the macroscopic properties of viscoses. But we may imagine that differences in content, size and or density of the aggregates may have an influence one the fibre formation process and ultimately on the fiber properties.

Conclusions

The properties of viscose fibers are influenced by both the spinning conditions as by the state of solution of the viscose. The effect of the state of solution increases with decreasing amount of CS$_2$ used for the viscose preparation. We know a lot about, the macro gel particles. But in many cases the filtration is not the limiting factor for a reduction of the CS$_2$ content of viscoses. We know that viscoses contain besides the particles influencing the filterability particles and aggregates in the submicroscopic region. We assume that these particles and aggregates may have an influence on the spinning process. For that reason it seems necessary to improve our knowledge about the effect of these particles and aggregates on the spinning process and the properties of the fibers.

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TURNING POINTS IN THE INNOVATION OF VISCOSE FIBER PRODUCTION

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ABSTRACT

This paper reflects an intensive world-wide technological survey amongst viscose fiber producers carried out in USA, Europe, Japan and other Asian countries. The study focuses on technological trends and potentials of the employed unit processes in the light of increasing competition from synthetic and natural fibers as well as emerging alternative cellulosic processes.

In the conventional rayon production recent years have been characterized by the introduction and implementation of environmental programmes including the closing of material and energy flows. Significant changes and modifications in the viscose process or machinery itself were only moderate and have been concentrating on cost saving investments and on product development to meet the market demands.
Hence it is of importance to evaluate in which sections of the production process the technological status develops a new generation of treatment systems and in which sections the development status seems to reach a certain plateau.

The survey was accomplished considering all major unit processes and represents the current state of the art as well as a wide spectrum of potential improvements on quality, capacity, flexibility, efficiency and a series of other indicators including the cross link of several process steps.

As a conclusion there are significant indications among technological leaders that an additional innovation cycle has been started to face future challenges and threats. Main directions point towards an increased flexibility of the equipment in order to broaden the product mix and towards a further production cost reduction on raw material and labor base.

Introduction

The world viscose staple fibre production has been shown to decline over the last several years worldwide. If one looks at the data in more detail, production increase can be seen if the former Sowjet Union is excluded from the analysis. For 1997 a production capacity of 2 Million metric tons is expected. However, the actual production will be only 1.62 million metric tons which means a utilization of 79 % [1].

The overall market share of cellulosic fibres could not compete with synthetic man-made fibres or cotton, therefore the process development for the production of these fibres was always aimed at the improvement of the excellent specific properties of these fibers to maintain the market relevance in the textile industry.

Prof. Albrecht recently wrote a good summary of the viscose fibre industry where he could pinpoint to the advantage by using a natural resource such as cellulose for fibre production [2].

The industrial behaviour of the viscose fibre producers clearly reflected the niche market status. This means that the number of viscose fibre producers, specifically in Western Europe, declined dramatically [3]. The amount of the total R & D budget did also decline in most companies or was shifted towards alternative cellulosic fibre processes.

This paper reflects a worldwide technological survey to improve the current picture about the state of the art in this technology and should evaluate the trends in this field.

- Main focus & frame for the study

- Overview of the viscose staple fiber process

- Analysis of trends in the process

- Key equipment characteristics

- Lenzing Technik approach

- Lenzing Technik services

- Summary and conclusion

Fig. 1

Main focus and frame of the study

THE STUDY

The study reflects a world-wide technological survey amongst viscose fiber producers.

The targets of the study are concentrated around 3 topics. These targets were the driving force for the discussion with experts.

TOPIC 1 - Evaluation of major trends & needs in the viscose process

TOPIC 2 - Determination of process parameters and consequences for the process

TOPIC 3 - Evaluation of characteristics of the used equipment in the process units

Fig. 2

In 1996 Lenzing Technik carried out a study and formed three target areas for analysis:

TOPIC 1 - Evaluation of major trends & needs in the viscose process

TOPIC 2 - Determination of process parameters and consequences for the process

TOPIC 3 - Evaluation of characteristics of the used equipment in the process unit

Participating companies

Europe	9 companies (staple, filament, casing)
America	2
Asia	4
TOTAL	15 companies

Fig. 3

15 companies contributed with expert discussions and answered the various questionnaires, which are the basis for the thesis gained in the study.

Restrictions

Among the participating experts there was a slight majority in the field of viscose staple fiber producers. Therefore some conclusions are specifically oriented towards staple fiber production.
The methodology used creates a thesis and not a measureable fact.

The study was carried out in cooperation with the Graz University of Technology/Austria in 1996.

Fig. 4

Among the participating experts there was a slight majority in the field of viscose staple fibre producers. Therefore some conclusions are specifically orientated towards staple fibre production. One certainly has to be aware that the opinions of the experts create a thesis and not measurable facts as in natural sciences. The study was carried out in cooperation with the Graz University of Technology/Austria, special acknowiedgement should be made to Christian Haas, who dedicated his master's thesis to this topic. Christian Haas's work specifically concentrated on equipment analysis to develop future innovation strategies.

Overview of the viscose process

A flowchart of the viscose process is given in figure 5. The given flowchart is actually a picture drawn in the 50's and was chosen on purpose of illustrating major trends in the last decades.

Fig. 5

Analysis of trends in the process

TOPIC 1 - Major trends and needs

Thesis 1: „Nothing changed"

Thesis 1: " Nothing changed"

The flow sheet drawn in the 50's is still valid

EXCEPTION: Treatment of pulp by electron beaming is likely to replace ripening and cooling

The first commercial plant for pulp treatment is currently being built in Germany and starts production end of '97.
Technology: IBA, Belgium*

NOTE: Recovery systems (spinbath, CS2, ...) are not even in the picture

* Lenzing Technik formed a cooperation with IBA for further development

Fig. 6

The given flow diagramme of the 50's is still valid for all major process units. Nevertheless, there is one significant change about to break through. The electron treatment of pulp is likely to replace ripening and cooling.
In total we see four changes with respect to the application of irradiated pulp:

The first commercial plant for pulp treatment is currently being built in Germany and starts production end of 1997. The employed technology is Rhotodron electron accelerator IBA/ Belgium. Lenzing Technik formed a cooperation with IBA for further development in the field of pulp application.

1. Lower CS_2 and NAOH consumption
2. Reduction of environmental impact due to chemical savings
3. Replacement of ripening and cooling
4. Activation of cellulose with the potential to improve fibre properties

Especially the activation of cellulose was proven to be beneficial for the process already in the 50's. Nevertheless it took some time to develop a commercially attraktive technology for the electron treatment.

Looking again at the flowchart of the process one notes that recovery systems (spinbath, Zn, CS_2, ...) are not even in the picture.

Thesis 2:   The production capacity of one spinning line increased significantly

Thesis 2 – TREND: The production capacity of one spinning line increased significantly

Recent installations follow clearly the "economies of scale" principle

TREND: towards 200 to/d per line

Fig. 7

This trend can be clearly seen if one looks at recent installations of viscose staple fibre units.

Coming from 20.000 t/y a few years ago we see now 35.000 t/y and expect an even sharper increase to 200 t/d per line. This trend certainly goes in line with the 'economies of scale' principle. The producers have to compete with a p(ice level similar to phces in the 80's. This can only be achieved if all cost factors including an increased line capacity are optimized.

Thesis 3: Environmental issues brought most innovations

Thesis 3 - NEED: Environmental issues brought most innovations

These innovations are outside the "cellulosic" line

• Spinbath and chemical recovery (e.g. back wash filtration of spinbath, reject filtration,...
. ..., > CLOSING of cycles)

• CS2/H2S recovery (e.g. catalytic conversion, adsorption systems,...)

• "End of pipe technology" is still required to meet legal standards
(without adding value to the product)

Fig. 8

Most process changes which we observe are outside the "cellulosic" line and did not add value to the product. A few examples are given.

−   Spinbath and chemical recovery (e.g. back wash filtration of spinbath, reject filtration,... > closing of cycles)

- CS$_2$/H$_2$S recovery (e. g. catalytic conversion, adsorption systems, − "End of pipe technology" is still required to meet legal standards.

TOPIC 2 - Process parameters

Thesis 1:   There is no reliable "product" measurement along the process

There is no reliable "product" measurement along the process

Measurements concentrate on p, T, conc., Q values. These values are used for automation and integration in DCS/PLC systems
Semi-continuous process steps and inaccurate indices could not eliminate product quality variations.

▶   Innovations concentrate on on-line "product" measurement

Thesis 2: Process control systems contribute significantly to labor savings and product stability and enabled the production of finer fibers.

Fig. 9

Measurements concentrate on pressure, temperature, concentration and flow values.

These values are of course of extreme importance and are used for automation and integration in DCS/PLC systems. However, the quality, especially of viscose, is not fully disclosed during the process and can only be verified with the relevant fibre properties at the end of the spinning line.

Due to semicontinuous process steps and partially inaccurate process indices a stability of the final product qualities cannot be reached fully. Innovation clearly shifted towards online product measurement.

Examples of recent innovations of Lenzing Technik are given below:

Recent innovations of Lenzing Technik for process control

• On-line particle count system for viscose

• On-line titrating apparatus (H2SO4, ZnSO4, NaSO4)

• Spinneret inspection system

• CS2 - analysis for air measurement

• On-line bleaching bath analysis

• Bale inspection system

NEW GENERATION OF FIBER TESTING EQUIPMENT FOR STAPLE AND FILAMENT
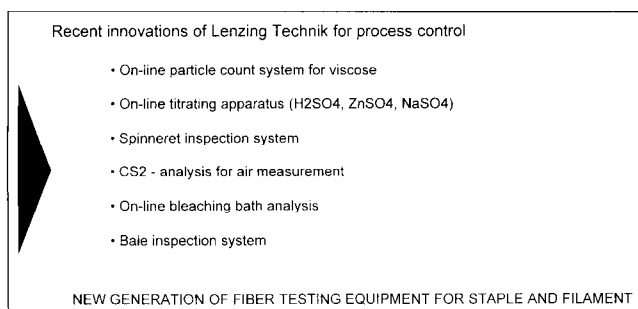
Fig. 10

−   On-line particie count system for viscose
−   On-line titrating apparatus (H$_2$SO$_4$, ZnSO$_4$, NaSO$_4$)
−   Spinneret inspection system
−   CS$_2$-analysis for air measurement
−   On-line bleaching bath analysis
−   Bale inspection system
−   New Generation of fiber testing equipment for staple and filament

Thesis 2:   Process control systems contribute significantly to labour savings and product stability

Despite the lack of 'product' measurement a tremendous increase in product automation took place in the last years. This enabled producers to reduce required personnel significantly and increased the flexibility for the production of various types of fibers.

Additionally PLC systems facilitated the production of fine fibers. Fine titers show an excellent response from market side and represent a clear trend in textile applications.

Key equipment characteristics

TOPIC 3 - Evaluation of equipment characteristics

Five process equipments are chosen as examples for analysis.
– AC-press
– Xanthogenation & dissolving
– Filtration
– Fiber spinning
– Cutting

The producers' criteria of the specific units were given in the questionnaires by the participating companies. The most important criteria are indicated in the figures. The current state of the art as well as trends for further development are also indicated in the figures 13 to 17.
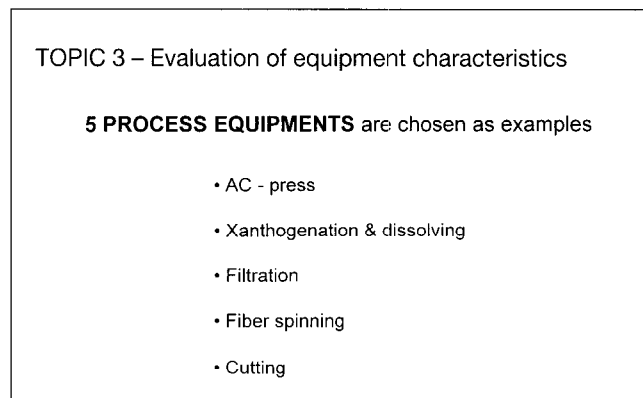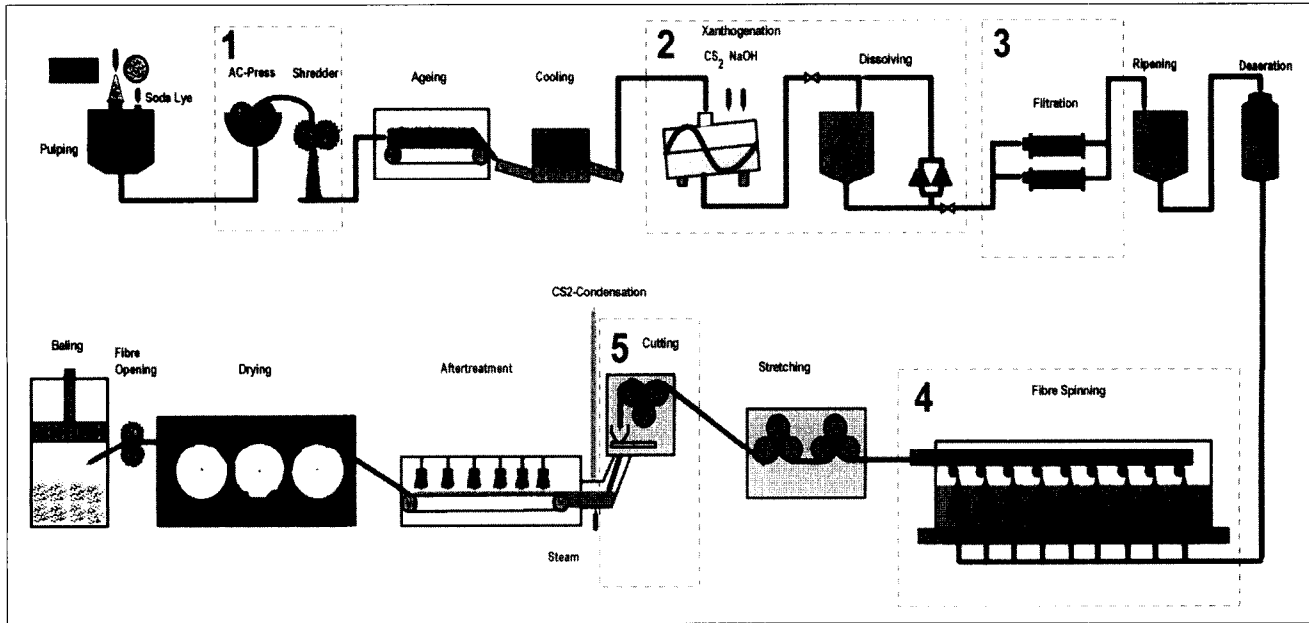
TOPIC 3 – Evaluation of equipment characteristics

5 PROCESS EQUIPMENTS are chosen as examples

• AC - press

• Xanthogenation & dissolving

• Filtration

• Fiber spinning

• Cutting

Fig. 11

Pulping Soda Lye **1** AC-Press Shredder Ageing Cooling **2** Xanthogenation CS₂ NaOH Dissolving **3** Ripening Deaeration Filtration

Baling Fibre Opening Drying Aftertreatment CS2-Condensation **5** Cutting Stretching **4** Fibre Spinning Steam

Fig. 12

AC-pressing

Producer criteria
(ranked by importance)

• Process design

• Degree of pressing

• Down time
(for maintenance)

• Spare parts

• Safety

Example: LT AC press

State of the art & trends
(indicated by producers)

Single steeping still prevails

Automatic back wash cleaning system

Improved material

Life time of press rollers
> 5 years

▼

Homogenous & constant
AC

Fig. 13

Xanthogenation & dissolving

Producer criteria
(ranked by importance)

• Safety

• Environmental performance

• Control systems

• Reliability

• Product quality

• CS2 consumption

• Cleaning

Example: LT Wet chum

State of the art & trends
(indicated by producers)

▼

Large units (for > 55 to/d fiber)

Excellent self cleaning

Reduction of
energy consumption

Uniform product

New manufacturing technology

Fig. 14

Filtration

Producer criteria (ranked by importance)		State of the art & trends (indicated by producers)
• Environmental performance • Operating costs • Maintenance • Filtration quality	 Example: LT KK-Filter	▼ Fully automatic inline back wash systems 100% Reject recovery systems

Fig. 15

Fiber spinning

Producer criteria (ranked by importance)		State of the art & trends (indicated by producers)
• Fiber quality • Reliability • Environmental performance • Safety • Machine design	 IMPORTANT: Waste gas collection, accuracy of drive system, material	Trend towards **200 to/d** line Short machine for increased flexibility Increased tenacity by machine design **up to 3,3 g/den** **Spinnerett**: ▶ > 100,000 holes

Fig. 16

Cutting

Producer criteria (ranked by importance)		State of the art & trends (indicated by producers)
• Cost efficiency • Variable cutting length • Reliability • Safety • Capacity	 Example: LT Cutter	more than 10 Mio dtex excellent cutting profile increased life time of cutting knives flexible cutting length possibility of short cut (trend towards NW) drive system improved material

Fig. 17

Lenzing Technik Approach

Lenzing Technik established an organisational structure which is orientated to specialised product groups in order to simulate further product development. The strategy clearly aims at pulp and viscose technology and testing equipment for fibers as well as process control. Electrical and mechanical engineering are subsequent disciplines which keep up to date with the novelties in this field.

Lenzing Technik Services

```
• Evaluation studies of process
• Basic and detail engineering for projects
• Assistance in improvements of equipment
• Fabrication / delivery of key equipment
• Standby capacity for consulting
• Personnel assistance in project execution
• Testing equipment for fibers and process parameters
• Process automation
```

Fig. 18

The service offered covers the entire range of project management from the beginning of the idea in the lab up to project realisation in the plant. Some examples are given below.

- Evaluation studies of process
- Basic and detail engineering for projects
- Assistance in improvements of equipment
- Fabrication/delivery of key equipment
- Standby capacity for consulting
- Personnel assistance in project execution
- Testing equipment for fibers and process parameters
- Process automation

Summary and Conclusion

```
Key statements

1. Spinning line capacities will further increase

2. Pulp radiation is on the edge to commercialization

3. Environmental issues keep to be a cost factor

4. Product measurement will simplify complicated process control

5. The innovation of equipment & process units is closely linked to the development in other fields
   (material science, DCS - systems, irradiation systems, ...).
   This circumstance is often misinterpreted by viscose research departments
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Fig. 19

The following key statements summarize the findings of the study:

1. Spinning line capacities will further increase
2. Pulp radiation is on the edge to commercialization
3. Environmental issues keep to be a cost factor
4. Product measurement will simplify complicated process control.
5. The innovation of equipment & process units is closely linked to the development in other fields (material science, DCS-systems, irradiation systems, ...).
   This circumstance is often misinterpreted by viscose research departments.

As a conclusion there are significant indications among technological leaders that an additional innovation cycle has been started to face future challenges and threats. Main directions point towards an increased flexibility and production capacity of the equipment and a further production cost reduction on raw material and labour base.

Literature

[1] Chemical Fibers International, Vo. 46, Dec. 96, p. 394
[2] Chemical Fibers International, Vo. 46, Dec. 96, p. 384
[3] Chemical Fibers International, Vo. 45, Feb. 95, p. 41

COMPARATIVE EVALUATION OF DIFFERENT CONCEPTS OF SULFITE PULPING TECHNOLOGY

Herbert Sixta, Lenzing AG, Austria

The superseding of the sulfite cooking process commenced in earnest in the 1950's with the development and technical establishment of the recovery of chemicals using the kraft pulping process. Apart from the very high recovery rate of the pulping chemicals, the fact that rich resin and raw materials of a poorer quality could be used, as well as vastly improved tenacity properties were some of the excellent values of the kraft pulping system compared to the sulfite process. The high degree of brightness of the unbleached pulp and the good bleachability, the most important advantages of the sulfite process, have not yet been able to make up for its disadvantages when compared to the kraft pulping process. In the same way, multi-stage sulfite processes, developed in the 50's and 60's, were unable to turn this trend around. Most developments aimed at extending the range of raw materials which did succeed to a certain extent by increasing the pH value in an initial phase - bisulfite or

neutral sulfite. In addition, it was possible to achieve better tenacity values in the case of a two-stage bisulfite-neutral sulfite process as well as a higher wood yield in the combination, neutral sulfite-acid sulfite process.

It wasn't until the development of the ASAM process, in the mid 1980's, that we really had a pulping process at our disposal which unites the advantages of the sulfite and the kraft pulping process without simultaneously demonstrating other disadvantages. In the present study the ASAM process was compared with the two conventional pulping processes, the acid magnesium bisulfite and kraft pulping process, using beechwood and producing TCF(totally chlorine free)-bleached paper pulps. The results demonstrate the clear superiority of the ASAM-process compared to the two competitive pulps with regard to the wood yield, tenacity properties and the stability of the degree of brightness.

Introduction:

In the early days of chemical pulp production, the sulfite process was the dominating technology mainly due to the high initial, brightness and the easy bleachability of unbleached chemical pulp. With the invention of selective bleaching concepts of kraft pulps in 1930, the kraft pulping technology began to gain ground in certain fields because of better paper strength properties. Since then kraft technology has steadily superseded the sulfite process.

Three main factors have made the sulfite industry less competitive, namely its sensitivity to the wood raw material, the lower strength properties of paper products and the difficulty of recovering the cooking chemicals and utilising waste products. In the meantime, efficient chemical recovery systems have been developed especially to use magnesium and sodium as a base. In the case of acid sulfite pulping, the high sensitivity to the wood raw material still constitutes a problem. A large part of the softwoods, the pines, larches and Douglas fir are considered less suitable for sulfite pulping. A certain part of the extractives of phenolic character such as pinosylvin, taxifolin (Douglas fir) as well as the tannins of bark-damaged spruce and oaks give rise to condensation reactions with lignin moities in the presence of acid sulfite cooking solutions. Furthermore taxifolin, having pyrocatechol groups, tends to reduce bisulfite to thiosulfite, thereby decreasing the stability of the cooking acid. Condensation reactions of the lignin with the phenolic extractives can be prevented if conditions of the sulfite cooking are chosen to favour sulfonation of the reactive parts of lignin. It has been established that the combination of low acidity and not too high temperatures seems to be essential to direct lignin reactions towards sulfonation. Therefore, new concepts of sulfite cooking technology with an initial, less acidic, neutral or even alkaline stage have been developed to overcome the problems with the use of high resinuous wood species.

A wide variety of processes has been proposed since the first patent was awarded to Graham[1] in 1882 who suggested a two-stage cooking process, using sodium sulfite as a first and a conventional calcium acid sulfite as a second step. A break-

through in pulping resinuous wood species was given by the development of Hagglund and co-workers[2], with the first stage more alkaline than the second. This concept was first realized in a full-scale operation known as the Stora and the Kramfors is processes for pulping pine and tannin-dainaged spruce, respectively[3,4]. To change the pH from the acid to the slightly alkaline range, the so called soluble bases, sodium, ammonia and magnesium, are required. Sodium and ammonia both make it possible to change the acidity of the liquor within the entire ph-range. Nilsson and Stockman demonstrated the possibility of using magnesium up to pH levels of 9 in a first alkaline sulfite stage[5]. It has been shown that the solubility of $MgSO_3$ suspensions in the pH range above 5 increased significantly in the presence of (recycled) spent liquor or higher salt concentrations (NaCl or Na_2SO_4). At Weyerhaeuser's Longview Research Laboratory, a technical concept of a two-stage magnesium sulfite process with first stage pH levels as high as 6.0 has been developed[6].

Sulfite pulping under alkaline conditions using either two-stage (bisulfite / neutral sulfite, neutral sulfite-AQ / soda-AQ etc.) or one-stage concepts (alkalisulfite-AQ, ASAM etc.) leads to the production of pulps with significantly improved strength properties, comparable to kraft pulps or even better than those in the case of ASAM pulps. The alkaline pH range is only accessible to sodium and ammonia. Sodium is, however, to be given preference since the ammonia liquors are less stable at higher temperatures.

Chemistry of Sulfite Pulping

The removal of lignin in the course of acid sulfite pulping is described as a two-step process[7] (Hägglund, 1951). First, sulfonation of the lignin in the solid phase occurs to produce a solid lignosulfonic acid. Secondly, hydrolysis of the solid lignosulfonic acid takes place and gives a water soluble product. Rydholm and Lagergren discovered that the lignin-wood bondings can be cleaved not only by hydrolysis but also by sulfitolysis[8]. The two phases of lignin removal always proceed concurrently during acid sulfite cooking. rate of delignification was found to be proportional to $[H^+]^*[HSO_3]$ which can be

explained by the assumption of a rate-determining solid phase-sulfonation preceding hydrolysis as well as sulfitolysis.

It is agreed that sulfonation primarily takes place at the α-carbon atom. The sulfonation reactivity of a benzyl alcohol or ether group is increased by both higher acidity and the substitution of the aromatic ring, preferably in p-position, exerting a +M-effect, like hydroxyl or phenoxy groups. The cleavage of (α-ether bonds leads to the intermediate formation of a benzylic cation being in an equilibrium with chinonmethide-like structures. The carbocation is finally sulfonated by the nucleophiles, hydrogensulfite anion / hydrated sulfit dioxide[9]. Sulfonation can also take place at the γ-carbon atom in the case of stilben structures, with highly electrophilic γ-carbon atoms[10]. It is well known that sulfonation competes with condensation reactions. The rate of the latter is proportional to the acidity in the solid-liquid interface. Therefore, the extent of condensation reactions increases with the continuance of cooking, where the ratio between bisulfite and proton concentration steadily decreases. Therefore, a high initial bisulfite anion concentration (=combined SO_2) is essential to avoid extensive condensation reactions, especially towards the end of cooking.

Alkaline sulfite pulping requires quite different reaction conditions compared to acid sulfite pulping. The mechanism of delignification shows close similarities to kraft pulping at pH levels above 12. The intermediate formation of chinonmethide, preceded by the cleavage of α-ether arylether bonds, is the rate-determining step in case of phenolic lignin structures. Subsequently, sulfonation occurs at the electrophilic α-carbon atom, followed by a sulfitolytic cleavage of ß-arylether bonds. The elimination of both the α-sulfonate group and the hydroxymethyl group at the ß-carbon atom is proposed as final fragmentation reactions.

Concepts of Sulfite Pulping

The following concepts of multi-stage sulfite pulping were developed in the fifties and sixties as an answer to the rapid development of the kraft process:

Bisulfite-Neutralsulfite Process & Bisulfite (Acid Sulfite) - Carbonate (= Sivola)

The bisulfite-neutralsulfite process is characterized by a change of the pH level from around 3.5- 4.0 in the first up to 7.0-8.5 (and higher) in the second stage. Using magnesium as a base, the pH level in the second stage was limited to around 6.5-7.0. Differences in pulp quality resulted depending on whether or not the first-stage liquor was drained before alkali was added to the second stage. Pulps with superior burst and tear levels were obtained when the alkali was injected directly into the first-stage liquor. When the first-stage liquor was drained before alkali was added, tearing resistances comparable to kraft were found, but the bursting strength was not improved. The pulp quality and the process behaviour has been shown to be significantly influenced by the type of cation used. The different dependency of pH on temperature was discussed to be one reason for the observed pulp quality differences. In the case of sodium carbonate, a stronger dissociation occurs at elevated temperature and the pH is not affected by increased water ionisation until temperatures above 140° C are encountered. The pH of the sodium sulfite solution is also not dependent on temperature[11]. The ammonia water and the sodium hydroxide-water system, however, show a clear dependency of pH on temperature. The pH of the alkali partially explains the pulp quality differences, although it is obvious that this is not the only factor

involved. For example, even though ammonium hydroxide and sodium hydroxide solutions of similar (hot) pH were used, they did not result in pulps of similar quality. It is possible that pulp swelling and the pH difference between the inside of the chip and the pulping medium are of importance and are influenced by the cation type. Sanyer et al. have investigated multi-stage sulfite pulping of both softwoods and hardwoods[12]. Selected results using spruce wood as a raw material and sodium as a base are shown in figures 1-4.

Yield vs. Permanganate number:
Comparison of four sulfite processes using spruce wood[12].
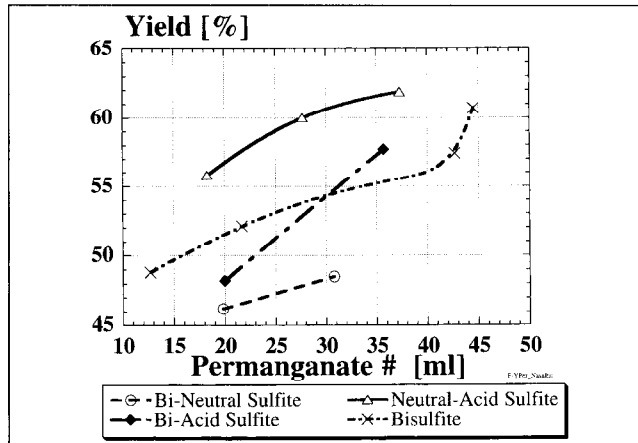
Fig. 1

Tear factor vs. Permanganate number:
Comparison of four sulfite processes using spruce wood[12].
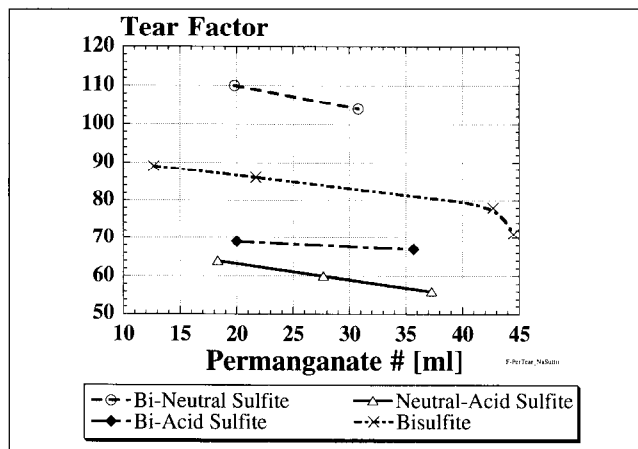
Fig. 2

Breaking length vs. Permangante number:
Comparison of four sulfite processes using spruce wood[12].
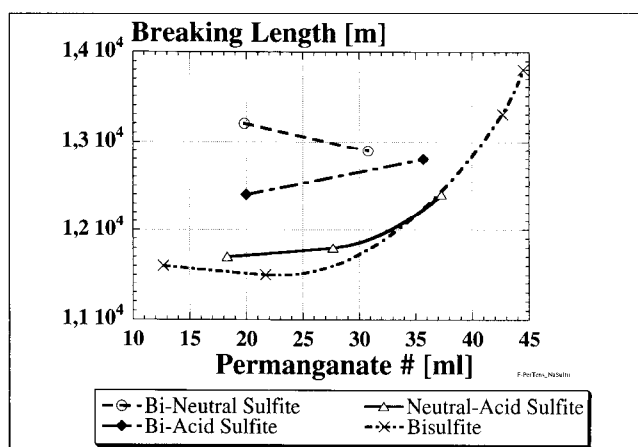
Fig. 3

Xylose and mannose content of unbleached spruce sulfite pulps: Comparison of four sulfite processes using spruce wood[12].
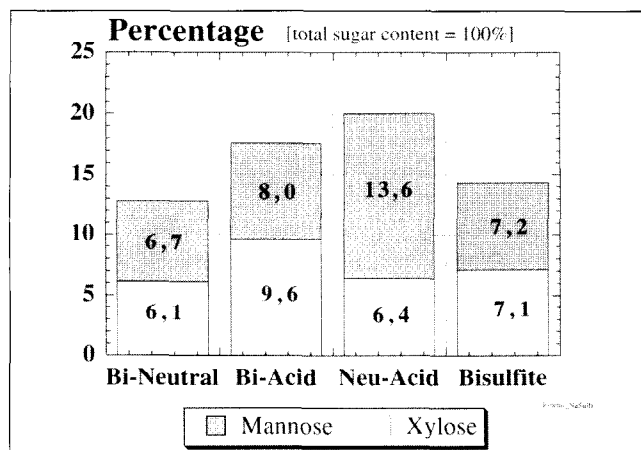
Fig. 4

Pulp yield is lowest at a given residual lignin content in case of the bisulfite-neutralsulfite process (figure 1). When cooking at a pH of 5.5 or higher, the delignification is exceedingly slow, even at temperatures up to 175° C. A rather high loss of hemicelluloses at a high level of residual lignin is the result of low pulping selectivity. Modifying the conventional Magnefite into a Two-Stage Neutral Magnefite process, where the second stage is carried out at pH levels slightly below 7, enables the production of pulps with significantly better strength properties at comparable yield values[14]. The better yield values compared to the use of sodium could possibly be explained by the lower pH-levels and thus a higher hemicellulose retention in case of magnesium. The increase in pH was accomplished by the addition of MgO to the digester during the cooking cycle. The first stage was cooked at 166° C, using a bisulfite liquor of pH 3.7. At the end of the first stage, the liquor was neutralized in the digester to a pH of 6.0 to 6.5. The pulp is then given a second-stage cook at 170° C. Precipitation of monosulfite was avoided provided that the total amount of SO_2 was reduced by one per cent and the pH level was not raised above 6.7.

Among the sulfite pulping technolgies investigated, the bisulfite-neutralsulfite process showed the best strength properties as indicated in figure 2, tear factor vs. permanganate number and figure 3, breaking length vs. permanganate number. Strength values were only 5 to 20% below those of the kraft pulps made from the same wood chips. Increasing the alkalinity of the terminal stage resulted in a significant increase in tearing resistance. No such marked effect of alkalinity was observed in bursting strength. Comparing the Magnefite with the Two-Stage Neutral Magnefite process, which was realised at the Weyerhaeuser Cosmopolis mill, better strength properties including tensile, burst and tear factors were obtained in the case of the latter. The increase of pH as a result of the addition of sodium carbonate in the second stage has been described as the Sivola process[15]. Strength properties comparable to kraft pulps but with a higher residual lignin content were reported for Sivola pulps where the bisulfite stage was terminated at an early stage of delignification[16]. The tear factor was directly related to the pH in the cooking liquor of the terminal stage.

Replacing the bisulfite by an acid sulfite pretreatment in very moderate conditions (high acidity and low temperature) converts the Sivola process to a very flexible process for the production of dissolving pulps. The Rauma process, a development which parallele the Sivola process, consists of a three-stage cook for use on spruce and pine wood. The first stage is a bisulfite stage, pH 3-4, for the impregnation of the chips. The second stage is like a conventional acid sulfite stage. At the end of this

stage, SO_2 is gassed off, when the desired viscosity of the pulp is reached. In the third stage, sodium carbonate is added to neutralise the cook and extract the low molecular weight carbohydrates. The pulp purity, measured as the α-cellulose content, is controlled by temperature, time and pH. At 170° C and a pH level above 8, α-cellulose contents above 95% have been obtained.

The higher brightness of bisulfite and acid sulfite is an important advantage over kraft pulps. Most of this is lost when the pulps are subjected to a neutral or even alkaline second stage. The bases, as well as the technology applied for increasing the pH in the second stage were important factors in determining pulp brightness. Generally, higher brightness levels were found when alkali was injected directly into the first-stage liquor[24]. The best results were obtained with the magnesium bisulfite/magnesium hydroxide process whereas the use of the ammonium-base systems resulted in very poor brightness levels. Brightness can also be improved by lowering the pulping temperature. A 20° C reduction in the first-stage temperature resulted in a brightness improvement of 9 points, with the disadvantage of a much longer pulping cycle (6.3 h at 145° C vs. 1.5 h at 165° C).

Bisulfite - Acid Sulfite & Neutralsulfite - Acid Sulfite

The bisulfite acid sulfite process was primarily developed for the partial use of extractive-rich pine heartwood and tannin-damaged spruce. Yield and strength properties are reported to be slightly better compared to acid sulfite pulps and even better than bisulfite pulps in the case of breaking length, e.g. Magnefite pulps (figure 3). The pretreatment of the chips with a sulfite cooking liquor of pH levels higher than 6-7 leads to a significant increase in yield compared to the known one-stage processes. The two-stage neutralsulfite-acid sulfite pulping process was developed by Stora Kopparberg for use with pine wood[17] and was first applied in their mill at Skutskär. The higher initial pH leads to the better impregnation and sulfonation of the wood. Condensation reactions between some of the most reactive lignin groups and some phenolic extractives of the wood are greatly suppressed. Therefore an acceptable delignification of the extractive-rich pine heartwood in the subsequent acid sulfite cook can be obtained. Further investigations have shown that the neutral first stage also effects the carbohydrate yield, thus resulting in a more selective delignification compared to conventional sulfite pulping[25]. The research group of Billerud found that the yield advantage obtained with the neutralsulfite-acid sulfite method is due mainly to a larger amount of glucomannan in the pulp.

Glucomannan stabilization

Rydholm and Annergren observed that part of the glucomannans in sprucewood become more resistant to acid hydrolysis during the sulfite cooking if the wood is pretreated with a neutral or slightly alkaline liquor[16, 19-21]. The increased resistance to acid hydrolysis is supposed to arise from a higher rate of lateral order caused by deacetylation of the glucomannans followed by the adsorption of the mannan backbone on the cellulose. The gain in pulp yield is closely related to the deacetylation of the glucomannans. The acetyl groups are easily split by alkaline treatment while acid treatment shows little effect. According to Rydholm, it is also probable that alkaline pretreatment facilitates the adsorption of the glucomannan to some extent by increasing the accessible surface of the cellulose through swelling. The stabilising reaction takes place within the fiber as it was shown that the glucomannans are most abundant in the S_2-layer, where the greatest part of cellulose is located[2]. The yield increasing effect of an initial neutral or slightly alkaline stage was observed by several researchers. A three per cent

increase compared to one-stage bisulfite cooking was discovered by Weeneras, a four per cent increase compared to bisulfite and a 6-8 per cent increase compared to bisulfite-acid sulfite was reported by Sanyer (figures 1 and 4). A carbohydrate mass balance made by Sanyer revealed a 5.6% and a 6.9% higher mannose concentration in the neutralsulfite-acid sulfite compared to the bisulfite-acid sulfite and the bisulfite-neutral-sulfite pulps, respectively (see figure 4). Different results have been published regarding the stabilising effect of hardwood pulping. Sanyer's results showed that in the case of hardwood pulping the yield remained constant at a level of around 43% for all different concepts of multi-stage sulfite cooking. A yield, almost four per cent higher, was achieved using kraft pulping technology on the same hardwood species due to a better retention of the xylan fraction. The small content of glucomannan in the hardwood species was argued to be the reason for the non-existent stabilisation effect of two-stage pulping.

In contrast with these results, Croon also observed a yield gain up to 4-5 per cent for hardwoods[22] He has found that a starting pH of 8-9 and a temperature of 150° C for 1-2 hours are suitable conditions in stage one for the pulping of birch and aspen woods. A birch pulp of kappa number 20 has a xylan content (22% vs. 26%) and thus a total pulp yield (54% vs. 54%) close to that of a birch kraft pulp. In line with these results, improved xylan retention can also be achieved in the case of multi-stage sulfite pulping using appropriate conditions in the first stage.

The neutralsulfite-acid sulfite concept has also been applied to magnesium base cooking due to findings that the precipitation of the magnesium sulfite / magnesium hydroxide system can be avoided up to pH levels of 9.0, as previously mentioned. The process, developed and realised by Weyerhaeuser, comprises a first stage cooking at 150° C using a cooking liquor having a pH ranging from 5.2 to 6.0. This liquor is removed at the end of the first stage and is replaced by an acid sulfite solution for second-stage cooking at 135° C. No particular advantage was realized by Weyerhaeuser when cooking at pH values above 6.0, whereas Nilsson and Stockman reported the maximum yield increase to be obtained only at a pH level of 9.0. The different observations might be due to the fact that an initial pH of 9.0 corresponded to a final pH of 6-7 for the first cooking stage.

Strength properties of neutralsulfite-acid sulfite pulps are comparable to those of acid sulfite pulps, burst and tensile strengths are even comparable to bisulfite (Magnefite) whereas the tear strength was definitely lower compared to acid sulfite pulps (figures 2-3). Beating requires less energy compared to conventional sulfite softwood pulp. A certain tensile strength is thus reached after a much smaller power consumption for the two-stage pulp. It also shows that this same tensile strength is reached for the neutralsulfite-acid sulfite pulp at a lower beating degree which means better drainage properties[23]. Surprisingly the two-stage pulp has a higher bulk than the conventional sulfite at a certain tensile strength.

Despite considerable effort to develop a real alternative to the kraft process, the steady displacement of sulfite pulping technology has continued to date. For some special products, especially for TCF-bleached dissolving pulps and in some exposed places, sulfite pulping is still of some importance. In the mid and late eighties extensive investigations were started by Patt, Kordsachia and their co-workers on the improvement of sulfite pulping technology[26-29]. As a result the following two processes have been developed:

• Methanol-Sulfite
• ASAM

Methanol-Sulfite

Their first development was the use of methanol during the magnesium-sulfite cooking of softwoods with a high resin content. A magnesium bisulfite cook with no ($SO_2/MgO = 3,2$) or only a low concentration of free SO_2, a rather high total SO_2-charge of 25% on o.d. wood a maximum temperature of 165° C and a methanol concentration of 25 vol% were reported to be the preferred conditions[30-32]. The advantages of this development were the possibility of using extractive-rich softwood species like pine, the significant increase of yield (figure 5) and a substantial improvement in strength properties (figures 6 and 7). Some of the results published by Kordsachia et al. are summarised in figures 5 - 7. The clear improvements were, however, not sufficient to achieve strength properties similar to kraft pulps.

**Yields vs. kappa number:
Comparison of spruce-acid sulfite, -bisulfite
and -bisulfite-methanol pulps[30].**
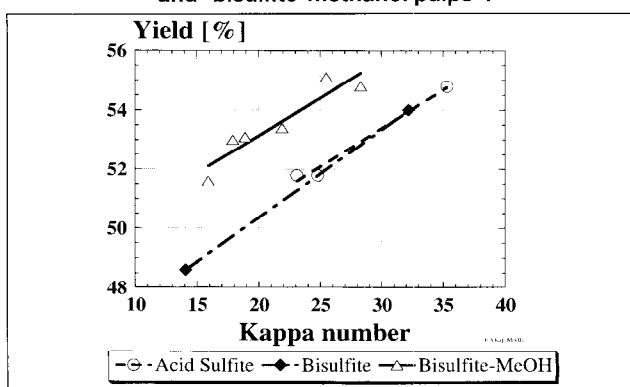
Fig. 5

**Breaking length at 35°SR vs. kappa number:
Comparison of spruce acid bisulfite, spruce bisulfite
and spruce bisulfite-methanol pulps[30].**
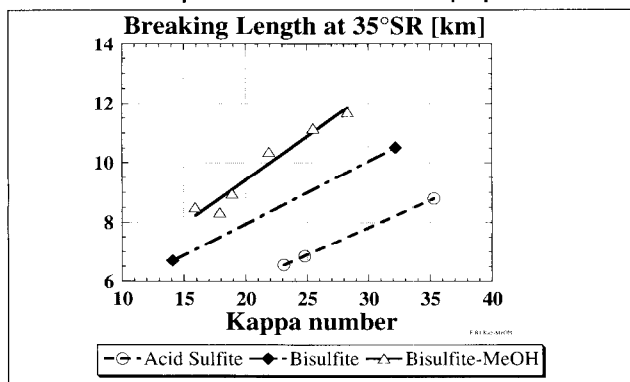

Fig. 6

**Tearing resistance at 35°SR vs. kappa number:
Comparison of spruce-acid sulfite, -bisulfite
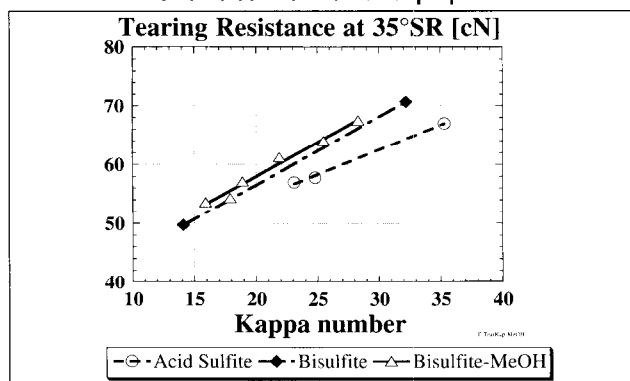and -bisulfite-methanol pulps[30].**

Fig. 7

ASAM

This was only possible with the development of the ASAM process (=alkaline sulfite anthraquinone and methanol) which was carried out at the University of Hamburg, Germany, in 1985. Since then, extensive investigations have been carried out at laboratory level and in a large pilot plant in Baienfurt, Germany, for several years. The development of the ASAM process covered all the necessary areas of pulp production, such as the chemistry[44-60] and the technology[29, 33-43] of cooking and bleaching, the technology and chemistry of chemical recovery[61-66] as well as process economy[27, 28]. Today ASAM is an established pulping technology which is able to meet the demands of the future to a higher extent than the kraft process. ASAM pulps can be obtained at a significantly higher yield, they show much better strength properties and are easier to bleach, in TCF-sequences in particular. Chemical recovery, often considered the most striking disadvantage of ASAM technology, certainly bears the long-term advantage of enabling the separation of sodium and sulfite components at the green liquor conversion stage. The enriched inorganic material, mainly heavy metals, can be removed very efficiently from the chemical loop which is an important pre-requisite for a closed water cycle in the bleaching plant.

In the first half of the nineties, extensive investigations were carried out in the Lenzing pulp research group to compare the chemistry and technology of the conventional pulping processes, acid sulfite and kraft, with the new highly developed processes, ASAM and SODA-AQ, using beech and eucalyptus for the production of both paper and dissolving grade pulps. Some of the results have been published elsewhere[67-71].
A selection of the results concerning the paper grade pulping of beech wood will be presented in the part of this paper which follows.

Experimental:

Wood:
Beech wood of 19.3% xylan (backbone), 1.6% mannan, appr. 2 % rare sugars (galactan, rhamnan, arabinan), 0.29% DCM-extractives, 0.40% total ash and a dry content of 54.9% was chipped at Lenzing AG with an average thickness of 4.0 mm, an average length of 26.7 mm and an average width of 14.0 mm.

Cooking:
A 2 and 10 l digester with external circulation. Heating and temperature control was conducted with both a heating jacket and an external heat exchanger. Circulation was kept at 530 to 580 ml/min in the case of the 2 l digester and 3350 ml/min in the case of the 10 l digester.

Bleaching:
O(P) -delignification:
A MC-mixer/reactor with a volume of 2.5 l was filled with pulp suspension. The reactor was closed and evacuated before the prepared bleaching chemicals were added to the pulp suspension by means of a sluice and the consistency was set at 10%. The oxygen gas was then introduced until the desired pressure was reached. The pulp suspension was heated to the preset reaction temperature by means of an indirect heating system. Mixing was carried out under fluidisation conditions for a total time of 10 sec. The temperature was kept constant throughout the total reaction time, whereas the pressure was linearly reduced to a final pressure of 3 bar.

Medium Consistency Ozone Bleaching:
The same mixer/reactor was used as was described above. The reactor was filled with an acidified (pH 2) pulp suspension to leave a defined free gas volume. The temperature was set to 50°C. Once the reactor was evacuated, compressed gas containing ozone was introduced until the desired pressure was reached. The specific amount of ozone was controlled by varying the pressure and ozone concentration. The mixing time was 10 sec under fluidising conditions. After stopping the mixer, the pressure was kept up for a certain time.

Peroxide Bleaching:
Peroxide bleaching was carried out in PE-flasks plunged into an agitated wather bath.

Analycal Characterization:
The kappa number [modified Tappi T236 cm-85], intrinsic viscosity [Scan CM 15:881, ISO-brightness [ISO 3688/2470] and Berger brightness = R_y + 3 (R_z - R_x); R_x, R_y, R_z characterise the reflectance of red, green and blue colour light. Yellowing factor after 72 h according to Reinhardt et al[72] = (WG$_0$-WG$_{72}$)/ WG$_0$100. Strength properties according to Zellcheming-methods [ZM V/3/62, ZM V/12/57], molecular mass distribution [GPC of pulps dissolved in LiCl/DMAc; RI detection; calibration with polystyrene standards], DOC / TOC [Astro UV reactor], carbohydrates [HPLC after complete hydrolysis of the polymer to the monomers].

Results
Cooking
A limited series of trials was performed for each cooking process to select the appropriate conditions for the production of unbleached pulps which were subjected to TCF-bleaching (summarised cooking conditions see table 1).

Cooking conditions

Parameter	Unit	ASAM	KRAFT	SULFITE
Total Alkali as NaOH	%	25	20	
Na$_2$SO$_3$:Na$_2$CO$_3$:NaOH	as NaOH	20:70:10		
MgO	% o.d.			3,0
Total SO$_2$	% o.d.			16,2
Methanol	Vol%	15		
Anthraquinon	%	0,1		
liquor-to-wood ratio		4:1	3,5:1	2,7:1
Impregnation time	min			80
Heating-up time	min	150	90	120
Time at Tmax	min	150	60	240
Cooking Temp. (Tmaxl)	°C	180	165	130

Table 1

The ASAM pulp was characterised by low kappa numbers, high brightness and viscosity values, whereas kraft pulping resulted in relatively high kappa numbers, low brightness and moderate viscosity levels. The sulfite pulp showed the highest brightness at a rather high kappa number level and an unexpectedly high viscosity as can be seen from table 2.

Characterisation of unbleached pulps

Parameter	Unit	ASAM	KRAFT	SULFITE
Total Yield	% o.d.	50,7	49,7	48,8
Reject	% o. d.	1,2	1,1	0,7
Viscosity	ml/g	1285	1119	1203
Brightness	% ISO	52,7	29,4	57,4
Kappa Number		7,8	18,3	16,7

Table 2

The highest pulp yield of 50.7% was obtained by ASAM pulping despite the significantly lower content of residual lignin compared to the kraft (-l0,5 kappa number units) and the sulfite (-8,9 kappa number units) pulps. Carbohydrate analysis clearly showed that the yield advantage of the alkaline cooked pulps was mainly due to the higher resistance of xylan under these conditions. The highest xylan content, 22.9% of o.d. (oven dried) pulp, was measured for the kraft pulp which corresponds to a retention of 63% of the wood xylan, followed by the ASAM pulp with 18% of o.d. pulp or 50% of the wood xylan and finally the acid sulfite pulp with only 11.7% of o.d. pulp or 31% of the wood xylan. The sulfite pulps, however, showed the highest slightly more than 98% of the wood cellulose content, which corresponds to a glucan content in the unbleached pulps of 77.4% and 80.9% of o.d., respectively. Only 91% of the wood cellulose was retained in the kraft pulp, representing a glucan content of 74% o.d in the pulp. It can, therefore, be concluded that pulping selectivity is most pronounced in the case of ASAM pulping thus combining the advantages of both kraft and acid sulfite pulping.

TCF-bleaching

The unbleached pulps were subjected to a five-stage TCF-bleaching sequence, OP-Z-EOP-Z-P. The conditions were kept constant throughout the bleaching stages to get a better understanding of the differences in bleachability (tables 3 and 4).

Bleaching Conditions using an OP-Z_1-EOP-Z_2-P-sequence

Parameter	Unit	OP	Z_1	EOP	Z_2	P
Temperature	°C	98	50	75	50	70
Time	min	120		90		180
Oxygen pressure	MPa	0,2		0,2		
Ozone charge	% o.d.		0,34		0,30	
NaOH-charge	% o.d.	3,0		2,0		0,6
H_2O_2-charge	Vol %	0,2		0,4		0,6

Table 3

Characterisation of TCF-bleached pulps

Parameter	Unit	ASAM	KRAFT	SULFITE
Bleaching yield	% o.d. pulp	96,8	95,9	92,5
Total yield	% o.d. wood	47,9	46,6	44,5
Brightness	% ISO	89,6	86,1	89,1
Viscosity	ml/g	909	801	906
Kappa number		0,5	1,1	1,1
Glucan	% o.d. pulp	80,9	76,3	86,2
Xylan	% o.d. pulp	17,7	22,5	10,8

Table 4

Viscosity vs. brightness: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
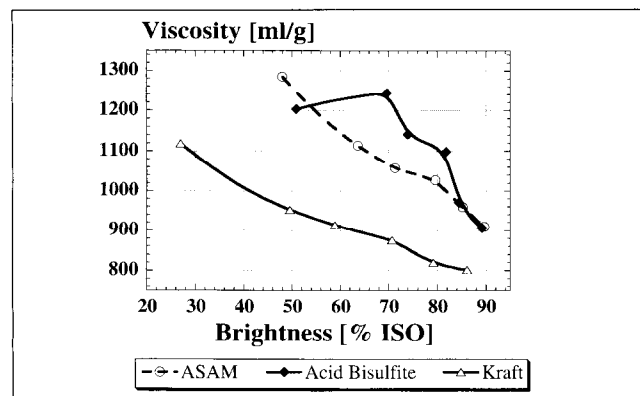
Viscosity [ml/g]

Fig. 8

Brightness vs. kappa number: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
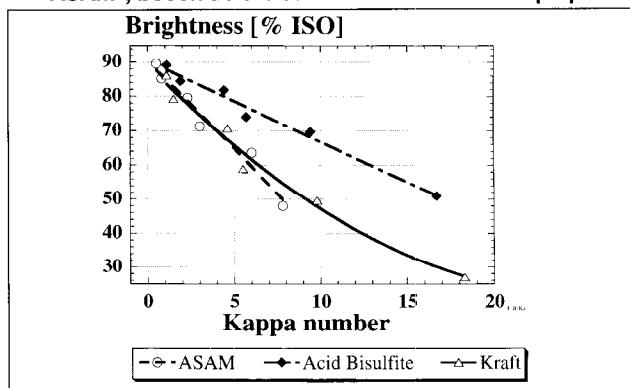
Brightness [% ISO]

Fig. 9

The sulfite pulps showed a very similar course of viscosity dependent on the brightness as indicated in figure 8. The final brightness was very close to 90% ISO in both cases at the same level of intrinsic viscosity (906 and 909 ml/g for acid sulfite and ASAM). Interestingely, the kappa number brightness relationship of the ASAM was more similar to the kraft than to the acid sulfite pulp (see figure 9). Consequently, the kappa number of the fully bleached ASAM pulp was lower compared to that of the acid sulfite pulp (0,5 vs. 1, 1) at the same level of final brightness. The alkaline and the acid cooked pulps also showed big differences in their bleaching yield. The bleaching losses of the acid sulfite pulp were almost twice as high throughout all stages as compared to the ASAM and kraft pulps (7.5% vs. 3.2% and 4. 1 %). The higher yield losses in the course of acid sulfite pulp bleaching can be identified with a higher loss of carbohydrates which can be attributed to the higher fraction of low molecular weight carbohydrates (cellulose and hemicellulose) due to their better accessibility towards heterogeneous bleaching reactions in general and to their susceptibility to alkaline peeling reactions in particular. Figure 10 displays the corresponding results in terms of yield kappa number relationships.

Brightness vs. kappa number: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.

Yield [%]
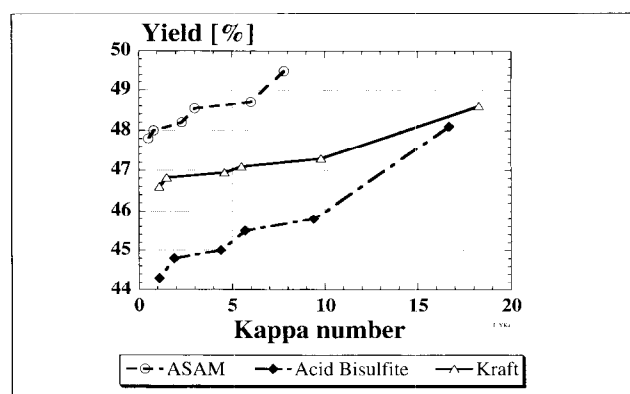
Fig. 10

Characterisation of Bleached Pulps

Brightness stability was characterised as the resistance to light induced yellowing. The pulps were irradiated with xenon light up to 100 h. Berger brightness was measured after 24 h, 72 h and 100 h of irradiation time. The course of brightness which is displayed in figure 11 clearly demonstrates the poor brightness stability of the acid sulfite pulps whereas the brightness of the ASAM pulp was almost unaffected. The following order of brightness stability was obtained when calculating the yellowing factor after 72 h according to Reinhardt et al.[72] ASAM (7.0) > kraft (13.2) << acid sulfit (32.1).

Berger brightness vs. exposure time to xenon light: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
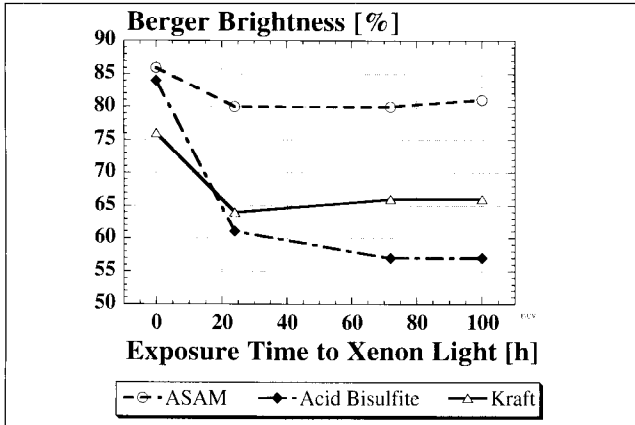
Fig. 11

The beating rate in a Jokro mill was rather fast and comparable for both sulfite pulps, the ASAM and the acid Mg bisulfite pulp, whereas the kraft pulp was significantly more resistant to beating (see figure 12). The strength properties, measured as tear and tensile index, were by far the best for the ASAM pulp. After 10 min of beating the tear index of the acid Mg bisulfite pulp was 5.6, of the kraft pulp 7.8 and of the ASAM pulp 9.6 mN/M2g (see figure 13). The corresponding tensile indices were 46.1, 59.8 and 75.5 Nm/g, respectively (see figure 14).

Tear index vs. beating time: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
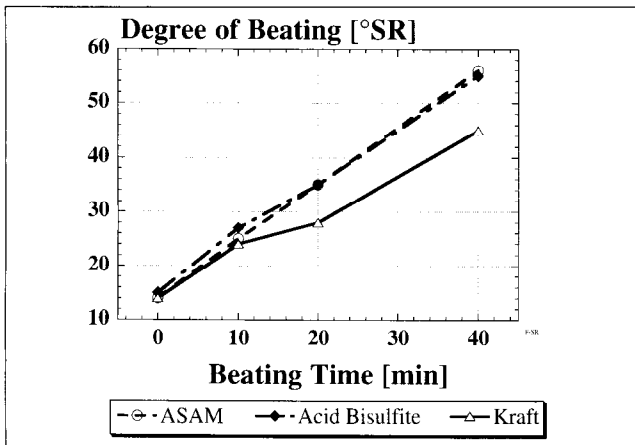
Fig. 12

Tear index vs. beating time: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
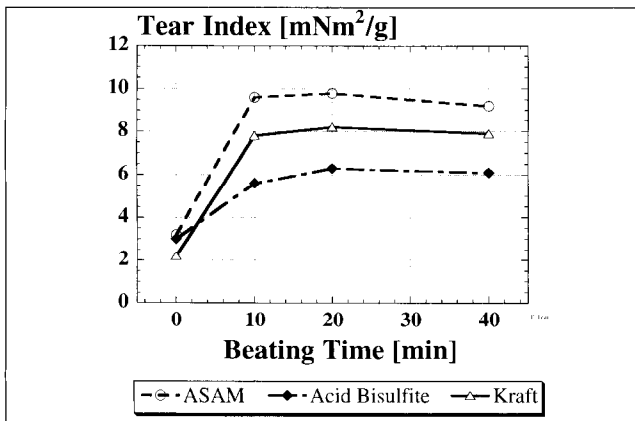
Fig. 13

Tear index vs. beating time: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
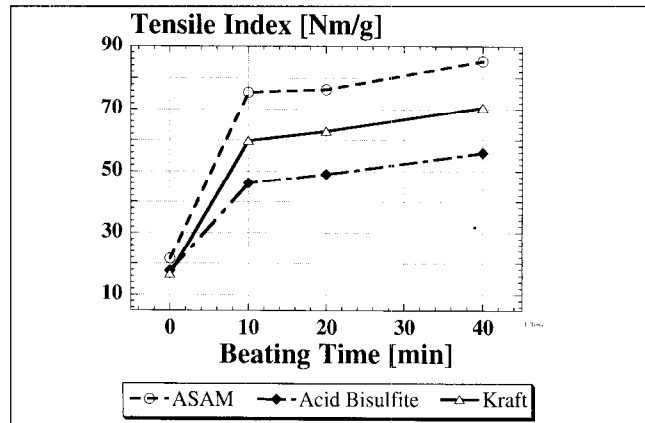
Fig. 14

At the same level of beating degree, significant differences in strength properties were still visible as can be seen in table 5.

Strength properties at 30°SR of fully TCF-bleached ASAM-, kraft- and acid magnesium bisulfite pulps.

Parameter	Unit	ASAM	KRAFT	SULFITE
Tear Index at 30°SR	mNm²/g	9,7	8,2	5,9
Tensile Index at 30°SR	Nm/g	74,1	63,7	47,2
Burst at 30°SR	kPam²/g	4,37	3,19	1,53
Tear growth test at 30°SR (Brecht Imset)	mNm/m	1082	814	596

Table 5

The higher strength potential of the ASAM pulp by a comparison with beech acid Mg sulfite- and kraft pulp was already identified by Kordsachia and Patt years ago[44]. The level of strength properties was even higher for ASAM and kraft pulps, possibly due to a higher unbleached kappa number and a lower final brightness in case of the TCF-bleached ASAM pulp.

The outstanding strength properties of the ASAM pulp can partly be explained by measurable morphological and molecular parameters. Using eucalyptus as a wood source it could be shown that acid magnesium bisulfite pulp has a much higher content of short fibres compared to the pulps cooked under alkaline conditions, like kraft and ASAM pulps. The proportion of fibres smaller than 0.50 mm was 76% in the case of the acid Mg bisulfite, 39% in the case of the kraft and 35% in case of the ASAM pulp[69]. It can be assumed that corresponding results are transferable to the use of beech wood. When investigating the molecular level by means of molecular mass distribution, distinct differences between the pulps become visible (figure 15). The low molecular weight peak (P1) follows a similar pattern in the case of alkaline cooked pulps. The position of the maximum P 1 is shifted towards a higher molecular weight in the case of the ASAM pulp, whereas the area of P 1 is higher in the case of the kraft pulp in accordance with the slightly higher hemicellulose content (see table 6). The second peak in the high molecular weight region (P2) has a lower half width and a higher maximum molecular weight in the case of the ASAM compared with the kraft pulp. The latter shows a clear degradation of the high molecular weight cellulose which is recognized by a shoulder in the high molecular area of P2. A small peak area and the maximum shifted to a very low molecular weight are typical of the acid Mg sulfite pulp's low molecular weight peak (P1). This information on the gel permeation chromatogram corresponds to a low

GPC of TCF-bleached paper grade pulps: Comparison of beech ASAM-, beech acid bisulfite- and beech kraft pulps.
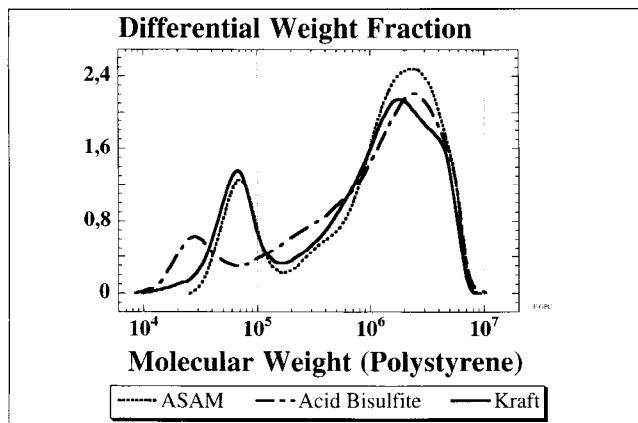
Fig. 15

Strength properties at 30°SR of fully TCF-bleached ASAM-, kraft- and acid magnesium bisulfite pulps.

Parameter		Unit	ASAM	KRAFT	SULFITE
Low molecular weight peak P 1	Maximum	g/mol PS	$7,05 \cdot 10^3$	$6,69 \cdot 10^4$	$2,80 \cdot 10^3$
	Rel. Peak Area	%	78	100	21
High molecular weight peak P 2	Maximum	g/mol PS	$2,18 \cdot 10^5$	$1,79 \cdot 10^5$	$2,22 \cdot 10^6$
	Half Width	g/mol PS	$4,86 \cdot 10^5$	$5,19 \cdot 10^6$	$5,92 \cdot 10^6$

Table 6

content of hemicellulose of very low molecular weight which certainly helps to partly explain the observed overall poor strength properties. Interestingly, the position of the high molecular weight peak (P2) is shifted even to a higher value compared to the chromatogram of the ASAM pulp. The proportion of the high molecular weight cellulose is, however, lower as part of these molecules are degraded to medium-molecular weights between 1×10^5 and 8×10^5 (PS) recognizable by both an accumulation of molecules in the region of the gel permeation chromatogram and a higher half width of the P2.

Conclusions:

Classical sulfite pulping technology for the production of paper grade pulp, the one-stage acid bisulfite process, is becoming less and less competitive compared to modern kraft technology. The main advantages of sulfite pulping, the high unbleached brightness and the good bleachability, especially under TCF-conditions, cannot compensate for its high sensitivity to the wood raw material, the much lower strength properties and the lower efficiency of the recovery of cooking and bleaching chemicals. The development of multi-stage sulfite concepts was undoubtedly successful in improving yield, lowering the sensitivity to high resinuous wood species and raising strength properties. The improvements, however, were not sufficient to again make the conventional sulfite process a serious competitor for kraft pulping technology.

There is no doubt that sulfite pulping will maintain its position in the pulping industry, especially when products of high brightness without the use of chlorine compounds, special surface properties and not too high strength properties are desired. Besides the simple one-stage concepts, the acid magnesium and the magnesium bisulfite (=Magnefite), one of the two-stage modifications, the neutralsulfite-acid sulfite process, will retain its importance mainly because of the comparatively low sensitivity to high resinuous wood species, the very high yield (primarily when using softwoods) and the (limited) possibility of using magnesium as a base.

In the mid and late eighties successful investigations were commenced by Patt, Kordsachia and their co-workers with the aim of developing a new pulping process which combines the advantages of both sulfite and the kraft pulping technology. The targets have been fully achieved with the development of the ASAM process. Since its invention in 1985, extensive investigations at laboratory and pilot plant level have been carried out to make the ASAM process an established pulping technology which is able to meet the demands of the future

• lowest possible environmental pollution
• minimizing the amount of fresh water
• outstanding product quality for both paper and dissolving grade pulps
• low demands on wood quality

better than any existing pulping process.

This paper describes the performance of ASAM pulping using beech wood for the production of TCF-bleached paper grade pulp. The results clearly confirm the superiority of the ASAM compared to the acid magnesium bisulfite and the kraft pulping process regarding wood yield, bleachability, strength properties and brightness stability. Despite the use of beech wood, strength properties between those of softwood kraft and softwood acid sulfite pulps can be obtained by using ASAM pulping technology.

Recovery of cooking and bleaching chemicals (sodium sulfite and sodium hydroxide) often seen as the main drawback of the ASAM process, is based on a proven technology which has been further developed in the last few years. The existing technology of sodium sulfite recovery shows two decisive advantages over kraft recovery which could be important for the pulping industry in the future. It enables both the separate recovery of sodium and sulfite components and a very efficient precipitation of inert material (especially silicon) by controlling pH in the green liquor which can be seen as a pre-requisite for the recovery of sodium hydroxide for bleaching purposes and for a closed mill.

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BACELL'S SOLUCELL - A NEW DISSOLVING PULP FOR HIGH QUALITY REQUIREMENTS

Walter Peter, Alberto Lima, Bacell S. A., Brasil

In 1996 BACELL, a joint venture between KLABIN, the biggest producer of pulp and paper in South America and LENZING, a world wide leading rayon company started the production of dissolving pulp. The design capacity is 115000 t/y prehydrolysis dissolving pulp from eucalyptus.

BACELL has implemented the most modern technology in terms of quality and environmental protection, like vapour phase prehydrolysis and TCF (totally chlorine free) - bleaching with ozone.

In the meantime an exzellent quality for the viscose fiber production could be established with good specification data like high alpha cellulose content and low kappa number.
The SOLUCELL was intensively tested for its behaviour in the viscose process and showed very positive results in the laboratory, the pilot plant and the mills: the reactivity was high, especially when some surfactant was added, the brightness stable and the fiber properties on a high level.

Bacell, a new player on the field of dissolving pulp production, is trying its best to be one of the top scorers. The conditions are pretty good for that, as Bacell has implemented the latest technology available.

1. Introduction

The Bacell mill is a joint venture between KLABIN, the biggest pulp and paper producer in South America, and LENZING, an Austrian rayon producer with worldwide activities. The mill itself is a retrofit of an existing sisal pulp mill, but it was completely modernized and to a big extent rebuilt.
The nominal capacity of this mill is 115000 tons per year of dissolving pulp and it is located close to the city of Salvador in Bahia, about 1500 km north of Rio de Janeiro.

The cornerstones of the project were:
- Implementing the latest available technology by using as much of the existing equipment as possibte and reasonable.
- Achieving a very high quality standard to make a relatively small mill profitabel
- Using the wood resources of the area, in this case Eucalyptus Grandis, Urophylla and a hybrid of both the so called Urograndis from plantations.
- Providing a high level of environmental protection.

The project was made together with Jaakko Poyry and definitively started in June 1994. In January 1996 we succeeded to start cooking and on March 8, the first prime grade dissolving pulp bale left the drying machine.

2. Technology

In terms of technology Bacell chose the most modern alternatives, implementing also some new developments like
- In cooking: the VISBATCH-process, which is a combination of the ENERBATCH-process plus a vapor phase prehydrolysis.
- In bleaching: the first TCF bleach plant for prehydrolysis kraft pulp.
- In dewatering: the TETRAFORMER, a new development of a low energy wet end.

2.1 Cooking

The sulfite process predominates world wide the production of dissolving pulp. Acid sulfite process is mainly used in single stage cooking because of its rapid hydrolysis of hemicellulose and beta-cellulose as well as its good delignification rate.

The first prehydrolysis kraft dissolving pulp mill started production in 1945, processing wood into cord fibres for tires. In 1992 the production of prehydrolysis kraft pulp was 1,1 million tons world wide, of which 40 - 45 percent was based on hardwood. Sulfite technologies are restricted to certain wood species, mainly hardwoods. Prehydrolysis kraft can also use softwood and wood species rich in resins.

In the case of BACELL, the choice of the prehydrolysis kraft process was given by the already existing recovery boiler.

2.1.1 Vapour phase prehydrolysis

Kraft pulps require a lower hemicellulose content to have the same reactivity as sulfite pulps. This is achieved by an acid pre-treatment at high temperature where the hemicellulose is split into oligomer carbohydrates and can be dissolved. So far this prehydrolysis was mostly carried out in an aqueous phase, which was a major disadvantage of the kraft process as it includes the handling and disposal of the prehydrolysate which requires high amounts of energy and evaporation capacity. Scaling and clogging in tubes and valves are frequently caused by resins and the reaction products of hemicellulose and sugar. In some few cases it was even used as source for pentosanes.
A vapour phase prehyodrolysis, as it is already exceptionally used in some mills in Brazil and Eastern Europe, should prevent these problems. IVA had optimized the procedure for steam heating of the chips, by providing a uniform injection and distribution of the steam in the digester. This uniformity in combination with a neutralization with hot black and white liquor and followed by a hot displacement leads to the same results as water prehydrolysis, avoiding its high energy consumption, increased evaporation capacity and pollution.

This process enables Bacell to reach a remarkable low pentosane content, quasi tailor made for each client, without the disadvantageous treatment of the hydrolysate.

2.1.2 Process Description (Fig. 1 and 2)

Prehydrolysis and cooking are accomplished periodically in three batch digesters, equipped with a steaming and a liquor circulation system. High pressure tanks for hot white liquor, neutralization liquor, hot and warm black liquor are the basis of the modern Enerbatch technology.

- Chip filling is accomplished by means of low pressure steam in a Svensson chip filling system, which assures a high chip packing degree in the digester.
- For steam heating and prehydrolysis, the digester is heated up with medium pressure steam; hydrolysis of hemicellulose is achieved by organic acids, mainly acetic acid, which are formed during the process and after 40 minutes the major part of the pentosanes are hydrolysed and dissolved.

- During neutralization and hot displacement the pressurized digester is filled with caustic liquor, consisting of hot black and hot white liquor. This neutralizes the organic acids formed during prehydrolysis and adds the alkali charge required for cooking. Part of the neutralization liquor is displaced to the neutralization liquor tank during the hot displacement.
- The digester is kept at cooking temperature until the desired degree of delignification is reached.
- Cooking is completed by displacing hot cooking liquor with washing filtrate from the washers. In this way the pulp in the digester is cooled down below 100°C and pumped into the blow tank. This cooling is essential to stop degradation reactions suddenly at a certain point.
- The white liquor is heated up by the spent liquor from the previous cook, maintaining the heat energy in the system. With this modern types of displacement cooking, the steam consumption can be reduced to around 40% of the conventional ones.

Steam prehydrolysis –
kraft displacement cooking process.

Fig. 1

Process – Step

Fig. 2

2.2 Bleaching (Fig. 3)

From the very beginning of the project, there were no doubts, that a new pulp mill must have a TCF-bleaching sequence. On the one hand to gain the advantages on the market, on the other hand to ensure that also the future environmental regulations will be fulfilled.

The only realistic alternative, producing ECF-pulp by using chlorine dioxide instead of ozone, requires more or less the same operating, but higher investment costs.

After an intensive evaluation, the bleaching sequence OO-A-ZQ-P was established, using only oxygen based chemicals as oxygen (O), ozone (Z) and hydrogen peroxide (P) besides sulfuric acid (A) and EDTA (Q) for metal removal.

Bleaching Conditions

	O	O	A	Z	Q	P
Time (min)	15	60	50	2	40	360
Temperature (° C)	105	110	60	55	60	80
Consistency (%)	11	11	5	10	9	11
pH	11	10,5	2,5	2,2	5	10,5
Chem. Dosage (%)	2,2 O₂		1,8 H₂SO₄	0,3 O₃	0,1 EDTA	0,8 H₂O₂
	2,0 NaOH (as oxidized white liquor)			0,5 H₂SO₄		0,9 NaOH

Fig. 3

2.2.1 Development

LENZING has installed its ozone bleach plant in 1992, being the first installation world wide at that time. Based on the exzellent experience concerning pulp quality and operations with sulfite pulp, our aim was to transfer this technology to prehydrolysis kraft pulp.

The basic work was done in LENZING's research department, in cooperation with KVAERNER Pulping and RIOCELL.

2.2.2 Process description

- Facing the very low lignin content of high quality dissolving pulp, it is evident that the oxygen delignification stage has to be extremely efficient. Therefore a two stage version without intermediate washing was chosen. The benefit of an intermediate washing was not big enough to justify an additional installation. The conditions in this reaction are rather drastic with temperatures up to 125°C, an oxygen pressure of 0,7 MPa and an alkali charge of approximately 25 kg/bdmt. So we can gain a delignification rate of more than 70% at a reasonable viscosity loss of around 25%. It is well established that trace levels of transition metals have a profound negative impact on ozone and peroxide bleaching as well as on the rayon process. The presence of transition metals, particularly Co, Fe, Mn and Cu ions result in both, excessive ozone and hydrogen peroxide consumption by self decay and in degradation of carbohydrates, apparently caused by free radical reactions. To use ozone and hydrogen peroxide effectively in the subsequent bleaching stages, transition metals must be removed or at least deactivated.

A major part of our eucalyptus is growing on the typical red, iron containing soil you can find in Bahia. So the logs have a relatively high content on iron and the risk to contaminate the process and the pulp was immanent.

A very efficient log washing at the wood yard diminishes the entry of soil to the system substantially.

Beside that, there was implemented a separate, open washing stage between O and Z, where the pulp is treated with sulfuric acid at a pH around 2,5.
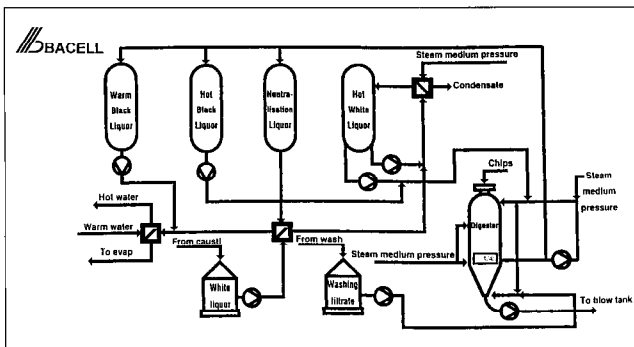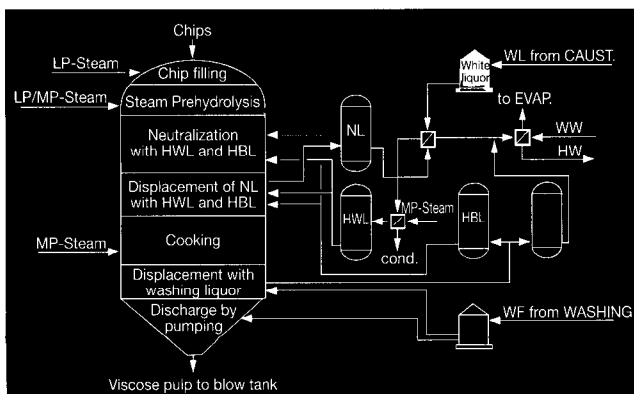
The ozone stage was also designed as a ZQ-stage, where the ozone reaction is followed by a chelation with EDTA.

- The final bleaching of BACELL corresponds to the concept realized in LENZING, as the basic engineering was done by LENZING TECHNIK.
- Ozone is very effective in reducing the lignin content to very low levels, which is an essential issue for dissolving pulps.
- The ozone bleaching is carried out as a Medium Consistency stage with two MC-mixers. Oxygen as basic material for ozone is produced on site in a PSA-plant, the off gas of the ozone bleaching is used for the delignification and white liquor oxidation.
- The final stage is an alkaline peroxide stage to adjust the brightness target. The reaction conditions of this peroxide stage can be rather moderate because of the high incoming brightness and the excellent activation by the preceding ozone stage. For that reason the viscosity loss can be kept rather low.

2.3 Drying Machine

The existing drying machine was modernized, using many parts of the existing drying section.

The wet end of the machine was completely rebuilt. The TETRAFORMER is a new development, combining the sheet formation properties of a Fourdrinier machine with the low energy consumption of a double wire press. After some trials on the Bolton pilot plant of BELOIT, this prototype was installed at BACELL for the first time.

3. Operations

The mill started operations in January 1996 and in March the first lot of prime grade dissolving pulp left the production line. Already after a short period, the quality for viscose fiber grade pulp could be stabilized on a high level and the feed back of our customers is a very positive one.

In the meantime there were made several tests to establish special grades for filaments, acetate and others in tight cooperation with clients.

The actual capacity of the mill was at the end of the year 1996 slightly below the design capacity, mainly due to restrictions in the recovery area.

- Cooking: the cooking plant was operating very reliably from the beginning. To our big surprise we reached very soon a kappa number of 5, although with low viscosity but without problems in the digester circulation as well as during washing and bleaching. Anyway to my knowledge, this should be one of the lowest kappa numbers ever reached from an industrial scale kraft digester.

  But there are still two major limitations from the cooking process. On the one hand, the consumption of effective alkali is around 15% higher than predicted. This means an important restriction in production capacity, which was lifted this month by increasing caustisizing capacity. On the other hand the viscosity on a given kappa number is slightly lower than expected from the lab tests. Lower viscosity losses in the bleaching process and a higher ageing resistance of our pulp can compensate this effect but anyway, we are still working on an improvement. The big question mark, the vapour phase prehydrolysis, proved to work very well, which allows us to adjust the pentosane content in a wide range.

- Fiber Line: The fiber line is keeping its promises concerning quality and chemical consumption. Especially the oxygen delignification is reaching the remarkable lignin reduction of more than 72%. It can also to a big extent smoothen the variation in kappa number and viscosity.

  The ozone stage is also fulfilling the expectations in terms of delignification, brightening and activation for the following peroxide bleaching. Only the viscosity control in a wide range via ozone dosage is not working as well as predicted. In this point we will make some further improvements together with the supplier by increasing gas mixing efficiency.

  Concerning transition metal removal the mill is exceeding the design values, so we can easily obtain a low metal content.

  The final peroxide bleaching is gaining more than 10% of brightness due to the previous activation.

  After some problems in the start up phase and some minor modifications, the drying machine with the tetraformer is now operating very well and has proved to be a good solution for a low energy dewatering system.

  We have also installed a rewinder, so we are selling our product in form of bales as well as reels.

4. Pulp Quality

In the first year of operation we could very well establish a good quality pulp for the viscose fiber market. Besides that Bacell is also developing dissolving pulp qualities for other branches like acetate, filament, nitrocellulose and others together with clients. The normal range of our quality is:

brightness:	88 - 90,5 % ISO
viscosity:	400 - 550 ml/g
alpha cellulose content:	> 94,5
R 18:	> 96,5%
R 10:	> 92,0%
kappa number:	< 0,5
extractives(DCM):	< 0,1%
ash:	< 0,12% including sodium oxide
metals:	Fe < 10ppm
	Ca < 100 ppm
	SiO_2 < 80pPM
	Mn: < 0,5ppm
	Mg: < 80ppm
	Cu: < 1,5ppm
	Na: < 300ppm
	dirt: < 5mm^2/m^2

But we are always open to discuss variations of specific parameters within the bounds of our possibilities:

- It is evident that the **brightness** ceiling is lower for a TCF pulp than for a chlorine dioxide bleached one. But investigations could prove that the preservation of brightness during the whole viscose process is the highest for Bacell pulp compared to all others tested. Probably this is due to the very low content of carbonylic and reducing end groups in our pulp.

- In comparison with our competitors the **viscosity** of our pulp is on a lower level. Higher resistance in the ageing stage is compensating this property. Anyway we are working on an improvement of our cooking process to reach a higher viscosity range.

- As already mentioned above, the vapour phase prehydrolysis is very efficient, so an increase in alpha cellulose content up to 96% or even more is possible and necessary for acetate grades. R 18 and R 10 are following the same correlation.
- The low lignin content, expressed by the kappa number is a benefit of the ozone bleaching technology with its good delignifying properties.
- The extractive content of our pulp is extremely low and so far we have not observed in our production line any scaling or deposits caused by pitch. This low level of natural resin makes the addition of a surfactant, for example Berol Visco 388, very efficient in terms of reactivity in the viscose production.
- The acid as well as the chelating treatment of the pulp is reducing the metal content substantially, therefore lower values can be achieved also.

The molecular weight distribution of the Solucell is exceptionally narrow with a low portion on short chains, which should give a remarkable advantage concerning fiber quality (Fig. 4).
The fiber length distribution is typical for an eucalyptus pulp and very similar to acid sulfite eucalyptus cellulose (Fig. 5).

Molecular Weight Distribution

SPK = Softwood-Prehydrolysis Kraft Pulp
BAS = Beech-Acid Sulfite Pulp
HPK$_c$ = Hardwood-Prehydrolysis Kraft Pulp, Cold Caustic Extracted.
EAS = Eucalyptus-Acid Sulfite Pulp

Fig. 4

Fibre Length Distribution
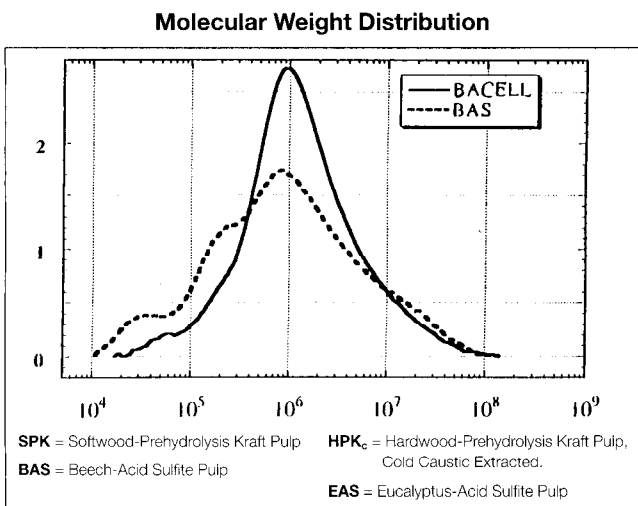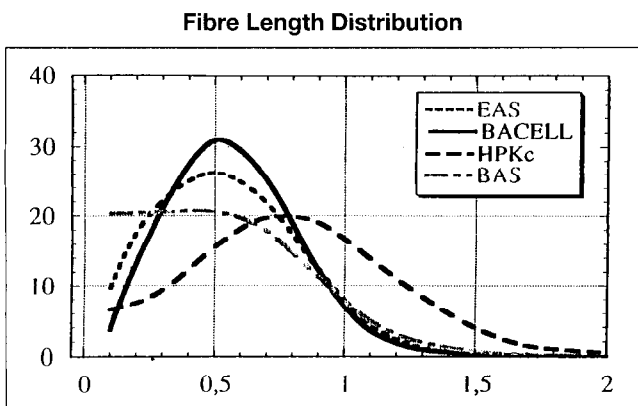
Fig. 5

5. Experience with SOLUCELL in the rayon process

5.1 Laboratory investigations

At first the Bacell pulp was tested on the so called „Treiber"-Viscose Plant at Lenzing by Dr. H. Sixta to receive basic data for ageing behavior and viscose quality.

- Ageing Properties: A comparison with a market pulp of a similar quality (hardwood prehydrolysis kraft with cold caustic extraction = HPKC) showed interesting results. Without using a catalyst the kinetics of both pulps were nearly the same (Fig. 6).
To our surprise the addition of a catalyst (25ppm Mn as KMnO$_4$) changed this relation. Bacell's Solucell proved to be more resistant to catalyzed depolymerization compared to other marked pulps. This behaviour is even more pronounced at higher temperatures (Fig. 7a and 7b).
- Viscose Quality: Our pulp gave a good viscose with low turbidity. But there was a very significant further improvement by the addition of surface active chemicals, probably due to the low content of extractives. By dosing 0,07% of Berol Visco 388 the particle concentration could be reduced by 80% (Fig. 8).

Ageing Properties without catalyst

Fig. 6

Ageing Properties without catalyst at different temperatures

Fig. 7a

Fig. 7b

Test Viscose Quality

Particle Conc. [ppm]

Treiber: No Berol
Treiber: 0,07% Berol
Pilot Plant: 0,07% Berol

Particle Diameter [μ m]

Fig. 8

Brightness Viscose Fibers

Tappi-Brightness [%]

BACELL   SPK   BAS
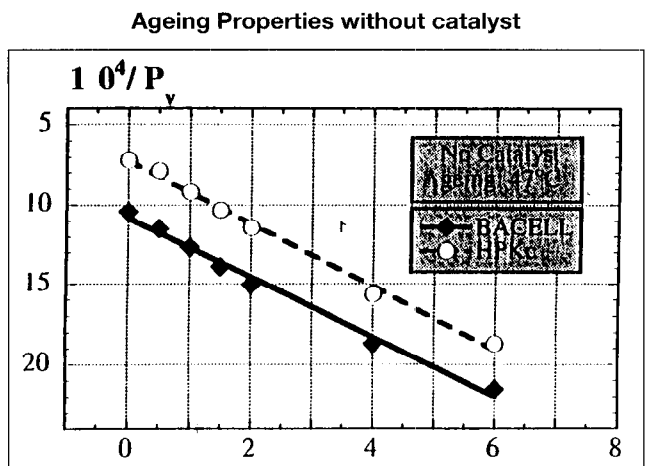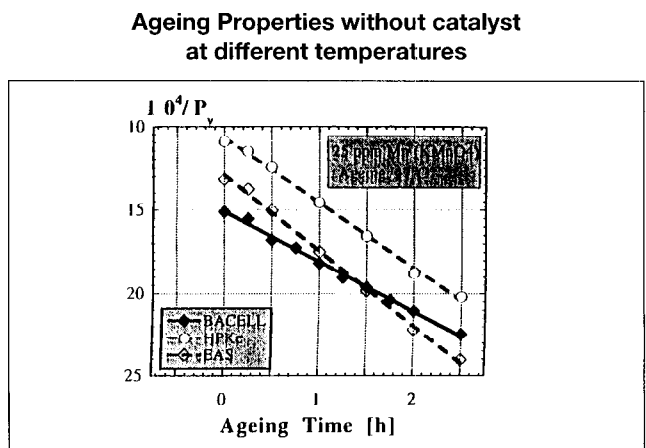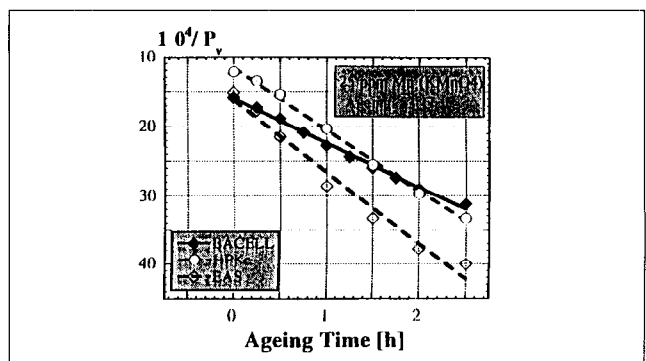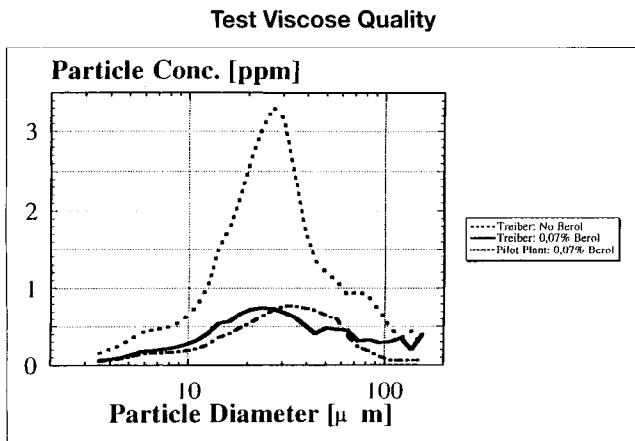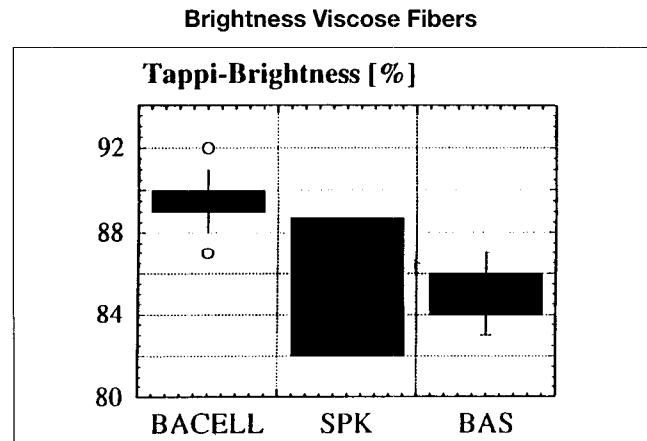
Fig. 10

5.2 Pilot Plant Tests

* Viscose Quality: Viscose of Bacell pulp showed outstanding filterability properties before and after filtration. Also the particle concentration was the lowest in comparison with other commercial pulp samples (Fig. 9). As a further advantage the content of small, non filterable particles is extremely low; the maximum is shifted towards higher particle diameters.

* Fiber Properties: The strength properties of these fibers were comparable to those made from other market prehydrolysis kraft pulps but clearly better than of fibers produced from sulfite pulps.
Remarkabie is the preservation of the brightness over the whole production process, which was the highest for Bacell pulp in comparison to all other pulps investigated (Fig. 10).

Test Viscose Quality

Particle Conc. [ppm]

Particle Diameter [μm]
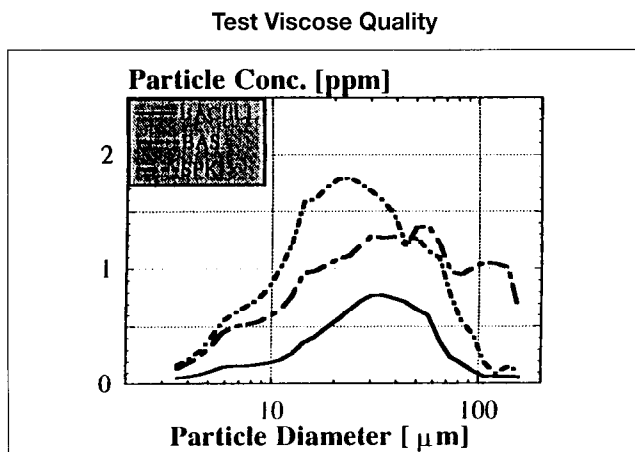
Fig. 9

6. Summary

* BACELL came on stream with a new dissolving pulp mill during 1996 with a prehydrolysis kraft pulp made from eucalyptus, which is already well established for the viscose fiber production.

* The introduction of most modern technologies in cooking and bleaching was successful. BACELL is producing the first and only prehydrolysis kraft dissolving pulp with a TCF-bleaching sequence.

* The results obtained in the first year of operation could, with some small exceptions, prove the design values.

* The quality of the pulp is an excellent one with low amounts of impurities like hemicellulose or ash, good reactivity in the rayon process, low metal concentration, low kappa number and a good and stable brightness.

* Both, the fiber length and the molecular weight distribution are narrow, which results in an advantage for viscose fiber properties.

* Viscose and viscose fibers made out of BACELL pulp in Lenzing's laboratory and pilot plant showed exzellent quality data.

* The feed back from our clients which are using our pulp already some months is very positive.

NEW MODIFIED VISCOSE FIBERS WITH IMPROVED ABSORBENCY

Josef Schmidtbauer, Lenzing AG, Austria

ABSTRACT

The nonwovens industry is permanently interested in new fiber materials with improved hydrophylic properties. Lenzing has developed two different modifications of viscose fibres with increased absorbency.

In cooperation with the Institute of Chemical Fibres in Lodz a modified viscose fiber was developed by incorporation of cellulose carbamate. Influences of the amount and the D.S. (degree of substitution) of carbamate, of spinbath composition, viscose ripening index and viscose additives on absorbeticy, crimp and the mechanical properties of the fibres are discussed.

Another method to enhance the absorbency of viscose fibres is the incorporation of sodium alginate.

In order to achieve a high yield of alginate incorporation, the spinning process has to be modified. The fibers are characterized by a high water retention.

The performance profiles of carbamate and alginate modified viscose fibres are quite different and suggest an application for specific end uses.

1. Introduction

High absorbency is an important, highly-demanded property in many nonwoven end-uses such as diapers, tampons, sanitary napkins, medical sponges, baby wipes, wiping cloths and the like. Each of these applications require a high capacity for absorbing and retaining water and other aqueous fluids, particularly body fluids. The nonwoven industry is therefore permanently interested in new biodegradable fibre materials with improved hydrophilic properties and absorbency characteristics. Different ways of improving the absorbency of viscose fibres have been proposed:

Ways to improved absorbency

- **Chemical** modifications of the viscose fibre by
  crosslinking,
  polymer grafting,
  derivatisation,
  incorporation.

- **Physico-mechanical** modifications by variation of
  titer,
  cross section,
  structure (surface, cristallinity, orientation).

Fig. 1

In this paper I would like to concentrate on chemical modifications of viscose fibres by incorporation of organic polymers. During the late seventies Lenzing developed Viscosorb, a viscose fibre with improved absorbency by incorporation of carboxymethylcellulose. Different ways were taken into consideration: the production of a modified viscose from carboxymethylated pulp, substitution reactions for the introduction of the carboxymethyl group into the cellulose and the incorporation of alkalisoluble carboxymethylcellulose. Water retention values of about 140% could be obtained.

With our know-how about the incorporation of carboxymethylcellulose as a background, we have recently developed techniques for the incorporation of two further substances: cellulose carbamate and sodium alginate.

The incorporation of cellulose carbamate is outstanding in one special aspect: after alkaline aftertreatment the fibre can be partially, perhaps even completely converted to pure cellulose again with the brilliant absorbency characteristics of the cellulo-se carbamate fibre still remaining. So we may classif this invention not only as one of incorporation of a cellulose derivative but also as a possible route of structural modification without permanent chemical modification.

Alginate is a biopolymer with interesting properties: apart from its exzellent absorbency characteristics it is physiologically safe and used as a food additive and for wound dressings. By incorporation of alginate rayon fibres with improved absorbency could be obtained.

1.1. Theoretical Background

A correlation between absorbency (water retention) and structural characteristics of cellulosic fibres has been reported by Krässig:

$$\text{Retention} \propto \frac{1}{(\text{crystallinity}) \cdot (\text{orientation})^2}$$

Based on this theory a reduction of the share of crystalline regions and a lower degree of orientation of the cellulose molecules should be favourable to achieve improved absorbency.

The mechanism of increased absorbency by means of chemical modification is, however, based on the introduction of additional hydrophilic groups, which enhance the interaction of water with the hydroxyl-group of the cellulose molecule.

1.2. Test methods

When we are talking about absorbency characteristics, I would like to refer to the following standard test methods as applied in our laboratory:

- the water retention value (WRV) characterizes the fibre's ability to retain water if defined centrifugal forces are applied. We can assume, that under the testing conditions of DIN 53814 the major part of the liquid absorbed between the fibres is removed.

- the water holding capacity or imbibition value (WIV) measures the amount of liquid which is absorbed between the fibres under free swell conditions according to the European Pharmacopoeia.

2. Incorporation of Cellulose Carbamate

Cellulose carbamate is produced by reaction of activated cellulose with urea or derivatives thereof. It is soluble in caustic soda. Cellulose carbamate is, however, not stable under these conditions. For this reason, temperatures must be kept low. Stability can be influenced by the D.S. of the substance and by the position of the introduced functionallty.

Cellulose Carbamate

Fig. 2
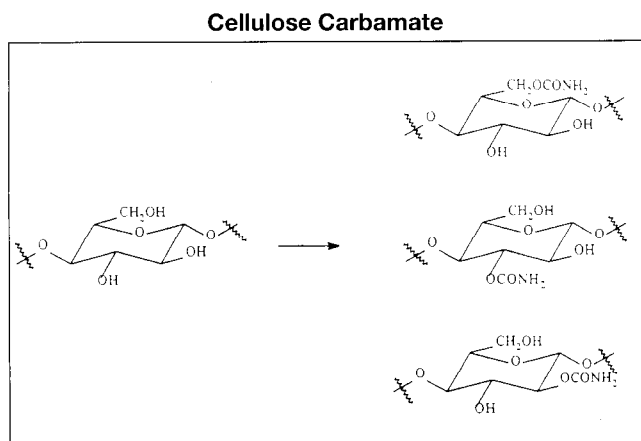
A cellulose carbamate incorporated viscose fibre was developed at the Institute of Chemical Fibres in Lodz/Poland. The procedure is described in various publications (Polish patents PL-AL 293243 and PL 168 080). A solution of the said cellulose carbamate in caustic soda was mixed with viscose and spun into an acid spin bath.

According to the method developed at the Institute, the fibres should preferably consist of 66 to 90 % cellulose carbamate (cellulose taken as 100%). The concentration of the cellulose carbamate solution should be lower than 15%, preferably between 6 and 10%. The N-content of the cellulose carbamate solution, which is a measure of the D.S. of the cellulose carbamate, ought to be in the range of 0,5 to 4,5 %. The fibres are reported to show higher water retention and higher tenacity.

Starting from this basic technology, we investigated into the incorporation of carbamate in the lower range of 2 to 9% and found surprising effects. The incorporation leads to a maximum in crimp and water imbibition at a carbamate content of about 5%.

A reduction of the concentration of the cellulose carbamate solution improves the spinning process and prevents the clogging of the spinnerettes and the occurance of spinning faults.

The incorporation of cellulose carbamate into the viscose fibres leads to a more or less linear increase in water retention with carbamate concentration. A value of more than 105% can be reached by incorporation of 30% cellulose carbamate. Viscose fibres that have not been modified show values of around 80%. Crimp and water imbibition reach a maximum around 5% cellulose carbamate in the fibre, which is therefore the preferred cellulose carbamate concentration for the production of an improved absorbency viscose fibre.

The relationship between crimp and water imbibition

Fig. 3
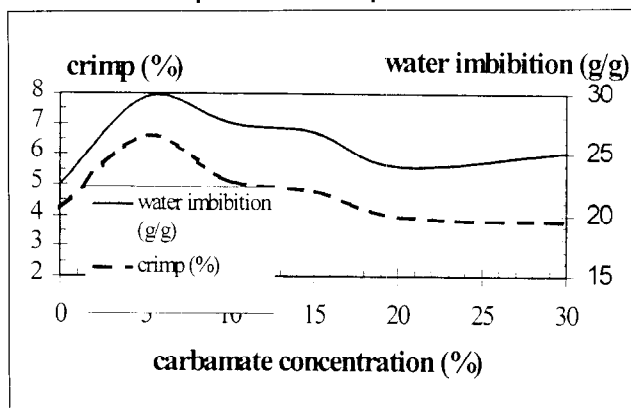
The influence of cellulose carbamate concentration (D.S. 2,9% N) on absorbency

Fig. 4
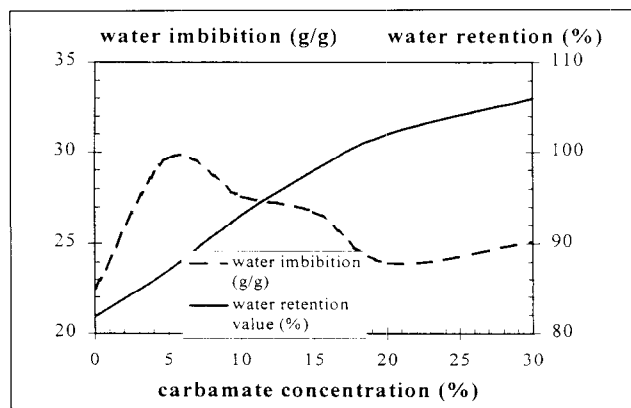
Tenacitv and elongation decrease with increasing concentration of celllilose carbamate. They fall, however, tolerably if concentrations in the range of 5% are considered.

The influence of cellulose carbamate concentration (D.S. 2,9% N) on mechanical fibre properties

Fig. 5
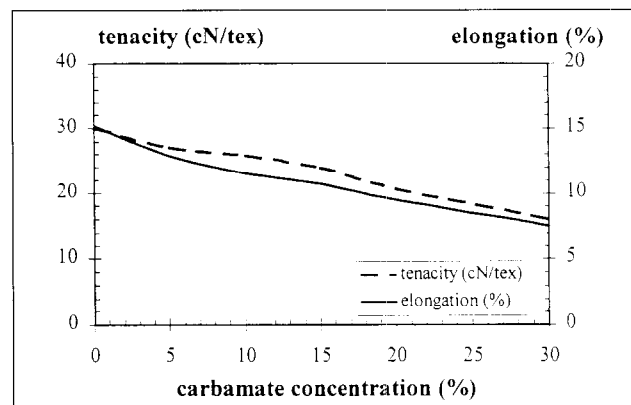
Up to now we have only discussed the influence of cellulose carbamate concentration. The D.S. of the cellulose carbamate was kept constant at a level of 2,9%.
If we use higher substituted cellulose carbamate, the following points will be observed:
The higher the D.S. (2,9%, 3,6%; 4,7%; 5,9% N), the higher the stability of a solution in caustic soda. The viscosity of the solution goes down. As for water imbibition, a rise in the D.S. does

not improve it. Nevertheless the maximum of the curves shows a trend towards lower concentration values. Lower crimp is observed.

Influence of D.S. on the water imbibition – concentration – curves
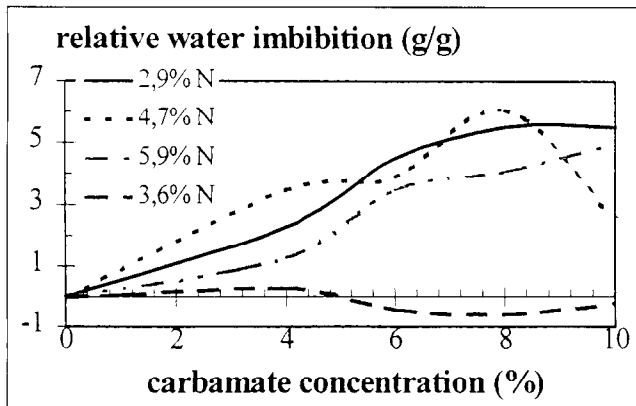
Fig. 6

Beside the D.S. of the cellulose carbamate, the position of the introduced functionallty plays an important role. If the carbamate-groups are evenly distributed in position C-(2), C-(3) and C-(6) of the glucose units, the solubility in caustic soda and the stability of this solution improves and viscosity is lower. However, the incorporation of this type of cellulose carbamate leads to lower crimp, accompanied by a drop in water imbibition. If we attribute crimp to inhomogenities in the coagulation of mixtures of viscose and cellulose carbamate, we are not surprised about these results. As a higher D.S. or a more even distribution of functionalities both improve the solubility of the cellulose carbamate in caustic soda, better homogenity can be achieved, which leads to lower crimp and lower water imbibition.

A variation of the spin bath, i.e. a change of sulphuric acid, sodium sulphate or zinc sulphate concentrations, does not lead to higher crimp and improved water imbibition. In most cases we can say that the higher the water imbibition, the lower the water retention value and vice versa.

Another important parameter in the incorporation of cellulose carbamate into viscose fibres is the viscose ripening Index. A lower index influences crimp and water imbibition positively.

Influence of viscose ripening index on absorbency and crimp
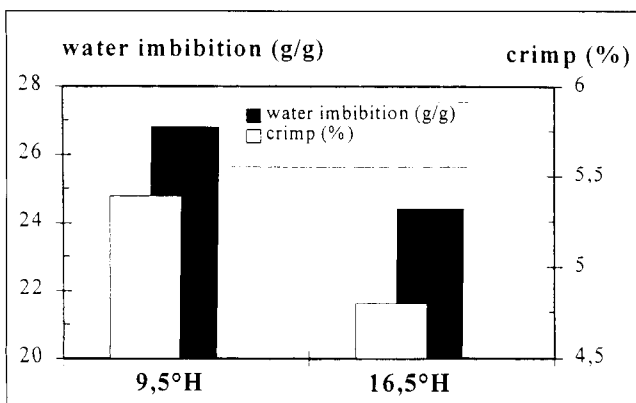
Fig. 7

The influence of viscose additives on absorbency, crimp and mechanical properties was tested by addition of 0,5% Berol 388,1 % Imbentin E 120/200 and 1% Symperonic 13/10. Only Berol 388 shows significant improvements of crimp and water imbibition as well as mechanical fibre properties.

In the following table the presented results are compiled.

Results of the incorporation of cellulose carbamate

parameter	absorbency	crimp	mechanichal properties
dosage of carbamate on cellulose	maximum of WIV at 5%; linear growth of WRV with concentration	maximum of crimp at 5%	slight decrease of tenacity and elongation at 5%
DS of carbamate	higher D.S. does not improve WIV; no influence on WRV	lower crimp at higher D.S.	no influence on tenacity and elongation
distribution of carbamate groups	even distribution reduces WIV; no influence on WRV	even distribution reduces crimp	even distribution improves spinning and mechanical properties
spinbath composition	little effect	little effect	
lower viscose ripening index	improved WIV and WRV	higher crimp	lower tenacity and elongation
viscose additives (Berol 388)	improved WIV no influence on WRV	higher crimp	improved mechanical fiber properties

Table 1

The probably most interesting feature of the described fibres is the possibility, of fully regenerating them to pure cellulose. Cellulose carbamate can be partially or fullv decomposed during regeneration or aftertreatment. It is surprising, that even after partial decomposition of the incorporated cellulose carbamate the absorbency characteristics do not change significantly. Under the aftertreatment conditions applied in our trials about 70% of the carbamate decomposed without any negative effect on absorbency properties.

Water imbibition and decomposition of cellulose carbamate
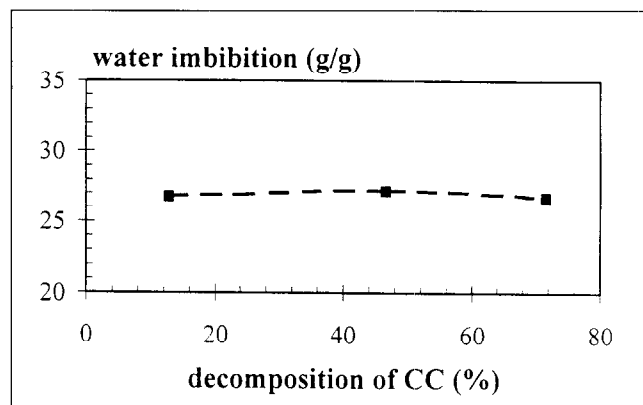
Fig. 8

3. Incorporation of Sodium Alginate

Alginate is a linear copolymer of (1→4)-linked β-D-mannuronate and α-L-guluronate with excellent absorbency. It is traditionally known as a powdery gelling and thickening agent for the food and pharmaceutical industries. In recent years, fibres have been produced from alginate and used for the production of high tech wound dressings. In that process soluble sodium alginate is spun into a calcium chloride bath under formation of calcium alginate. Sodium/calcium alginate fibres can also be produced, with a higher sodium content being related to better absorbency due to the solubility of sodium alginate in water.

Alginate

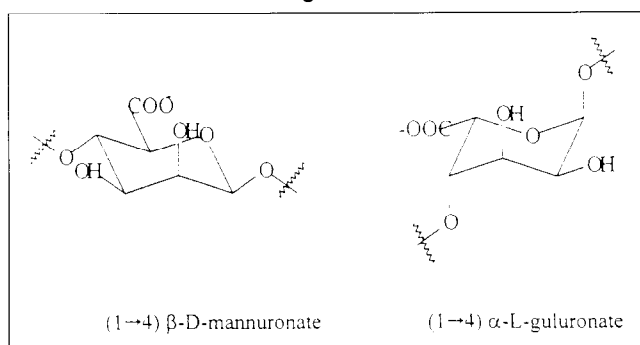(1→4) β-D-mannuronate   (1→4) α-L-guluronate

Fig. 9

We have now developed a process for the incorporation of less than 10% (cellulose tanken as 100%) of this highly absorbent polymer into rayon to remarkably improve its absorbency. Depending on the different steps of secondary treatment alginic acid or a salt thereof is incorporated into the cellulosic fibre. Comparable fibres are well known from a process described in DE-OS 27 50 622, which describes the incorporation of 11 to 66% sodium alginate into rayon.

Our Intention, however, allows the production of alginate incorporated fibres containing, only 3 to 10% of the comparably expensive alginate. Even so, water retention values of as much as 150% can be obtained. Tenacity and elongation of the fibers decrease according to the alginate content but still remain in an acceptable range.

Careful control of temperature and residence time in all process steps performed under acidic and therefore hydrolyzing conditions is necessary, as these steps show a remarkable influence on fibre properties.

Absorbency characteristics of alginate incorporated fibres
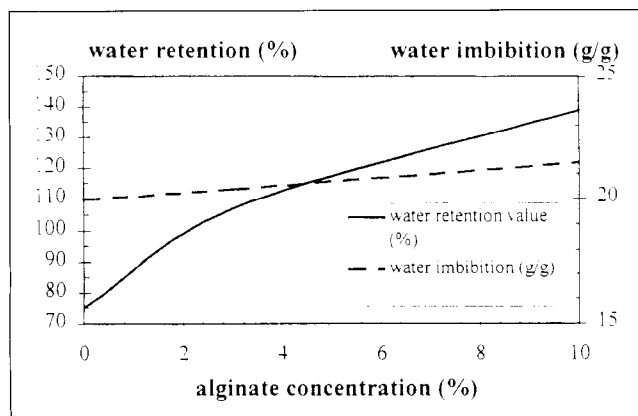
Fig. 10

Mechanical properties of alginate incorporated fibres
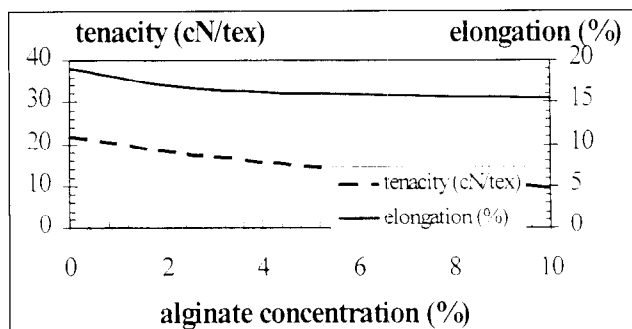
Fig. 11

4. Conclusion

To meet the demands of the nonwoven industry we have followed the route of incorporating cellulose derivatives or biopolymers as a means of improving the absorbency of viscose fibres.

We developed two types of fibres with improved hydrophilic properties but different absorbency characteristics. Viscose fibres modified by incorporation of cellulose carbamate show a maximum of water imbibition at 5% CC while water retention is only slightly improved. The maximum in water imbibition correlates with the maximum in crimp.

The absorbency characteristics of viscose fibres containing 8-10% alginate are quite inverse: while water retention is almost doubled, water imbibition seems to be uneffected.

Now we are going to evaluate possible applications for our absorbent fibres taking into account that different perfomance profiles are required for different nonwoven or textile products. As far as the carbamate fiber is concerned however, one major problem to be solved on the route to commercialization is the limited avallabillty of the raw material cellulose carbamate. The fact, that by special aftertreatment of the fibre the cellulose carbamate may be decomposed to form pure cellulose, opens a completely new way of improving absorbency characteristics significantly without permanent chemical modification.

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- PL-A1 293243

RECENT RESULTS WITH LYOCELL FIBRES IN TEXTILES

Tom Burrow, Courtaulds Tencel, UK

Tencel® is Courtaulds brand name for their lyocelt fibre in fabrics used in apparel and other fashion markets. Since the introduction of the fibre to the market, the Tencel business has grown rapidly. Tencel fabrics have gained a reputation for a soft, silky handle and exceptional drape and fluidity that can be combined with a peachskin finish. These fabric properties have made Tencel a fabric of choice for high class ladies casual wear, soft denims and men's shirts.

Tencel is amongst the strongest and stiffest cellulosic fibres ever produced. It has a high dry tenacity and modulus. It retains much of its strength when it is wet; the wet tenacity is higher than that of cotton.

The desirable properties of Tencel fabrics are developed during fabric finishing. Fibrillation of the fibres at the surface of the fabric is responsible for the peachskin finish, which gives the characteristic surface touch to the fabric. The fibrillation is generated by extensive work on the fabric in the wet state that is possible because of the exzellent fibre properties. The handle of Tencel fabrics, which is due to their drape and fluidity, also develops, as the fabric is finished.

Fibrillated Tencel Fabric
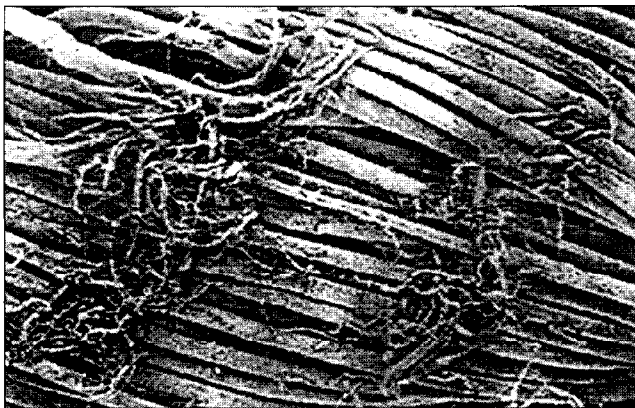
Fig. 1

The main topic I will cover in this paper is why Tencel fabrics can be so soft and fluid and drape so well when the fibre from which they are made is one of the stiffest cellulose fibres ever made. Understanding how the drape and fluidity of Tencel fabrics develop has also helped us to explain other phenomena such as permanent setting, seam behaviour, seam pucker and creep and growth effects. But first some background on Tencel fibre manufacture and properties.

Tencel Fibre

The cellulose in Tencel fibre has a high degree of orientation and crystallinity and a higher molecular weight than viscose. As a result, the fibre strength and modulus of Tencel are very high; the strength is twice that of cotton and similar to many polyesters. The strength is much less affected by wetting than other man made cellulosics; the wet strength of the fibre is higher than that of cotton. But, like other cellulose fibres, Tencel absorbs water

well. When fibres swell, they can cause the dimensions of the fabric to change; the fabric shrinks and increases in thickness. The driving force is the breaking of hydrogen bonds that are reformed at new positions when the fabric is dried. The fibres take up a conformation that corresponds to the relaxed state of the fabric.

Tencel is made from cellulose, as are cotton, cupro and viscose and its variants. The cellulose used is woodpulp from sustainabie managed forests. It is converted to fibre by a process that is, in environmental terms, the best available technology not entailing excessive cost.

TencelProduction Flowchart
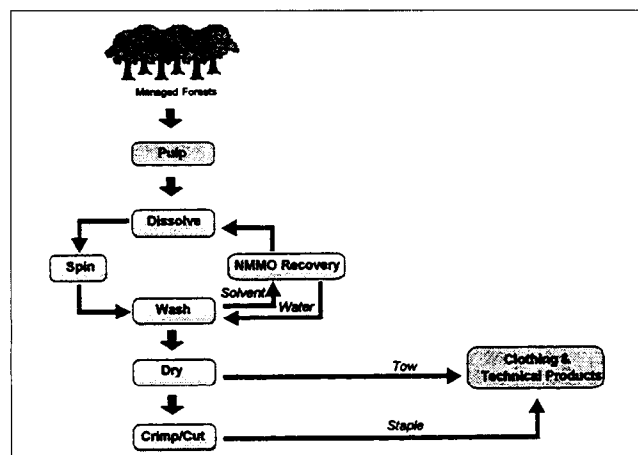
Fig. 2

The woodpulp is dissolved in a solvent (amine oxide) to form a viscous liquid. The liquid is extruded into a water bath through fine jets. As the solvent is washed out the fibre forms, is dried and then collected as a tow. The fibre can then be crimped and cut with fibre finishes applied as appropriate to particular applications.

The amine oxide solvent used is recovered for repeated re-use as part of a highly effizient production process. Waste is minimal and considered harmless. The only significant raw materials used are woodpulp and water.

The first full-scale plant was built at Mobile Alabama and is now fully operational. A second commercial plant is now under construction at Grimsby and due to start production later in 1997. Further plants are being planned.

Tencel Fibre Properties

Tencel is the strongest of the man-made staple fibre cellulosics. Its strength exceeds

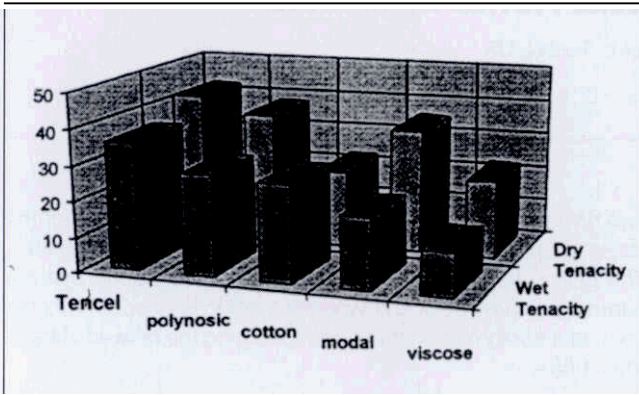## Dry and Wet Fibre Tenacity (cN/tex)

Fig. 3

cotton both wet and dry and car **even** compete with a typical polyester staple.

The high fibre strength of Tencel translates into high tensile strengths in the spun yarn and in finished fabrics. Tencel can easily return a fabric tensile strength 25% greater than for a cotton fabric. The advantage is **even** greater in **tear** strength Figure **4**
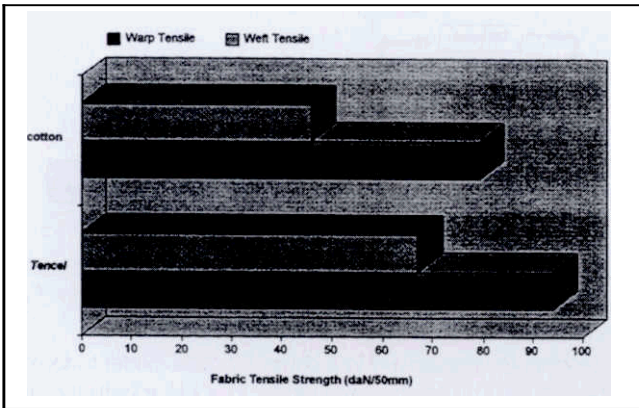
Fabric Tensile Strength

Fig. 4

Tear strengths for Tencei car **be** double that expected from cotton.

Fabric Tear Strength
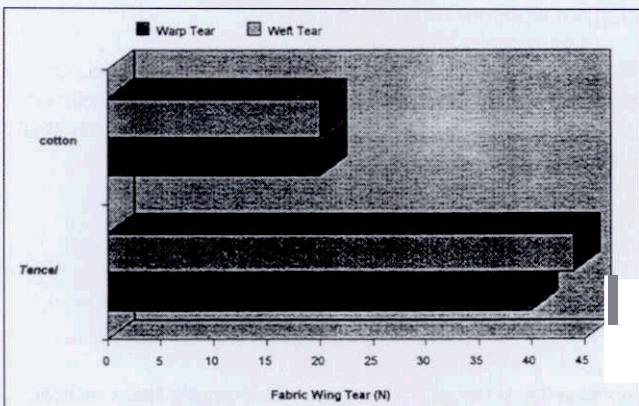
Fig. 5

Basic Concepts

As well as these exzellent physical properties, Tencel fabrics can give exceptional aesthetic properties The origin of the Tencel aesthetic is now much better understood. It relates to the way Tencel behaves when it is wetted and dried and during textile finishing.

The starting point for our work on the behaviour of Tencel fabrics was an understanding of why Tencel, and other cellulosic fabrics, shrink when they are wetted. Several workers in the United Kingdom Including Grosberg, Hearle and Pearce developed this concept many years ago. Professor Kawabata also uses the principle in some of his work.

When a loomstate fabric is wetted, the fibres swell. This **makes** the yarns swell. Tencel fibres increase in diameter by about 35%. Because the fibres in the yarn lie very parallel to each other, the yarn also swells by up to 35%. The swelling of the yarns causes the crimp in the yarns to increase. This reduces the distance between the ends of the yarn As a result, the fabric shrinks when it is wetted.

Loomstate fabric
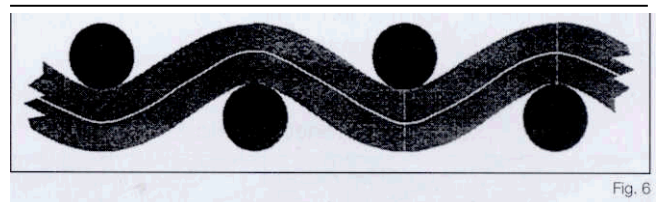
Fig. 6

Water Swollen Fabric
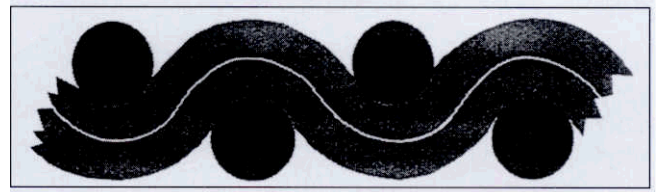
5 . 7

When the fabric is dried again. **the** fibres are **set** into the shapes they had when the fabric was wet. Therefore, the dry fabric shrinks compared to the loomstate fabric.

The Origin of Drape and Fluidity

Tencel fabrics have a drape and fluidity combined with bulk that k unexpected for fabrics of their weight. It is these properties which give Tencel piece goods and garments much of their appeal. Drape and fluidii are developed during dyeing and finishing.

When a Tencel fabric shrinks, the crimp in the yarns increases. The conformation of the yarns stays **the same** as it was in the wet state when the fabric is dried again. As the yarns shrink back to their original size, spaces develop between them at the cross over points.

Dried Fabric
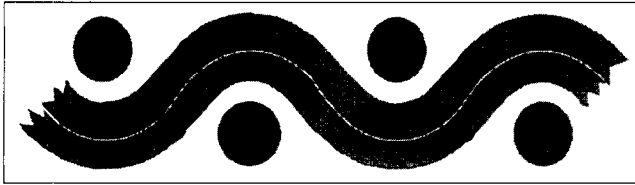
Fig. 8

In the original loomstate fabric, the yarns are in close contact, exerting a normal force on each other which makes the fabric stiff and paper like. The yarns can not move easily as they are constrained by friction from their neighbours. After the fabric has been through the shrinkage process, the yarns can move easily. The angle between the yarns at the crossover points can easily change, the fabric can be sheared, up until the point when all the yarns touch and the fabric locks up. This means that the fabric has enhanced drape; it can easily adapt to double curvatures. It also appears to be fluid; it moves easily for quite small applied forces.

Swelling of the fibres causes the yarn diameter to increase and is the main driver for the development of spaces between the yarns in the fabric. As these spaces develop, the fibres in the yarns rearrange themselves in order to correspond to the new yarn conformation. Because a lot of work is done on the fabric during finishing, there is ample opportunity for these rearrangements to take place. The fibres are subsequently set into the shapes they have corresponding to their new positions. This in turn sets the fabric into its fluid state.

It is important to note that Tencel fibre is always set into the shape it had when it was wet, when it is dried again. This is a fundamental aspect of the behaviour of Tencel fibre, yarn and fabric.

Permanent Creasing of Cellulosic Fabrics

Many cellulosic fabrics suffer from creasing problems when they are incorrectly constructed or finished. In some cases (eg linen fabrics) these effects have been used positively to produce distinctive character.

Our studies of the swelling behaviour of Tencel fibres and fabrics have led to an explanation of why these permanent creasing effects occur in cellulosic fabrics.

When a woven fabric is bent perpendicular to one set of yarns, the yarns which are on the outside of the bend at that point in the fabric are bent around the yarns running parallel to the bend. The radius of curvature of the yarn remains about the same, but the angle of wrap increases. The yarns on the inside of the bend tend to straighten.

Bent fabric
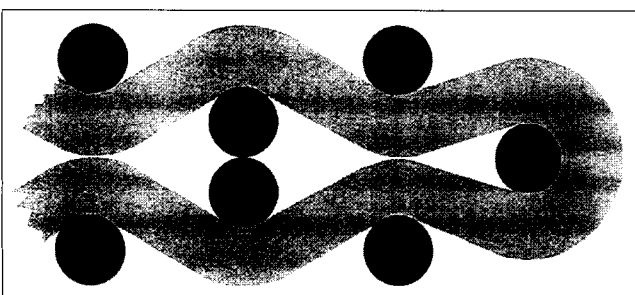
Fig. 9

When a Tencel fabric is wetted, the yarns swell. If the fabric is fairly tightly constructed, then it will jam; the swelling will fill up the spaces between the yarns and the yarns will be in close contact. This is the reason that the fabric becomes stiff when it is wet.

Bent wet fabric
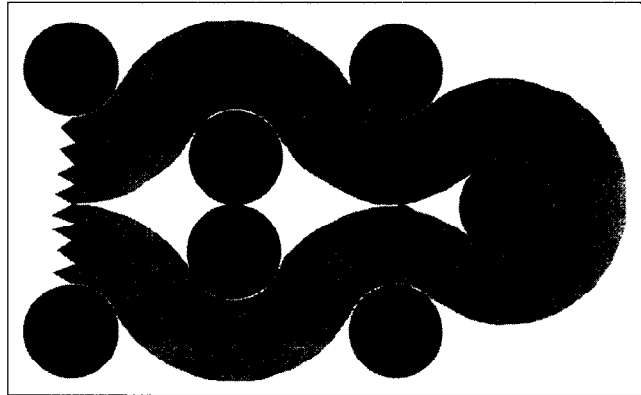
Fig. 10

If the fabric is bent before it is wetted, then when the swelling occurs, the path length of the yarns running across the bend has to increase in order to accommodate the swelling. When this happens, the yarn is tensioned and stretches. If the tension exceeds the frictional force acting on the yarn where it interlaces with the other yarns, then the yarn will move to relieve the tension. The result is that there will be extra yarn around the outside of the bend and less yarn on the adjacent floats on the inside of the bend.

In a fabric that jams when wet, this displaced yarn can never move back.
Straightening the fabric when it is wet cannot move the yarn to its original position.

The yarn is soft, the fabric is jammed and moving the yarn is like trying to push cooked spaghetti through a hole in a colander.
When the fabric has been dried the yarn is set into the shape it had in the bent fabric. Straightening out the fabric will not make the yarn move back to its original position because its shape corresponds to its new position. The result is a permanent crease in the fabric.

The same effect will also occur if a wet fabric is bent. The yarns will be tensioned to the same degree and will move in the fabric to relieve the tension. Once this movement has taken place, the original position of the yarns is irrecoverable.

If the construction of the fabric is such that it will not jam when wetted, then permanent creases are less likely to occur. In the wet state the fabric will not be stiff and the yarns will be able to move over each other. The yarns, which have been displaced due to the bending of the fabric, will be able to return to their original positions if the wet fabric is straightened.

Tencel is different from other fibres because of its high swelling, its high modulus and the smooth surface of the fibres. The high swelling means that the path length increase for the yarns around the outside of the bend is large; the stretch needed in the yarns is high. Because lyocell has a high modulus, the fibre develops a high tension for a relatively small, applied strain. Thus a small increase in the path length may result in a tension which

can exceed the frictional force holding the yarn in position in the jammed fabric. The smooth surface of the fibres means that the frictional force is relatively low. Therefore it is very important to get the construction of the fabric right in order to prevent this type of creasing. It has been shown that changes as small as one pick per centimeter can make the difference between a fabric that creases and one that does not.

Control of Permanent Creasing

Experimental work has demonstrated that changing the construction of a fabric can reduce its tendency to form permanent wet creases. Finite Element Analysis is being used as a means of predicting what changes to construction will have a positive effect.

Caustic treatment of Tencel fabrics can be used to exaggerate the swelling that occurs when the fabric is in the wet state and hence reduce jamming of the fabric.

Growth of Fabrics

Like some other fabrics such as wool, the dimensions of a Tencel fabric can change as the humidity changes. This is because the crimp that is set into the yarns is sensitive to moisture content. As the fibres absorb moisture, they swell and the yarns straighten. The crimp reduces. Beyond a critical point, the yarns have swollen so much that they touch and the fabric then starts to shrink again.

Hygroscopic Expansion of Tencel
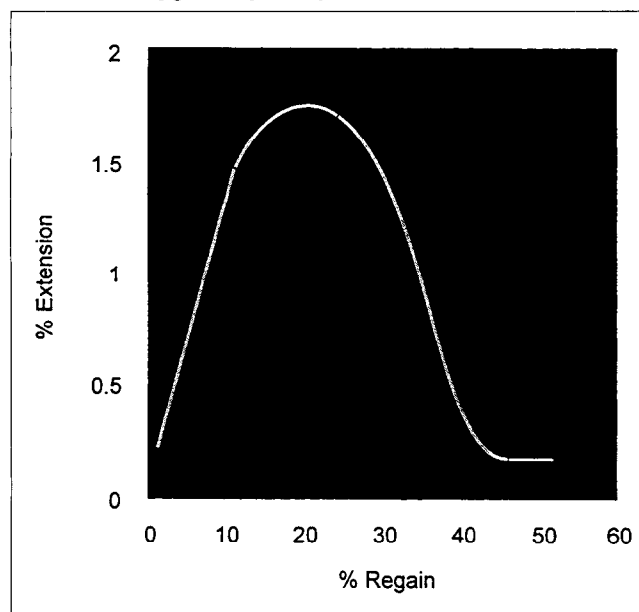
Fig. 11

The result of this is that a Tencel fabric may extend in length as the humidity increases until it reaches a Maximum. It may then shrink again back to its original dimensions. The maximum growth depends on the fabric construction and on the history of the fabric. The changes are small, but may be significant in garments with long panels.

Seam Behaviour

Seam slippage can be a problem in many kinds of fabric. Usually it is weft seams (seams sewn parallel to the weft) that give problems. With Tencel fabrics, it is more likely to be the warp seams that give problems if they occur. In Tencel fabrics, the weft seam strength is, on average, 40% higher than the warp seam strength.

The straight weft is locked in position by the crimp in the warp yarns, which gives strong weft seams. However, the warp can slide over the straight weft. This weakens the warp seams. If garment makers are aware of this difference from other fabrics, they can design the seams to allow for it. If they do so, there should not be a problem.

Seam Pucker

Seam pucker is a highly desirable effect in denim and in many other fabrics used for casual garments. Its appearance adds to the character of the garment and is often one of the key selling points. Our studies of the behaviour of Tencel fabrics have led to an explanation of why seam pucker occurs in cellulosic fabrics.

When a denim garment is produced, the seams, if sewn correctly, are generally smooth and even and free of pucker. When the garment is washed, the seams pucker. This is due to the increase in the thickness of the fabric when it is wetted.

The portion of each stitch that goes down into the fabric has to increase to allow for the increase in thickness of the fabric as it swells. There is therefore less sewing thread left for the part of the stitch that runs along the surface of the fabric. The stitch length is reduced, but the length of the fabric in the stitch remains the same. As a result, the fabric must pucker in the seam. When the garment is dried, the yarns stay in the shape they had in the wet state.

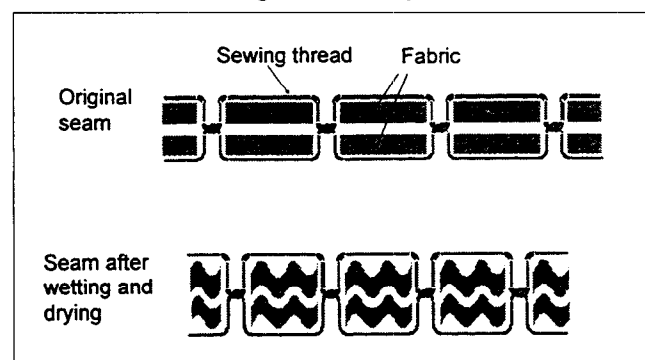### The origins of seam pucker

Fig. 12

Conclusions

The behaviour of Tencel fabrics is strongly influenced by the swelling of the fibre when it is wetted. Forces generated by the swelling change the geometry of the fabric. This leads to the generation of spaces in the fabric that give Tencel its characteristic drape and fluidity. The changes on wetting can also explain other phenomena, such as growth effects and seam pucker.

FORMATION OF LYOCELL-TYPE FIBRES WITH SKIN-CORE STRUCTURE

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Fraunhofer Institute, Germany

As compared with rayon fibres, man-made fibres spun from amine oxide solution still have to be developed further in view of an extended range of textile properties and controlled fibrillability. Different properties of rayon and lyocelltype fibres are due to structural differences including the degree of crystallinity, crystallite dimensions, and the orientation of noncrystalline cellulose chain segments.

On a morphological level, the cross-sectional skin-core structure of rayon fibres was not found with amine oxide fibres produced in the conventional way.

Our aim was to control structure and properties of fibres from amine oxide solution in a wide range by changing the conditions of precipitation. At first we investigated the effect of alcoholic precipitation media in a one-step procedure. Depending on the alcoholic composition, fibres are formed with a reduced supramolecular order and a more distinct pore system. Skin-core structures with a less dense core are formed in some cases. A layered alcohol-water system was employed in a two-step coagulation process resulting in fibres with a less dense nonfibrillating skin and a dense core providing the demanded tensile strength and modulus /1/.

Introduction

One century after the introduction of the viscose process, the amine oxide technology to produce regenerated cellulose fibres of the lyocell-type is making its industrial breakthrough. Advantages of the new technology are that it is environmentally friendly and simple. Disadvantageous are, as compared to the viscose process, the low variability of the fibre property profile and, for certain applications, the fibrillation tendency of the lyocell-type fibres.

In the present paper we report a new possibility /1/ to control the fibre properties and, in particular, to restrain the fibrillability. To this end, we have been investigating structure-property relationships of lyocell-type fibres as a function of the coagulation conditions. Our aim was to produce fibres with a skin-core structure such that the core is formed by molecules well ordered and highly aligned to the fibre axis while the outer region (skin) shows a lower orientation and thus a reduced fibrillation tendency. An example of such an architecture - we have referred to at the 1994 viscose seminar in Stockholm (Fig. 1, ref. 2) - is devised by nature: the cotton fibre. Here, the cellulose fibrils in the interior are highly oriented while in the outer layers the orientation is more or less random. In accord with our approach to develop amine oxide fibres with skin-core structure, the paper has the following paragraphs:

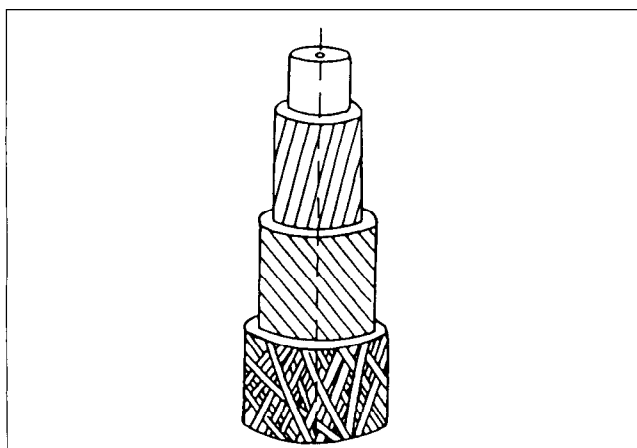### Schematic view of the morphological architecture of cotton

Fig. 1

- Summary of structural peculiarities of amine oxide fibres precipitated in water

- Structure of amine oxide fibres due to alcoholic precipitation baths

- Formation, structure and properties of amine oxide fibres with the desired skin-core architecture.

Structure of Lyocell-Type Fibres Precipitated in Water

Compared to viscose fibres, the structure of the first generation amine oxide fibres exhibits some peculiarities, as reported in detail previously /2 - 4/. Those fibres have a circular cross-section which is markedly different from the lobulated shape of textile viscose fibres (Fig. 2). Also, as revealed by electron microscopy, the morphologies of the two fibre types differ significantly. The amine oxide fibre shows a dense cellulosic network structure complementarily to which there exist small finely distributed voids with dimensions ranging from 10 to 100 nm. The structure is uniform throughout the cross-section, except for a small boundary layer with highly densified material. In contrast to that, textile viscose fibres have a skin-core structure with larger voids (around 25 to 150 nm) in the core region and a densified skin layer which is about 5 to 25 nm thick in our example. With X-ray diffraction it was found that for amine oxide fibres precipitated in water the degree of crystallinity is higher and the crystallites are longer and thinner than for textile viscose fibres (Tab. 1).

Supramolecular Structure of Viscose and Amine Oxide (NMMO) Cellulose Fibres

Sample	Crystallinity %	order parameter $k*100/(nm)^2$	crystallite dim. D_{101}/nm	D_{040}/nm
Enka viscose	27	1.58	5.4	11.4
NMMO type precipitated in water	42	1.75	4.2	14.3

Table 1

Cross sections of cellulose regenerated fibres (TEM)

Fig. 2

Comparison of regenerated fibres orienation factors (total, crystalline, noncrystalline)
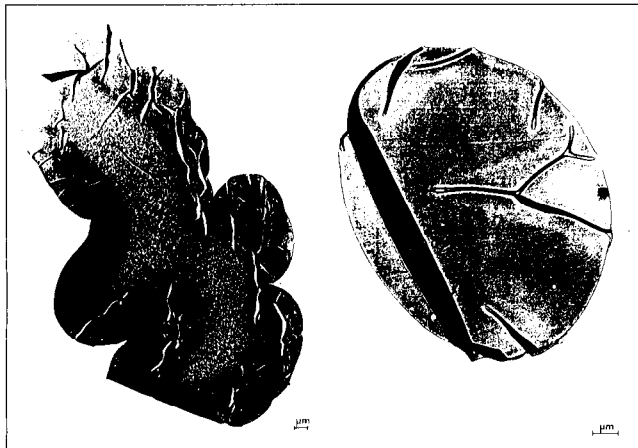

Fig. 4

In contrast to viscose, the crystallite cross-section of amine oxide fibres seems to be less asymmetrical, i.e. more circular. This is certainly connected with the fact that amine oxide fibres do not show in the X-ray fibre pattern (Fig. 3) the „Blättchen-effect" of crystallite orientation typical for viscose fibres. In the schematic drawing of Fig. 4 orientation factors of first genera-tion amine oxide fibres and the differences to those of viscose fibres are shown and a main problem with the development of viscose-like NMMO-fibres becomes clear: while the crystalline orientation f_c of amine oxide fibres and the various viscose fibre types lies at a comparatively high level, significant differen-ces are found for the total chain orientation f_t and the orientation f_t of the amorphous chain segments. Obviously, it is possible to control the textile properties of viscose in a wide range by chang-ing the amorphous orientation, and just this is difficult with amine oxide fibres. The peculiarities of amine oxide fibres of the first generation like high crystallinity, long and thin crystallites and a high amorphous orientation prevent a sufficient lateral fringing of cellulose chains and thus explain the fibrillation effect. The fibrillating structure of amine oxide fibres which is apt to lateral splitting, is demonstrated in the SEM fracture micrograph of Fig. 5. On the whole, due to the structural peculiarities, a fibre with high modulus and low elongation at break is obtained, the property profile of which resembles that of a technical fibre.

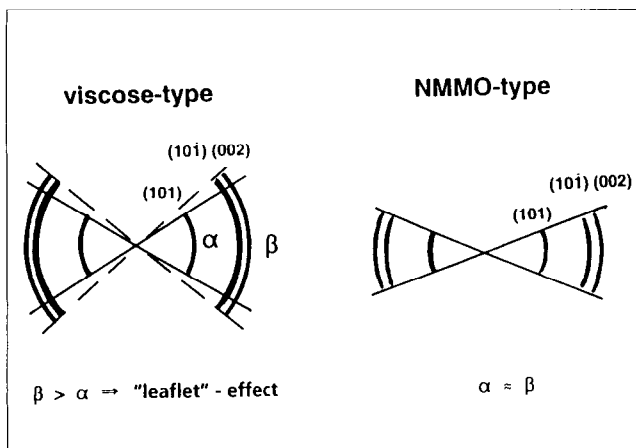## Tendency of fibrillation of a NMMO-type fibre (SEM)

Fig. 5

Structural Peculiarities of Amine Oxide Fibres due to Alcoholic Precipitation Baths

For the development of amine oxide fibres with improved textile properties a decrease in crystallinity and total orientation is attempted in general. One possibility to achieve that is to precipitate the cellulose-NMMO-solution into an alcoholic bath, as pointed out already in Stockholm in 1994 /5/. Meanwhile, further systematic investigations about the influence of alcoholic precipitation media on structure and properties of the resulting fibres have been carried out on a laboratory scale. Using a spinning solution with 9% cellulose in an 87/1 3% NMMO-water system, we tested ethanol, isopropanol, butanol and hexanol as coagulation media. Besides this series of monovalent alcohols with increasing chain length the effect of diols and several alcohol-water mixtures as precipitation media was investigated exemplarily. In the following, the series of mono-valent alcohols is discussed first.

Fig. 6 shows the TEM micrograph of a fibre precipitated in ethanol. it is not very different from the analogous picture of a fibre precipitated in water. The cross-section is oval and an evenly dense morphology with slightly decreasing network density towards the centre is seen. In Fig. 7, a comparison of TEM cross-sectional patterns of fibres precipitated into isopro-panol, isobutanol and hexanol is presented. Already at the magnification chosen here, remarkable differences become

Schematic equatorial X-ray fibre patterns

viscose-type   **NMMO-type**

(101) (002)

(101)

α   β

(101) (002)

(101)

β > α ⇒ "leaflet" - effect   α ≈ β

Fig. 3

obvious. As compared to the precipitation media water and ethanol, isopropanol leads to a distinct skin-core structure of the fibre. In this case the core structure is less compact and contains pores up to the range of microns (Fig. 8). The more densely structured outer zone contains smaller pores of about 15 to 60 nm and is surrounded by a thin layer of further increased density. In the case of isobutanol, the interior of the fibre is characterised by an extensively decomposed structure of compact net work bridges and large pores of diameters up to 300 nm. As compared to isopropanol, the width of the skin zone is reduced. For the fibres precipitated into hexanol, the structure is very loose and uneven. Voids exist with diameters up to the micron scale and partially, the voids resemble radial capillaries of other precipitation structures (viscose, PAN). The fibre boundary is formed by a thin continuous layer.

TEM-micrographs of fibres from NMMO-solution precipitated in ethanol

cross-section   outer region   centre
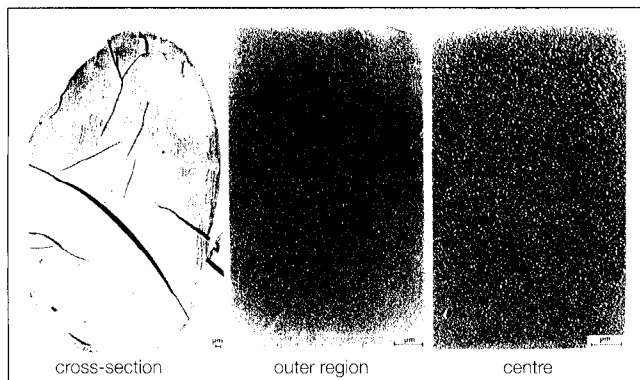
Fig. 6

Micrographs of cross-sections of NMMO-type fibres precipitated in different alcoholic media

isopropanol   isobutanol   hexanol
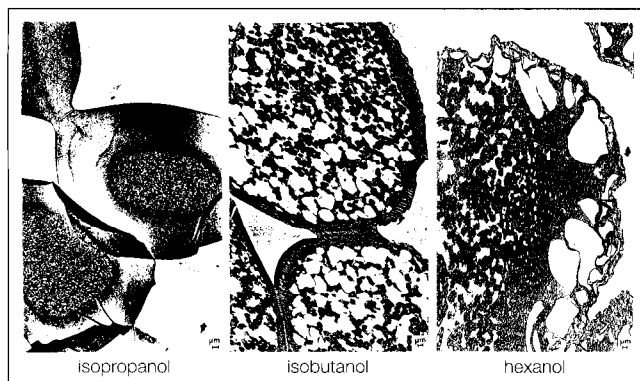
Fig. 7

Skin-core-structure of a NMMO-type fibre precipitated in isopropanol

cross-section   skin   core
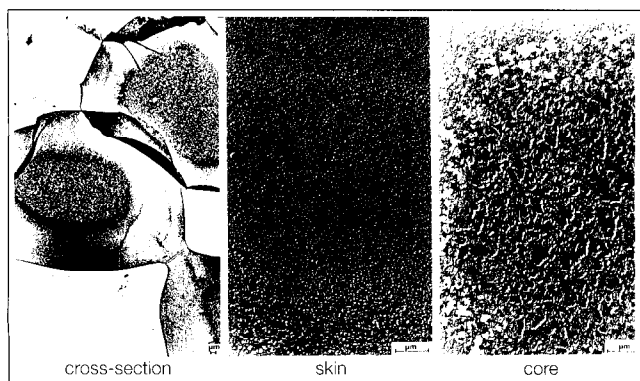
Fig. 8

The comparison of X-ray flat film photographs for the fibres of the above series as presented in Fig. 9, shows that with increasing chain length of the higher alcohols the order and orientation of the fibres decrease. The preliminary end point in this series is given by the fibre precipitated into hexanol having a crystallinity of 25% and so low an orientation that it cannot be evaluated quantitatively. The effects of monovalent alcohols as precipitation media on the structure and properties of the resulting fibres are summarised in Tab. 2.

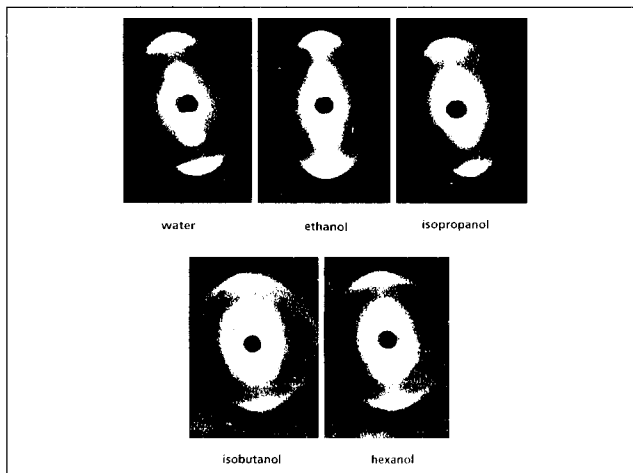## X-ray fibre diagrams of NMMO-type fibres precipated in different alcoholic media

water   ethanol   isopropanol

isobutanol   hexanol

Fig. 9

Influence of alcoholic precipitation media as compared with water

precipitation medium	morphology	orientation/ supramol. order	modulus	fibrillation
water	homogeneous			
ethanol	homogeneous			
isopropanol	skin-core	decreasing	decreasing	decreasing
isobutanol	skin-core			
hexanol	demixing			

Table 2

From these results we conclude that a compact structure, as a rule, has a higher orientation and higher crystalline order than the looser network structure which even decays in the extreme case. Furthermore, it must be said that the skin-core structures obtained so far, namely, with a more compact outer region and a less oriented core, do not conform with the structure aimed at. Rather, it is more like the negative image of the desired arrangement. However, in the way described above, a variation of the fibre property profile towards an improved elongation at break and a decreased fibrillability has been reached.

For the sake of completeness we have to add that the use of alcane dioles as precipitation medium increases the (negative) skin-core contrast while mixtures of dioles and water lead to a decrease of the effects, but without a change or reversal in the general differences.

Skin-Core Structures of Amine Oxide Fibres due to Two-Stage Precipitation

From our investigations about the precipitation into single phase alcoholic baths we have learned that such a coagulation process is in general softer than with water and produces a less ordered and more demixed structure. In order to limit this desired effect to the surface layer of the fibre, we have been

studying the precipitation of cellulose-amine oxide fibres using a two-stage arrangement: the first bath with an alcoholic medium and a second one with water (Fig. 10). For alcohols not mixing with water (e.g. hexanol, heptanol) the two media can be arranged (in the most simple case) as two layers, one above the other. The fibre, spun from above, passes through the air gap and enters at first the alcoholic bath. Here, the coagulation takes place in the skin region with structures typical for alcoholic precipitation: a coarsely demixed cellulose-void system with a dense surrounding layer and with low orientation and order of the cellulose molecules in the whole skin region. In that way the fibre jet is stabilised, i.e. the molecules in the interior of the fibre stay highly aligned. After entering the second, aqueous layer the 'hard' precipitation in the interior of the fibre follows. During this process, the high orientation of the cellulose molecules is retained to a large extend and the well known finely dispersed cellulose network with high orientation and high degree of supramolecular order is formed. In Fig. 11a TEM micrograph of such a characteristic structure, obtained by precipitating in hexanol and water is shown. "One sees the skin region with large, inhomogeneous cavities, the size of which ranges from 200 nm to 2 microns. The interior is finely disperse, the typical cellulose-void structure of a precipitation in water is realised. Keeping in mind the results for the precipitation into the single phase precipitation baths, the conclusion seems to be justified that the electron microscopical structure of the skin corresponds to a supramolecularly less ordered structure while that of the core is related to a more ordered and oriented one. This is fully corroborated by the property combination obtained, as shown in Tab. 3.

Influence of alcoholic precipitation media as compared with water

precipitation mode	modulus [cN/tex]	strength [cN/tex]	elongation [%]	fibrillation stability [s]
one stage water	1290	22	13	8
one stage hexanol	330	5	8	5400
two stage hexanol/water	785	13	12	>8000

Table 3

In particular it becomes clear that for the example of a two-bath precipitation chosen here, the fibrillation tendency (expressed in the Tab. 3 as the time the fibre, loaded with 80% of its wet breaking force, can withstand a damaging water stream) is largely reduced. The mechanical main properties (modulus, strength), on the other hand, are less reduced than for the pure alcoholic precipitation.

Within a certain range, the structures of skin and core can be changed by varying the composition of the precipitation baths. The thickness of the skin is determined by the time the fibre jet stays in the first bath which is, in turn, influenced by the spinning speed and the path length through the first bath.

Conclusions

Summarising it can be concluded that the two-bath precipitation presented here is one basic possibility to produce the desired skin-core structures in lyocell-type fibres allowing in that way to control the property profile and, particularly, the fibrillability of textile fibres in a broad range.

Formation of skin-core-fibres by two-stage precipitation of cellulose-NMMO-solution
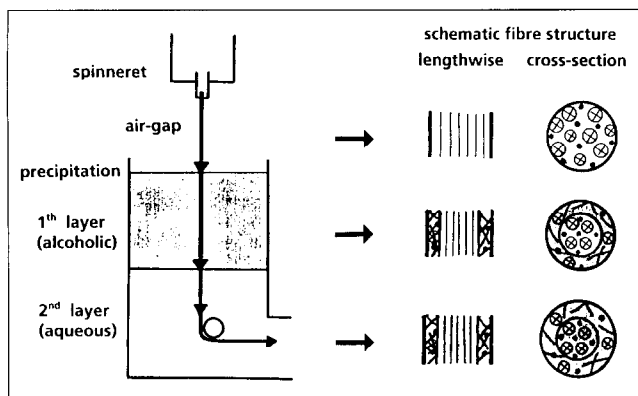
Fig. 10

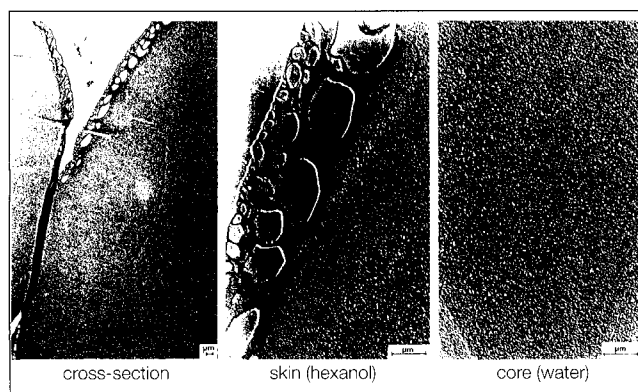### Skin-core-structure of a amine oxide-type fibre by a two-stage hexanol-water precipitation

cross-section   skin (hexanol)   core (water)

Fig. 11

References

1 Patent DE 44 46 491

2 H.-P. Fink, J. Ganster, J. Fraatz, M. Nywlt: „Relation between structure and properties of cellulosic man-made fibres" Akzo-Nobel Viscose Chemistry Seminar, Stockholm, May 30 - June 3, 1994

3 Lenz, J. Schurz, D. Eichinger: Lenzinger Berichte 74 (1994) 19

4 H. Coulsey, S. Smith: "Formation and structure of a new cellulosic fibre", 34th Int. Man-made Fibres Congress, Dornbirn, Austria, Sept. 20 - 22, 1995

5 P. Weigel, J. Gensrich, H.-P. Fink: "Structure Formation of cellulosic fibres from amine oxide solvents", Akzo-Nobel Viscose Chemistry Seminar, Stockholm, May 30 - June 3, 1994

6 Patent DE 44 20 304

NewCell® – A NEW OPTION FOR TEXTILE INDUSTRY

W. Rimpp, Akzo Nobel Faser AG, Germany

1. The Necessity for a New Fibre/Fabric

On one hand textile industry constantly searches for innovation i.e. for new fibres or better for new fabrics. As the consumer usually has very little knowledge about fibres - fabrics and later garments are the first stage when the consumer realizes the efforts undertaken by the textile industry for him or her.

On the other hand business is getting more and more complex. Introducing new fibres or in the case of NewCell® new filament yarns into the market first of all would even increase complexity for textile industry and therefore does not necessarily make life easier during the first phase. Once having mastered the initial problems and coming down the learning curve new products with superior properties could very well represent a considerable relief in production which pays off for the textile industry during a later phase. In this sense the introduction into the market of Lyocell in general and NewCell as the first Lyocell filament yarn in particular may be compared with the transition from the horse carriage to the motorcar as a mean for personal transport.

What determines the business of the textile industry currently? Based on market research conducted in Western Europe in 1996 three reasons have been identified why the market could be interested in a new filament yarn like NewCell® - the crisis of textile industry, the change of the consumer (behaviour) and the growing ecological awareness.

2. Reason # 1: The Crisis of Textile Industry

The actual and ongoing problems of the textile industry may be explained by three elements: The dilution of formerly existing fashion rules, the increased speed of textile business and the growing competition.

2.1 The Dilution of Fashion Rules

The Fashion Rule does not exist anymore in a sense that a trend will be created, converted into garments and the majority of the consumer follows for a certain period this trend and buys the new clothing. Despite all efforts to identify the relevant trends, to segment and subsegment the market one might come to the conclusion that the only mainstream trend is individualisation. Will this mean that we end up with "personal" clothing? Anyhow - the variety of trends increases certainly and when no clear trends and styles can be identified anymore by the experts of the textile industry the consumers decide for themselves that "anything goes" is the new rule.

If the consumer does not feel the desire anymore why he or she should buy new clothing, the primary task of the textile industry should be to create a fresh impetus for buying. A new product to come like NewCell® will facilitate this process.

2.2 The Increased Speed

Over the last years the speed of textile business has increased constantly and it still can't be foreseen when and whether this development will come to a standstill. Fashion cycles are getting shorter and shorter. From a system of 2, 4 or 6 collections per year we are moving towards a "continuous collection" with tremendous effects especially on logistics. In many cases forecasts turn out to be wrong and planned action becomes improvised reaction in order to keep capacities busy with all the margin eroding effects.

Consequently more constant success factors are needed in textile business.

2.3 The Growing Competition

Competition is growing. New trends, products and technologies spread all over the world with breath taking speed. A today's niche is a mainstream topic tommorow. This led to an alarming lack of uniqueness. The consumer sees in the boutiques of Tokio, New York or Rio de Janeiro the same goods he has already noticed in Paris, Milan or Düsseldorf.

Price becomes a dominating factor within the mass market. The comfortable middle price quality zone which has nourished for such a long period especially the European textile industry is eroding. The surprisingly growing luxury market cannot compensate for the turnover dropped-out.

There is no longer an European, American or Asian textile business. There are no protected zones anymore. All efforts by legislation or lobbyism to protect markets have turned out to be rather ineffective.

If we can't stop or reverse these developments, we need a concept, which provides global uniqueness!

3. Reason # 2: The Change of Consumer

Not only the textile business has changed dramatically over the past years but the consumer did so as well.

3.1 The Improved Information of the Consumer

The consumer has never been better informed. The new media provide access to almost any information without any delay. As he increasingly demands more value for money quality comes stronger into focus (again). On the other hand the consumer has tremendous difficulties to recognize the quality of garments at the point-of-sale. The consumer looks for quality signals and therefore needs assistance.

The integrity of product, brands and companies is being questioned increasingly. As the Brent Spar-affair proved companies like Shell which have enjoyed a very positive reputation over decades became the underdog overnight.

Due to around-the-clock information being available for the consumer there are less and less secrets. I.e.a company which tries to reduce costs by using child labour in Asia will be outed without almost any delay. Ethical correct shopping ("shopping for a better world") will be the behaviour of the future.

Honesty and authenticity will be crucial success factors of quality oriented products!

3.2 The New Complexity of Demand

Every single buying decision is released by emotional and rational factors. Depending on the personality of the consumer the ratio between these elements will be different. The absence of one of these categories will prevent any buying decision.

The new consumer wants emotion as well as function. He wants nature as well as high-tech. Emotionally the preference for the natural fibers cotton, wool and silk is still prevailing. His ratio has taught the consumer over the past decades about the functional superiority of the synthetics. A product of natural origin (emotion) like NewCell® with properties coming close to or even outperforming the synthetics (ratio) proved to be able to serve both demands in an exceptional manner (1).

The consumer asks for value-for-money and still wants to be seduced.

A new textile brand concept has to incorporate superiour emotional as well as functional qualities!

3.3 The New Religion: Individualisation

As the per-capita-consumption of clothing in some countries in Asia, especially in those passing the threshold of industrialization, proved over the last years, clothing will become more important again. Simply because they are much easier to afford than cars or even houses, clothing is preferred as a product which is best suited to express one's individuality.

When you ask people whether a (textile) brand name is important for them, in most cases they would deny (officially). It seems to be an expression of personal intelligence to deny being seduced by brands. Nevertheless all statistics show that consumers go for brands. For the average consumer a brand means orientation in terms of quality and in social terms. By dressing preferrably with the same brand(s) it helps them to differentiate from the masses simultaneously as it helps them to join the social group aimed for. In future the fabric and the fabric brand will play a more important role as means of differentiation.

Consequently there is a strong need for a unique and highly individualistic fabric brand concept!

3.4 The Desire for Subtle Luxury

Although people would rarely admit that they go for luxury, the consumer's desire for luxury is unbroken (2). However the demonstration of success will be much more discreet and unobtrusive. The success of companies like Brioni (for hand-tailored men's suits) over the last years, whose products are only recognised by insiders is the proof. Wearing the label of the designer garment on the outside of the sleeve is totally "out". "New Understatement" is the name of the game.

A new fabric brand which represents high value is the ideal means to express subtle luxury - may be not even noticeable to the surrounding but certainly to the pleasure of the consumer himself!

4. Reason # 3: The Growing Ecological Awareness

Although actual economic problems in Western Europe and Japan seem to have pushed ecology topics into the background, this only applies to the visible expression of actions taken i.e. to legislation. Nevertheless in almost every society there is at least a growing ecological awareness with different speeds of conversion into practice.

4.1 The Long-term Growing Ecological Consciousness

There is a long-term growing Ecological Consciousness. Ecology, so far the topic of the "young alternatives", will be a mainstream topic of the future.

This does not mean that socalled "ecology brands" - the flop of the Eighties and early Nineties (3) - will experience a comeback. The consumer doesn't ask for "ecology brands", he simply asks for ecologically correct behaviour, regardless for what product, irrespective where it has been produced or where it is to be sold. Ecology will no longer mean uniqueness and certainly there is

no (more) way to achieve a price premium for a product which complies with certain environmental standards. It will be a must for any producer to behave ecologically correct in order to remain competitive.

Ecological correctness must be a basic element of a new fabric brand!

4.2 The Legal Necessities

Beyond the expectations of the experts, the society and the consumer there will be strict legal standards in three areas:

Production Ecology, which means environmentally friendly production from the raw material to the finished product i.e garment (4).

Human Ecology, which demands the absence of any pollutants in the end product - a big challenge especially for Dyeing & Finishing.

Disposal Ecology, which could result in recycling concepts forcing producers to take back their products at the end of their life-cycle.

A new fibre brand has to comply with the demands of all three areas!

5. The Key Factors of the NewCell Concept

Being aware of the above mentioned elements which increase complexity of the business of the textile industry one might draw the conclusion that textile industry does not only need new products in form of fibres, machines, chemical agents and technologies to keep the engine "innovation" running, but also needs some aid how to market the products generated under these adverse circumstances.

The marketing concept of the first Lyocell filament yarn NewCell® is based on four elements:

the Ecology factor,
the Nature Factor,
the Value Factor and
the Fashion Factor.

5.1 The Ecology Factor

For marketing NewCell® this means environmentally friendly production including dyeing & finishing through all stages is a must. "Ecological correctness" beyond legislation is an obligation for all partners in the textile chain.

The finished products must be absolutely free of pollutants.

Ecology will mean reassurance to the consumer, but not uniqueness for the NewCell® brand. As usual the buying decision itself will be mainly determined whether the look and the hand pleases the consumer.

5.2 The Nature Factor

Although related to each other we differentiate between the Ecology Factor and the Nature Factor.

The brand will meet the unbroken long-term consumer preference for natural fabrics. NewCell® is a true natural fibre, because of natural origin and the concept of ecological correctness. The natural origin and natural character will be in focus of the entire communication for the NewCell® brand.

5.3 The Value Factor

The high value of the NewCell® brand will be based on its natural character, its special emotional qualities and its superiour functional performance. A clear signal of the brand's value will be its price.

The integrity of the value concept will result from a most consistent price policy: One NewCell" price concept will be practised worldwide.

5.4 The Fashion Factor

NewCell® offers a broad range of end-uses and creative applications. The NewCell® concept is geared to textile applications. Intensive development work with partners of fabric production, dyeing & finishing and fashion will ensure a high fashion orientation and attractivity of the end products.

6. The NewCell Market Segments

During the first three years from market entry NewCell® will focus in terms of production program, technical service and marketing on the market segments Elegant and Casual ladies Outerwear, Lingerie and Hosiery. As mentioned above the basic features are Nature, Ecology, Value, Fashion, Emotion and Function. The launch priorities are "female", "fashion", "value" "next-to-skin" and "feel of fabric".

6.1 Elegant Ladies Outerwear

The major benefits of NewCell® in the market segment "Elegant Ladies Outerwear" are:
– the new look and the new touch
– the option to decide between silky gloss or noble mattness on the fabrics
– the huge range of aesthetics, from transparent to wool-like fabrics
– the offer of an all-season-product
– the high versatility of different finished products, which can be derived from one piece of grey fabric.

6.2 Casual Ladies Outerwear

Being aware that the distinction between the market segments "Elegant Ladies Outerwear" and "Casual Ladies Outerwear" is rather artificial, it can be stated that the NewCell benefits in the latter segment are:
– the new look, as NewCell® is the first Lyocell filament yarn
– the particular soft touch, regardless whether the fabrics are fibrillated or not
– the option to decide between a more noble or a more sporty look
– the fluid drape and the rich colours possible, whether piece-dyed or printed
– the possibility to produce much lighter garments as with spun yarns
– the excellent dimensional stability of the fabrics and the garments if properly finished
– the fact that NewCell® is easier and consequently less costly in dyeing & finishing than fabrics made of Lyocell spun yarns as the difficult and expensive processes to release the primary fibrillation (macro fibrillation) and to remove it afterwards by enzyme cleaning or the like are not necessary with NewCell®.

6.3 Lingerie

The major benefits of NewCell® in the market segment "Lingerie" are:
– the soft touch, i.e. "peach skin" is a real novelty for Lingerie
– the high wear comfort
– the more solid drape of NewCell® compared to other cellulosics (5)
– the extraordinary stitch clarity
– the given machine washability, even at 60°C
– the versatility to meet the demands of the different end-uses
– the fact that a reinforcement of the fabrics by synthetics is no longer necessary due to the superiour functional performance of NewCell®.

6.4 Hosiery

Although still a lot of development work has to be done, already now the following benefits can be recognized in the market segment "Hosiery":
– the new look and the new touch (6) (in fact NewCell® is the first yarn innovation in this market segment since more than 40 years)
– the offer of a natural fibre for these end-uses
– the silk lustre
– the high wear comfort, due to its ability to absorb moisture
– the potential to produce NewCell® also in very fine deniers demanded for transparent hosiery.

7. The Benefits for the Textile Chain

The future orientation of the new product NewCell® and the new concept should lead to a strong competitive edge. A high long-term consumer acceptance or even preference based on the key factors Nature, Ecology, Value and Fashion will create continuous demand, thus giving more security to textile business. The production facilitation based on easier dyeing & finishing, versatility of the aesthetics and the functional superiorities of NewCell® will provide cost reductions in the textile chain.
Finally all elements mentioned before should ensure a high profitability for the benefit of all members of the textile chain.
NewCell® is not just a new product - NewCell® is also a new concept and therefore should be an interesting new option for textile industry.

References:

1 based on Market Research conducted in Germany by RSG, Düsseldorf in 1996.
2 New about the recent trend for luxury is, that the same consumer who buys a luxurious piece of clothing in a designer's boutique might shop some basic textile items in a department store!
3 In most cases "ecology brands" did not fullfill the necessities to release a buying decision. As most collections have been rather boring the emotional approach was not given. Just trying to bet on rational arguments made them fail.
4 NewCell is Ecotex 100 certified - naturally.
5 Lingerie fabrics made of NewCell are said to be less sloppy than known cellulosics.
6 In fact more than 98% of this market segment is dominated by polyamide.

FIBRILLATION – PROBLEM ODER CHANCE?

Stephanie Picht, Akzo Nobel Faser AG, Deutschland

1. Was ist Fibrillation

Neben der besonders hohen Trocken- und Naßfestigkeit, dem geringen Schrumpf und dem hohem Tragekomfort verfügen Lyocell - Fasern über eine andere besondere Eigenschaft: der Fibrillation.

Als Fibrillation wird das örtlich begrenzte Abspalten fibrillärer Elemente längs der Faserachse unter gleichzeitiger Einwirkung von Mechanik und Feuchtigkeit bezeichnet.

Die Ursache für diese besondere Eigenschaft ist der hohe kristalline Anteil mit hohem Orientierungsgrad in Richtung der Faserlängsachse.

Fibrillation v. NewCell
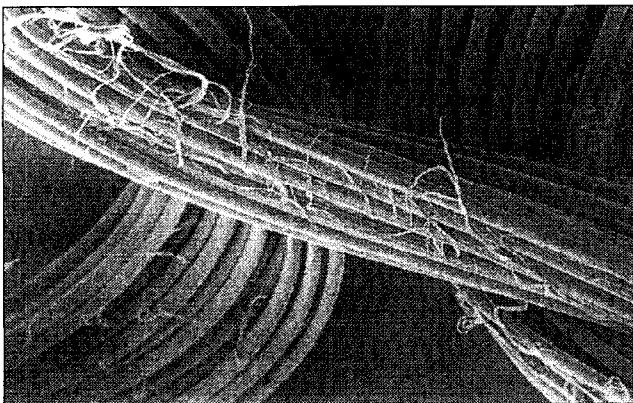
Abb. 1

Bei mechanischen Veredlungsprozessen, die in nassen Medien ablaufen (z. B. Färben, Tumbeln) findet zunächst eine Quellung statt.
Durch die Quellung und gleichzeitigen mechanischen Beanspruchungen werden die Fibrillen aufgebrochen.

Die Veränderung der Faser ist sowohl an der Faseroberfläche (optisch) als auch am Griff festzustellen.

2. Unterschied Fibrillation Lyocell – Filament / Stapelfaser

Zu den Lyocellfasern gehören zur Zeit Tencel und Lenzing Lyocell als Stapelfasern und NewCell als Lyocell - Filament.

Das Fibrillationsverhalten der Stapelfaser und des Filaments unterscheiden sich deutlich voneinander.

Aufgrund der offenen Faserenden in einem Stapelfaserartikel tritt unter Einwirkung von nassen mechanischen Prozessen eine viel stärkere Fibrillation ein als bei einem Filamentartikel. Ein anderer Unterschied ist, daß bei den Lyocell - Stapelfasern die Fibrillation in zwei Stufen erfolgt. D.h. zunächst erfolgt die Fibrillation an den freiliegenden, offenen Faserenden (Makrofibrilation).

Diese Fibrillation ist sehr viel größer als die Fibrillation auf der Längsseite der Faser. Sie wird durch enzymatische Prozesse in einem zusätzlichem Schritt entfernt, so daß anschließend die 2. Fibrillation, die sogenannte Mikrofibrillation ausgelöst werden kann.

Makrofibrillation Lyocell Stapelfaser
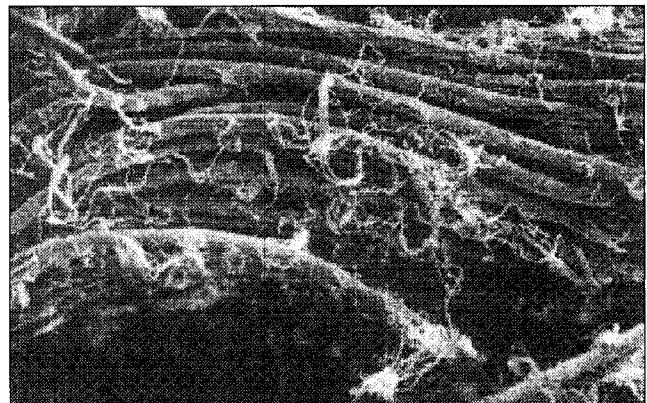
Abb. 2

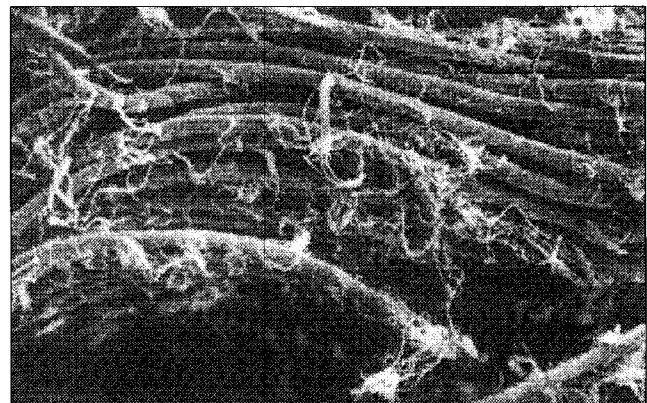**Mikrofibrillation Lyocell Stapelfaser**

Abb. 3

Artikel aus NewCell zeigen aufgrund des Filamentcharakters ein anderes Fibrillationsverhalten. Bei diesen Artikeln existieren keine offenen Faserenden, so daß die Fibrillationauslösung nur in einer Stufe erfolgt.

Deutlich wird der Unterschied in bezug auf das Fibrillationsverhalten bei Betrachtung der Ausrüstungsrouten von den Lyocell-Stapelfasern und dem Lyocell-Filament NewCell:

NewCell	Tencel/ L. Lyocell
Vorbehandlung	Vorbehandlung
|	|
Färbung	1. Fibrillation
|	|
mech. Behandlung	enzym. Behandlung
|	|
Endausrüstung	2. Fibrillation
	|
	Endausrüstung

Die Ausrüstungsroute von NewCell gestaltet sich wesentlich einfacher, da dort die gesamte Stufe der enzymatischen Behandlung entfällt.

Da NewCell keine Makrofibrillation sondern lediglich eine Mikrofibrillation entwickelt, entfallen zwei aufwendige Prozeßstufen.

Der Ausrüstungsprozeß wird dadurch wesentlich zeitreduzierter und somit wirtschaftlicher.

3. NewCell -verschiedene Ausrüstungsgänge

Die Fibrillation ist für den Veredler auf der einen Seite ein Gestaltungselement.

Auf der anderen Seite wird dem Veredler durch diese Eigenschaft aber die Einhaltung bestimmter Ausrüstungsregeln abverlangt, da sich dieses Gestaltungselement gegebenenfalls bei Nichtbeachtung als Problem herausstellen kann.

Durch Auswahl einer „ungeeigneten" Vorbehandlung, „ungeeignete" Ausrüstungsbedingungen (z. B. zu niedriger Anfangstemperatur), einer „ungeeigneten" Maschinenauswahl oder „ungeeigneten" Maschineneinstellungen, kann es zu Faltenbildungen, ungewollter Fibrillation, Schleifstellen oder den sogenannten „Krähenfüßen" kommen.

Neben diesen Parametern ist es für den Veredler wichtig, in Abhängkeit von der Konstruktion, der Zusammensetzung (100% Artikel oder Mischungen), dem gewünschten Endausfall und dem vorhandenen Maschinenpark für jeden Artikel die individuelle Ausrüstungsroute festzulegen, um die Eigenschaft der Fibrillation als Gestaltungselement optimal nutzen zu können.

3.1 Praxisbeispiel

Mit einem 100% NewCell - Artikel (Kette: 83dtex f50, gl S90/ Schuß 100 dtex f50 gl 2200 T/m, L 1/1) wurden vier unterschiedliche Ausrüstungsrouten durchgeführt.

A: entschlichten (breit), vorfibrillieren (Strang), laugieren (Strang), färben (Strang), trocknen (Strang)

B: entschlichten (breit), laugieren (breit), färben (Strang), trocknen (Strang)

C: entschlichten (breit), färben (KKV), trocknen (Strang)

D: entschlichten (breit), vorfibrillieren (Strang), färben (XKV), trocknen (Strang)

Die Endausfälle der Artikel zeigen deutlich den Einfluß der unterschiedlich Ausrüstungsrouten.

Artikel A und B wurden fast ausschließlich Strangbehandlungen unterzogen, hier ist eine starke Fibrillation (Grauschleier) zu erkennen.

Artikel B wurde im Gegensatz zu Artikel A im breiten Zustand vorbehandelt und anschließend erst im Strang gefärbt.

Das ist auch deutlich am Endartikel zu erkennen, die Oberfläche wirkt gleichmäßiger, nicht so unruhig, die Fibrillation wirkt feiner.

Artikel C und D zeigen deutlich weniger bzw. keine Fibrillation und somit auch eine andere Oberfläche, diese Artikel wirken glänzender, trotzdem ist ein weicher, fließender Fall erkennbar.

Bei allen vier Ausrüstungsrouten schließt sich als letzte Stufe die Endausrüstung an, d.h. es wird eine leichte Hochveredlung mit herkömmlichen Cellulosevernetzern aufgebracht um den fibrillierten oder nicht fibrillierten Endzustand zu stabilisieren.

Ist ein nicht fibrillierter Artikel gewünscht, sollte in jedem Fall nur in breitem Zustand ausgerüstet werden. Daß trotzdem eine mechanische Behandlung angeschlossen werden kann, um griffliche Verbesserungen zu erzielen zeigt Artikel C.
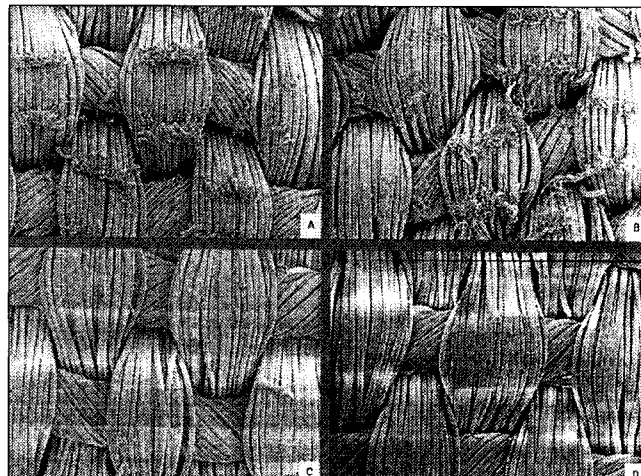
NewCell – unterschiedliche Ausrüstungsrouten

Abb. 4

4. Schlußbetrachtung

NewCell - Artikel bieten durch ihre besonderen Eigenschaften vielfältige Möglichkeiten bezüglich Griff und Oberflächenoptik. Ein sehr bedeutsames Instrument um diese Eigenschaft optimal nutzen zu können, ist die Veredlung.

Durch geeignete Auswahl der Ausrüstungsrouten bzw. Maschinen ist die Fibrillation kontrollierbar und keinesfalls als Problem zu sehen.

Verschiedene Begriffe wie „Pfirsichhaut", „Opaleszens", „samtig" versuchen die Chancen der Oberflächengestaltung von NewCell - Artikeln zu beschreiben.

Dennoch sind diese schillernden Begriffe nur eine Verkürzung dessen, was optisch und grifflich in einzigartiger Weise mit NewCell - Artikeln möglich ist.

GEZIELTE QUALITÄTSOPTIMIERUNG MIT LENZING LYOCELL®
IN DEN ERSTEN STUFEN DER „TEXTILEN KETTE"

Johann Feist, Lenzing Lyocell GmbH & Co KG, Austria

Die Darstellung der Textilen Kefte reicht vom Rohstoff „Holz" irberdenZellstoff, bis zu der Faserproduktion, den Prozessen Spinnerei, Weberei, Strickerei, Ausrüstung und Konfektion, hin bis zum Endverbraucher. Allgemein betrachtet, sind die ersten Stufen der Pipeline – „Faser-

produktion und Garnerzeugung" – richtungsweisend für die textilen Eigenschaften des Endproduktes. Gleichzeitig zu der näheren Betrachtung dieser Prozessstufen, gestatte ich mir einen allgemeinen Überblick über unsere Faser zu geben.

1. FASER

Gattung :
Diese neue Cellulosefaser stellt eine Sensation hinsichtlich Ökologie und Produkteigeschaften dar. Die BISFA hat dafur den Gattungsbegriff " LYOCELL , festgelegt. **CLY.**

Der Markenname lautet fijr unsere Faser – *Lenzing* **Lyocell®**.

Lösungsmittelspinnprozess
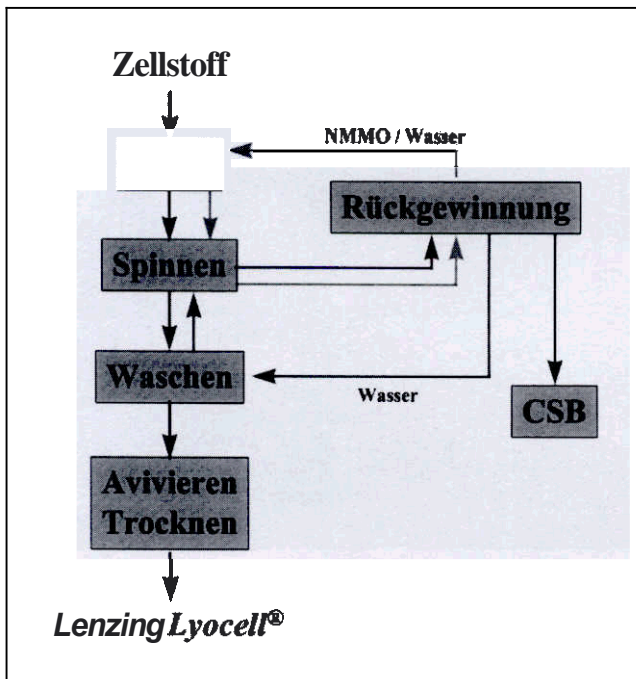
Bik

Es handelt sich um eine Cellulosefaser, die durch den Einsatz eines organischen Lösungsmittels NMMO (N-Methyl-morpholin-N-oxid) und Wasser, durch Lösungsmittelspinnen erzeugt wird. Der Prozess weist wesentlich weniger Schritte und Chemikalien als andere Prozesse auf, wobei sowohl das verwendete Lösungsmittel, als auch das Wasser nahezu vollstandig im Kreis geführt werden können.

1.1 Fasereigenschaften
Nachstehend werden die auffallendsten Fasereigenschaften angeführt:
- Hohe Faserfestigkeit
- Nassfestigkeit ca 85 %
- Hohe Schlingenfestigkeit

- Hohes Arbeitsvermögen Festigkeit - Dehnung
- Hohe Kapazitätsausnützung Faser - Garnfestigkeit
- Hohe Farbaffinität
- Niedrige Schrumpfwerte
- Kontrollierte Fibrillierung
- Permanente Kräuselung - Erhohte Dehnung

1.2 Faseroptimierung

Die sogenannte „Faser nach Maß" die von den Anwendern immer wieder gefordert wird. kann nur im ubertragenen Sinn durch sine laufende Kontrolle der gesamten TEXTILEN KETTE und eine damit verbundene Faseroptimierung angepeilt werden.

Eine wirksame Faseroptimierung kann nur zu entsprechenden Resultaten unter Zusammenabeit von:

> F + E FASER
> Faser - PRODUKTION
> Gam - PRODUKTION
> Alle Stufen der Kette besonders AUSRÜSTUNG
> Konsument - Trageeigenschaften

fuhren.

Hiebei fallt der Faserproduktion die Rolle zu die Selektion der entsprechenden Fasereigenschaften, sowie die Konstanz der Fasern zu gewahrleisten, um die Spinnereien mit dem fur das Produkt optimalen Rohstoff zu versorgen. Dies entspricht dem bei den Naturfasern bekannten Zusammenstellen von Spinnpartien.

Eine FASER die fijr anspruchsvolle Nischenproduktionen, sowie für eine große Vielfait von gehobenen Einsatzgebieten zur Anwendung kommen soll, kann dem Anforderungprofil nur bei permanenter Anpassungsarbeit, gerecht werden. Die erzielten Praxiswerte sind als Indikatoren fur die Überprüfung der **Faser**qualität oder der eingeleiteten Entwicklungen unerläßlich.

FASERPARAMTER:

Die nachfolgenden Parameter bestimmen im wesentlichen die Fasereigenschaften:
> Avivage
> Schnitt
> Kräuselung
> Zellstoff
> Prozeßparameter

Die Wertigkeit ist je nach Anwendungsgebiet nicht in fixer Reihung zu betrachten.
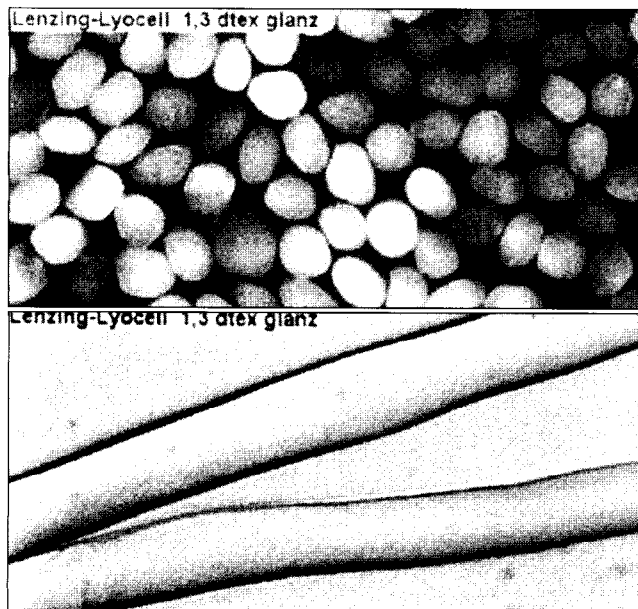
2. SPINNEREI

Faser-Schnitt

Lenzing-Lyocell 1,3 dtex glanz

Lenzing-Lyocell 1,3 dtex glanz

Bild 2

EINSTELLUNGEN

Position	Spinnmittel	Einstellungen	Drehzahl-Produktion	Bemerkungen
ÖFFNER	Nur 1 Schlagstelle Nadelwalze Sägezahnwalze	Abstand Speisewalze - Schlagkreis 2-4 mm	MAX 1000 upm	Mehr Öffnungs punkte mehr - Nissen
KARDE	VORREISSER Zahnwinkel 5-15° 35 - 40 Spitzen		MAX 1000 upm 650 upm	250 mm Durchmesser - Vorr. 350 mm Durchmesser - Vorr.
	Offene Roste, Messer Vorkardierelemente			Wenn <u>nicht</u> vorhanden, Zahnwinkel erhöhen und Drehzahl steigern
	TAMBOUR MAX 700 Spitzen		MAX 400 upm	
	ABNEHMER MAX 400 Spitzen	Tambour Abn. 4/1000"	25-35-70 kg/h	
	DECKEL MAX 400 Spitzen	Tambour- Deckel 12/10/9/9 /1000		Flexible Garnituren

Bild 3

KARDENSERVICE:

Es wird empfohlen, nachdem die Kardenbeschläge enorme Laufzeiten aufweisen (ca. 500 to Minimum.) auf ein Nachschleifen der Garnituren im Interesse einer guten Kardenvliesqualität zu verzichten .

ABFALL: Ein Indikator für die Kardierarbeit.

KARDE	Vorreisserabfall:	Verklebungen sollten im Rostabfall ausgeschieden werden
	Deckelabfall:	Ausgekämmter Deckelstrips, muß verklebungsfrei sein

2.1 Vertriebsidee

Die Faser nicht in der herkömmlichen Art dem Spinner anzubieten, sondern dieses Angebot auf eine begleitende Betreuung des angestrebten Produktes bis zum Fertigprodukt zu erweitern, bringt klare Vorteile :

Möglichkeit zur Produktionsplanung für alle Stufen
Rasches Erkennen von Schwachstellen
Optimierungspotenzial kann voll ausgenützt werden
Zeitfaktor für Produktentwicklung kann verkürzt werden
Chancen für erfolgreiche Produktentwicklung steigen

Absicherung des Preises durch Spezialartikel
Kein Billigprodukt - hochwertiges Image

Service in technischer u. kommerzieller Hinsicht, ebenso Werbungsunterstützung ohne Zusatzkosten.

2.2 Einstellungsvarianten

Für die Optimierung der Garnqualität sind insbesondere den Stufen ÖFFNEREI - KARDERIE bei der hier zum Einsatz kommenden Fasertype 1. 3 dtex/3 8mm, mit der notwendigen Sorgfalt zu begegnen.
Ein entsprechend hochliegender Garnqualitätsstandard ist nur mit einem Kardenvlies mit entsprechend niedrigen NISSEN-WERTEN zu erzielen.
Nachdem weltweit gesehen ein beträchtlicher Anteil von Karden (ca 50 %) älteren Generationen vom Maschinentyp her entsprechen, ist eine gezielte Auswahl von Spinnmitteln und Einstellungen, erforderlich .

Die Auswahl der nötigen Maschinenanordnungen sollte aus einem KURZSORTIMENT
Ballenbrecher
Feinöffner
Faserbeschickung
Karde bestehen.
Je nach dem zum Einsatz kommenden Maschinentypen sind die folgenden EINSTELLUNGSVARIANTEN zu wählen.

2.3 Qualitätsstandards

Die unter Zugrundelegung eines optimierten Maschinenparkes erreichbaren Qualitätsdaten sind innerhalb der 5% Werte der Usterstandards für Viscosegame anzusiedeln.

Es ist notwendig die Garnwerte für die Beurteilung zu relativieren
Maschinenpark
Maschineninstandhaltung
Spinnmittelwahl
Faserqualität
haben wesentlichen Einfluß.
Das Ziel ist ein GARN mit Qualitätszertifikat .

2.4 Produktionsgeschwindigkeiten

Für manche Produktentwicklungen ist eine Anpasssung der Produktiosgeschwindigkeiten notwendig .
Ansonsten sind aufgrund der Faserfestigkeit und der Faserbeschaffenheiten für die Produktiosstufen folgende Verarbeitungsgeschwindigkeiten möglich:

Strecke:	800 m/min	
Flyer:	1.200 m/min	
Ringspinnmaschinen:	18.000 nspi	38 mm Ring, 36 Läufermeter
Spulmaschine:	1.200 in/ min	
Openend:	128.800 nrot	31 Rotor

2.5 Auspinngrenzen

	Nm	tex	dtex	Fasem im Querschnitt
Ring	150	6.7	1.3	52
OE	80	12.5	1.3	96

Aufgrund der sehr guten Festigkeitswerte und des Arbeitsvermögens ist die Aussspinnung in der Praxis in den angegebenen Bereichen durchaus mit guten Bedingungen möglich.

2.6 Einflussgrösse Stapel:

Um die Auswirkung der Stapellänge auf die Garnqualität und die Laufeigenschaften zu testen haben wir folgende Schnittvarianten zugrunde gelegt: bei Fasertype 1.3 dtex/38 mm.
RINGGARNE
Nm 50 wobei der Schnitt mit 27 mm wegen nicht praxisgerechtem Lauf ausgeschieden wurde.

Schnittlänge   27   30   34   37   40   mm

Versuchsreihen

Uster CV% wird bei abnehmender Stapellänge etwas schlechter
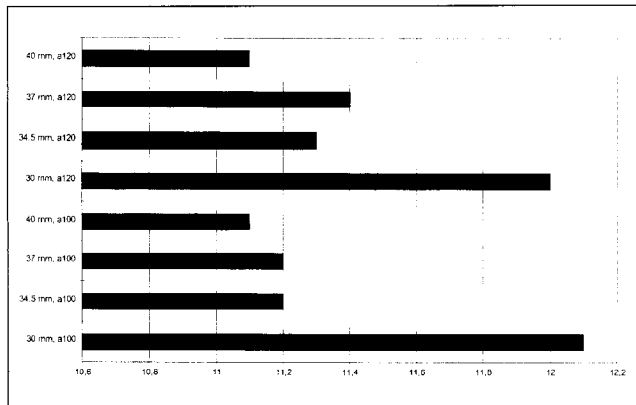
Bild 3

Dünn- und Dickstellen nehmen mit kürzerem Stapel zu
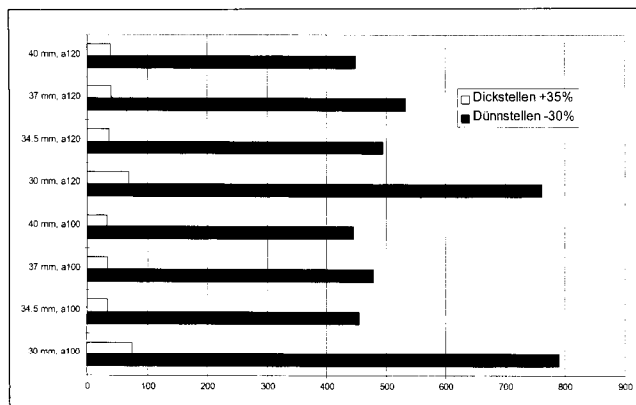
Bild 4

Mit abnehmendem Stapel sinkt die Bandhaftung und ebenso die Nissenzahl. Bei 34 mm scheint eine Barriere vorzuliegen die wieder einen Anstieg der Nissen anzeigt.

Noppen
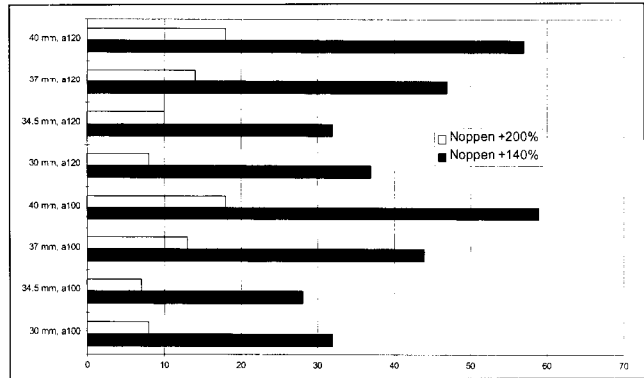
Bild 5

cN/tex, Dehnung% Abnahme der Festigkeit bei reduzierter Faserlänge
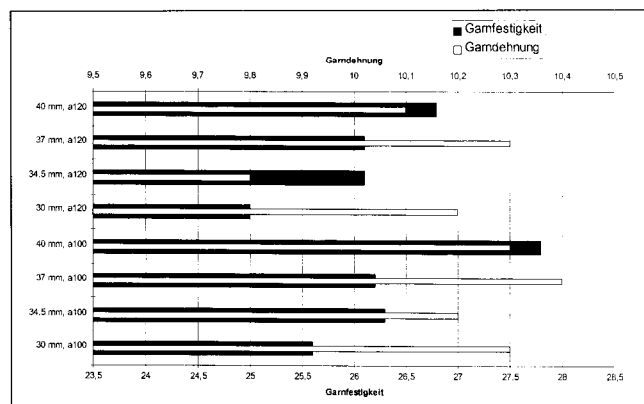
Bild 6

Classimat generell sehr gute Ergebnisse bei 34 und 37 mm.
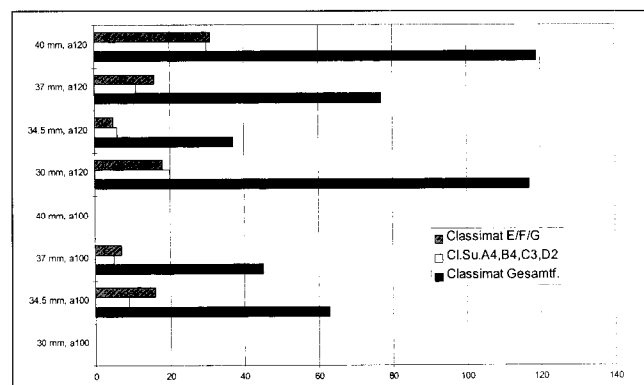
Bild 7

Haarigkeit nach Uster nimmt mit Stapeleinkürzung zu.
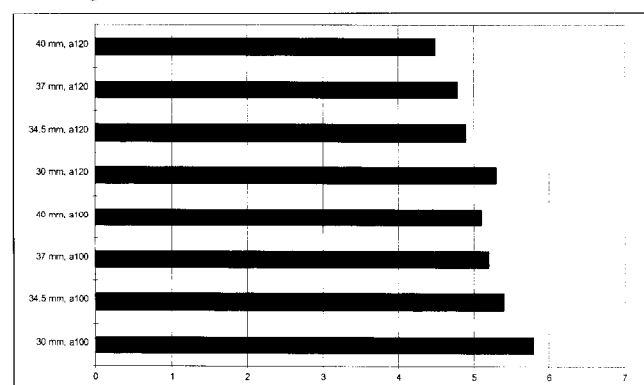
Bild 8

OE-GARNE

Nm 60

Schnttliange 27   30   34   37   40   mm

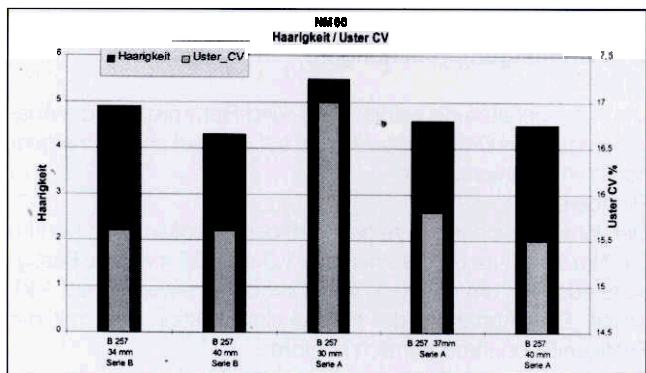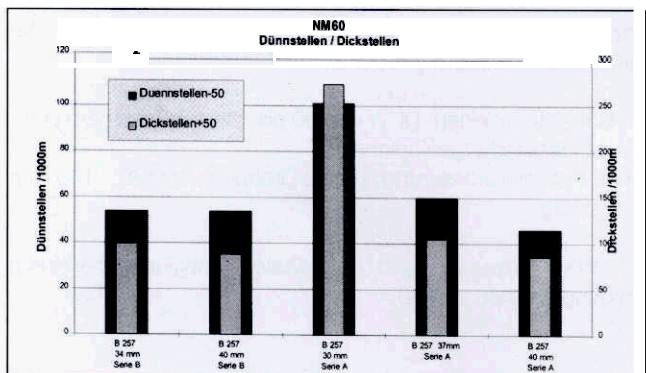### UsterCV%-Haarigkeit steigen bei abnehmendem Stapel

Bild 9

Diinn-Dickstellen der Bereich 34 ‑ 37 mm ist hier noch immer vertretbar

Bild

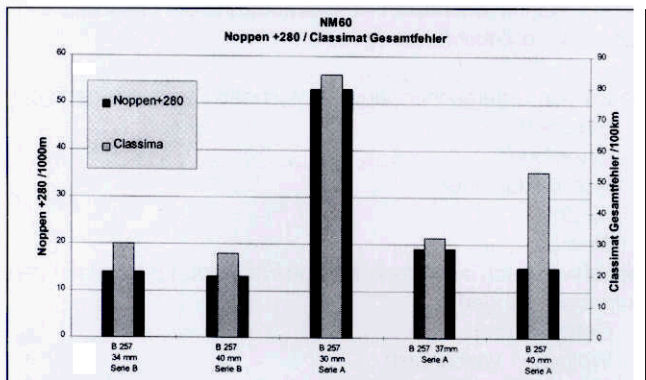### Nissen-Classimatfehlerahnliche Tendenz wie bei Diinn-Dickstellen

Bild 1

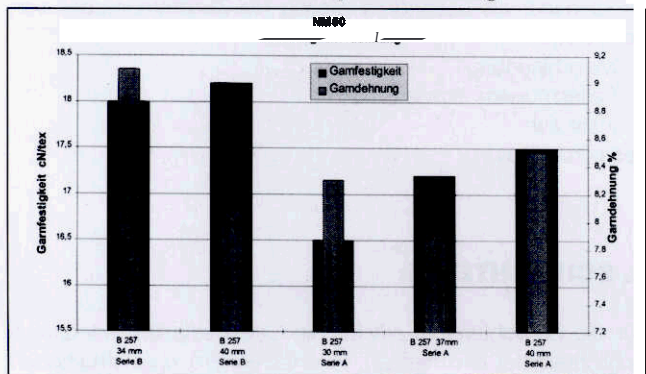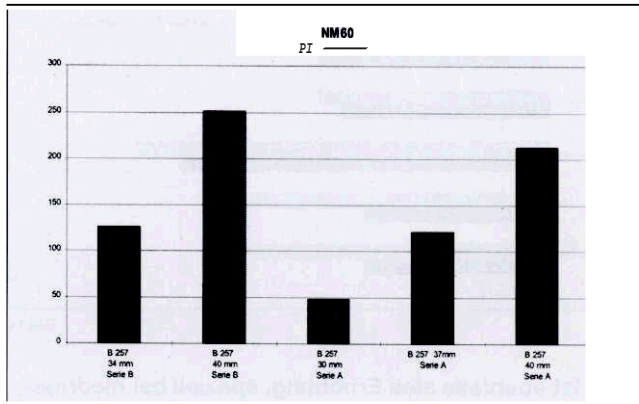### cN/tex-Dehnung% Die Festigkeit geht ziemlich konform mit Stapeleinkürzung

Bild 1

Die Fadenbruche zeigen sehr schön wie ein kurzerer Stapel, bei kleinem Rotordurchmesser 31 mm sich in den Fadenbruchwerten niederschlagt

Bild

Es zeigt sich deutlich, daß die Stapeleinkurzung der Qualitätsverbesserung entgegenkommt. Dies wird durch die pernamente Krauselung der Faser ermogiicht und zeigt auch wie die hohe Biegesteifigkeit der Faser positiv auf eine Stapeleinkurzung reagiert. Beim OE Garn ist eine bessere Einbringung der Faser in den Rotorbereich mit Abnahme des Faserstapels möglich. Eine regelmäßige Einspeisung wird mit einem 27 mm Rechteckstapel nicht mehr gewahrleistet. Hier ist eine zu ruckweise Einspeisung gegeben, es ware eine bessere Fuhrung der Fasern im Bereich Tisch - Einzugswalze - Auflösewalze notig.

Eine zu große Variation in den Schnittlängen wurde jedoch den flexiblen Einsatz der Spezialfaser teilweise einschränken. Auch ist unbedingt eine Kontrolle der Auswirkungen in allen Stufen der Verarbeitungskette notig .

2.7 Einflussgrösse Avivagetypen:

Die Untersuchungen in der Richtung zeigen, daß es sich um einen sehr diffizilen Bereich handelt. Aufgrund der Faserbeschaffenheit, Querschnitt - OVAL, Langsansicht - strukturlos, Biegesteifigkeit - hoch, ist beim Einsatz von zuviei Gieitern schneil ein Punkt erreicht, wo empfindliche Festigkeits- u. Dehnungsverluste auftreten. Es ist daher wichtig, daß die notige Balance zwischen Haft- u. Gleitrnitteln gefunden wird, um die optimalen Verzugs- u. Laufeigenschaften zu gewahdeisten.

2.8 Einflussgrösse Spulprozess:

Eine Verschlechterung durch den Umspulprozess in den Garnwerten ist je nach Spulgeschwindigkeit und eingesetztem Spulmaschinentyp gegeben, Nm 5 0 - Ring.

Wir haben unter Zugrundelegung einer Spulmaschinengeschwindigkeit von 1300 m/min und einer Spulmaschine AC23 8 Schlafhorst das Verhalten von Lenzing - Lyocell - Garn untersucht.

Die nachstehenden Diaggramme zeigen:

UsterCV% Garn, beim Umspulen geht der Drehungs-beiwert erstaunlich massiv in die Zunahme ein
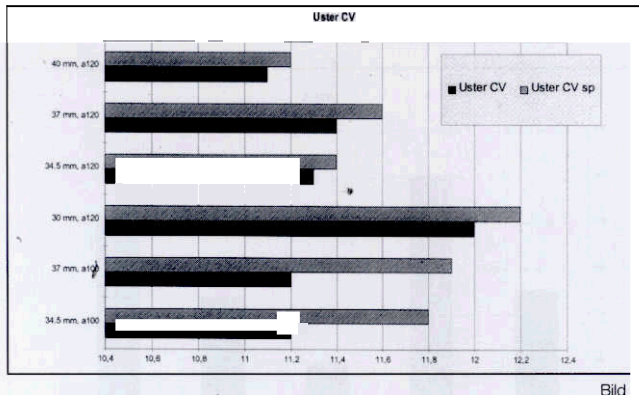
Bild

Hier ist ebenfalls eine Erhohung, speziell bei niedrigerem Drehungsbeiwert zu erkennen.
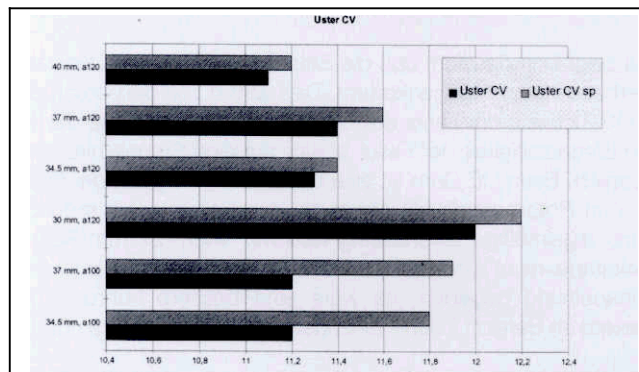

Bild 1

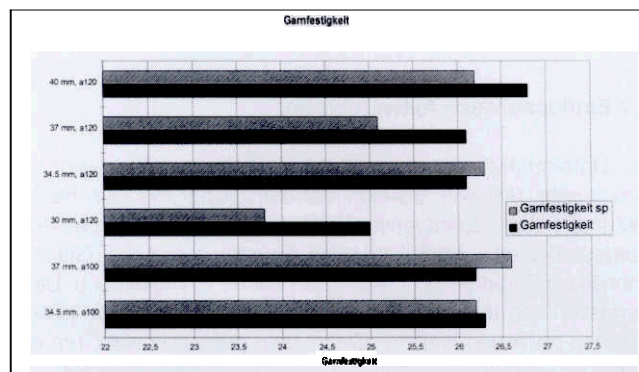## Garnfestigkeit zeigt eine vertretbare Abnahme durch den Spulprozess.

Bild 1

2.9 Mischungen:

Grundsatzlich ist die gesamte Palette von Fasem. wie Viscose, Modal, PES, Acryle, PVA. PA, Leinen, Wolle, Baumwolle in Mischungen mit Lyocell zu verarbeiten. Dies sowohl in Streckenmischungen, wobei 1 Mischstreckenpassage und 2 Doublierpassagen empfohlen werden, als auch in Intimmischungen.

Fur die gultigen Einstellwerte ist der Mischungsprozentsatz, die Materialbeschaffenheit und manchmal die Avivagetype des beizumischenden Materials, ausschlaggebend.

Die Auszeichnung als Lyocellprodukt ist jedoch nur dann gegeben, wenn mindestens ein 60 % Lyocell- Faseranteil eingesetzt wird.

Eine Beimischung zu den angefuhrten Fasertypen, zeigt eine deutliche Aufwertung derselben in einer Verbesserung des Griffes, der Optik, sowie der Trageeigenschaften .

2.10 Drehunguntersuchungen:

Die Aufgabenstellung war im Ring- und Rotorprozess die Auswirkungen des Drehungsbeiwertes auf die Lauf und Garneigenschaften zu untersuchen
Ringbereich :
Die Voruntersuchungen zeigen, daß der Festigkeitswert bei Nm 34, Nm 50 u. Nm 85 - Garnen aus 1.3 dtex/38 mm eine Festigkeitsreduktion um ca. 10 % vom Drehungsbeiwert 90 auf 140, ergibt. Eine Abnahme der Haarigkeitsreduktion geht mit der Festigkeitsabnahme ziemlich konform
Rotorbereich:
Hier möchten wir auf die bereits durchgenommen Spinnversuche der Fa. Schlaihorst hinweisen (Lenzinger Berichte 77/1997, Seite 24), aus denen hervorgeht, daß der Drehungsbeiwert nur abzusenken ist wenn die Rotordurchmesser vergrößert und damit die Spinnspannung reduziert wird.

So läßt sich mit einem DBW von 90 bis 140 ein Rotorgarn unter Industriebedingungen spinnen.
Ohne Rotorvergrosserung ist eine Drehung von 130 - 140 rpm notwendig.

In beiden Prozessen liegen die hochsten Festigkeiten bei einem Drehungsbeiwert von 90.

2.11 Neue Verfahrenstechniken:

Neue Verfahrenstechniken werden aufgrund der Faservielseitigkeit sicher in Zukunft Einzug halten.

So werden Untersuchungen mit folgenden Systemen gemacht
   Sirospun
   Chorspun
   Kompaktspinnen
   Air Jet
   Diverse
Hiebei wird auch die fortschreitende Entwicklungsarbeit mit den Aufgabestellungen
   Defibrillierung
   Wolltype - Varioschnitt
   Spinngefärbte Fasem
einfliessen.

Es ist daher als besonders wichtig die Zusammenarbeit von Verarbeitern
   Maschinenbau
   Faserzeugem, sowie
   Instituten
hervorzuheben .

3. SCHLICHTEREI:

Fur die Schlichterei ist ein fur die Cellulosefasern adequates Schlichtemittel einzusetzen, in unseren Prozessinstruktionen sind Schlichtmittelerzeuger und Rezepturen angefuhrt.

4. WEBEREI:

Auf allen gängigen Webstuhltypen ist ein Verarbeiten ohne merkliche Probleme gegeben. Die Standardwerte für folgende Verarbeitungsstufen sind wie folgt:

Zetteln: 0.3 - 0.5 Fadenbrüche / 1 Mio Fadenmeter .

Weben: 1 Fadenbruch / Webstuhlstunde (ca. 120 km Faden).

ALLGEMEIN:

Ziele:

Bekanntheitsgrad der Faser fördern

Innovationsbemühungen unterstützen

Globaler standortunabhängiger Einsatz

Qualitäts- u. Produktivitätsprung sicherstellen

Nischenproduktion unter möglichster Kostenneutralität

Auf breiter Basis Weiterentwicklung und Optimierung betreiben

Rechtzeitige Sicherstellung von neuen Einsatzgebieten

Hoffentlich ist es mir gelungen mit dieser allgemeinen Übersicht die Vielseitigkeit dieser neuen Faser aufzuzeigen und so hoffe ich, die Anwender der TEXTILEN KETTE anzuregen diese Neuheit in der Praxis zu erproben und zu erleben.

Wir sind gerne bereit dabei tatkräftig zu unterstützen!

VEREDLUNG VON LENZING LYOCELL®

Fritz Brauneis und Markus Eibl, Lenzing Lyocell GmbH & Co KG, Austria

In diesem Beitrag wird im besonderen auf die Veredlung von Lenzing Lyocell® - Maschenware eingegangen.

Maschenware aus Lenzing Lyocell® zeichnet sich durch den angenehmen weichen Griff und den hervorragenden Fall aus. Wie Baumwolle besteht Lenzing Lyocell® aus Cellulose und ist daher atmungsaktiv, natürlich und Feuchtigkeits-aufnehmend. Die einzigartige Struktur von Lenzing Lyocell® führt aber zu einem, mit Baumwolle nicht erreichbaren, hervorragende Tragekomfort. Bedingt durch das hohe Naßmodul von Lenzing Lyocell® sind Flächengebilde aus dieser Faser sehr dimensionsstabiil, d.h. es tritt bei der Haushaltswäsche kaum ein Schrumpf auf.

Die, in der Literatur schon hinlänglich beschriebene, Neigung der Lyocellfasern birgt bei der Veredlung von Maschenware aus Lenzing Lyocell® einerseits die Möglichkeit völlig neue Griffvarianten zu kreieren, andererseits muß der Veredlungsprozeß entsprechend gewählt werden um nicht zu einer unregelmäßigen Fibrillierung der Warenoberfläche zu kommen. Weiters muß für die Herstellung von waschbaren Maschenwaren aus Lenzing Lyocell® die Fibrillationsneigung nach der Veredlung entsprechend vermindert werden. Dafür stehen mehrere Methoden zur Auswahl.

Die Gebrauchstüchtigkeit von Maschenware aus Lenzing Lyocell® wird von der Einbindung der Fasern in das Garn beeinflußt.

Bei der Scheuerung von nassen textilen Flächen aus Lyocell werden die Fasern fibrilliert. Naturgemäß werden nur die Fasern fibrilliert welche gescheuert werden und daher nur jene an der Oberfläche. Abstehende Fasern (Haarigkeit) und Fasern, welche während der Behandlung aus dem Garn herausgearbeitet werden fibrillieren zuerst. Somit werden Fasern im Garn nicht fibrilliert, sondern nur Fasern an der Warenoberfläche! Da diese fibrillierten „Büschel" relativ lang sind verschlingen sie sich ineinander und bilden unansehnliche Pills.

Bild 1

Dieser Prozeß wird ist als Vorfibrillieren, Erste Fibrillation oder Primärfibrillation bezeichnet.

Eine Reinigung des Gewebes erfolgt anschließend durch eine Cellulasebehandlung. Da die fibrillierten Pills eine höhere Oberfläche haben als nicht fibrillierte Fasern werden die Pills bevorzugt angegriffen und entfernt. Man erhält eine saubere Warenoberfläche.

Bild 2

Wenn bei der weiteren Verarbeitung Mechanik auf das Textil einwirkt werden nur Fasern fibrilliert welche in das Gewebe eingebunden sind und es entstehen daher keine Pills.
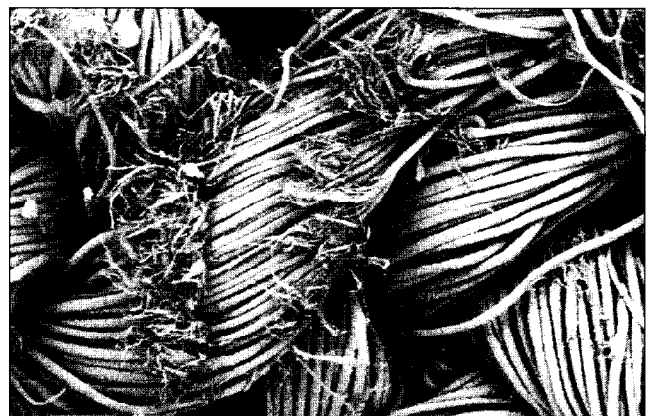

Bild 3

Somit hat der Prozeß des Fibrillieren/Defibrillierens nichts mit einem „Entbasten" bzw. Abschälen von Lyocell-Fasern zu tun. Vielmehr werden nur jene Fasern entfernt welche sich leicht aus dem Garnverbund herauslösen können! Somit ist die Qualität der Garne für das Verarbeitungsverhalten außerordentlich wichtig.

Die Variation der Drehung von Ringgarnen hat ergeben, daß die Einbindung der Fasern in Garne mit einem Drehungsbeiwert von 110-120 deutlich besser ist als für Garne mit einem Drehungsbeiwert von 100. Da besonders bei der Veredlung von Maschenware die Entstehung von Pills aufgrund herausgearbeiteter Fasern besteht, sollten zur Herstellung von Maschenware nur Garne mit einem Drehungsbeiwert von mind. 110 besser 115 verwendet werden.

Die Einbindung der Fasern wird auch durch Verzwirnen bzw. durch den Einsatz von Siro-Garnen verbessert.

Bei gleicher Maschendichte nimmt die Verflusung während der Vorfibrillierung mit zunehmender Garnfeinheit zu. Das ist sowohl auf die offenere Struktur, wie auf die verminderte Einbindung zurückzuführen. OE-Garne führen bei gleicher Garnnummer zu einer geringeren Verflusung als Ringgarne.
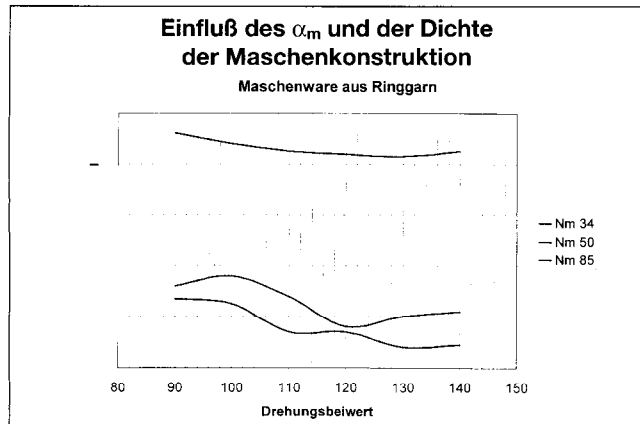

Einfluß des α_m und der Dichte der Maschenkonstruktion

Maschenware aus Ringgarn

— Nm 34
— Nm 50
— Nm 85

Drehungsbeiwert

Bild 4

Vergleich Spinnsystem

Neigung zur Verflusung
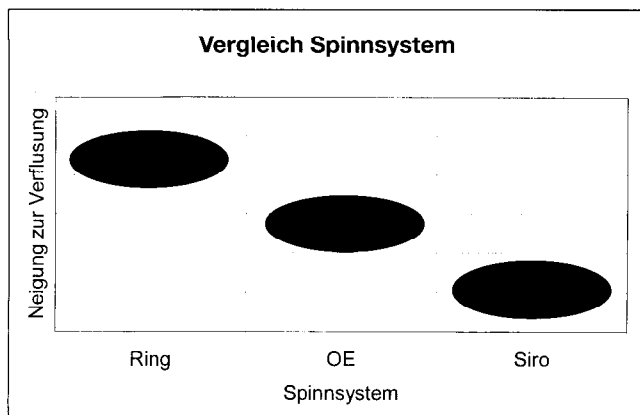
Ring   OE   Siro

Spinnsystem

Bild 5

Dieses Ergebnis wird auch durch die Messung der Garnquellung in Wasser von Garnen verschiedener Drehung bestätigt (B.Köll Lenzing AG). Mit zunehmender Drehung nimmt die Quellung ab, wodurch eine bessere Einbindung der Fasern bei Naßprozessen zu erwarten ist. Die geringere Quellung der Garne führt naturgemäß auch zu einem besseren Laufverhalten der textilen Flächen in Strangbehandlungsmaschinen und somit zu einer Reduktion der Gefahr der Bildung von Lauffalten.

Dickenquellung von Garnen Einfluß des Drehungsalpha

Dickenquellung %

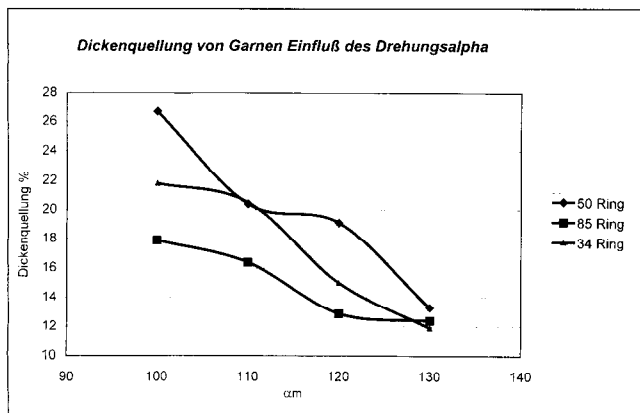◆ 50 Ring
■ 85 Ring
▲ 34 Ring

αm

Bild 6

Versuche mit 1,3 dtex Fasern verschiedener Schnittlängen haben gezeigt, daß bei einer Faserlänge unter 37 mm die Verflusung der Warenoberfläche bei der Veredlung deutlich zunimmt. Das liegt an der erhöhten Anzahl von Faserenden, welche leicht aus den Garnen herausgearbeitet werden können und nach einer Fibrillierung zu Pills führen.

Veredlungsabläufe:

Als günstig hat sich als erster Schritt ein Sengen erwiesen, zur Zeit haben jedoch leider nur wenige Veredler eine Schlauchsenge zur Verfügung.

Eine anschließende Laugierung mit 68 °Bé verbessert die Laufeigenschaften der Ware im Jet und verringert die Gefahr der Lauffaltenbildung.

Für die Verarbeitung von Maschenware haben sich besonders Overflow- bzw. Softflow-Jets bewährt. Die hohe Bauweise ist jener der länglichen Bauart vorzuziehen, weil durch die höhere Mechanik die Fibrillierung und Defibrillierung intensiver erfolgen kann.

Die Verarbeitung kann in Schlauchform und, wenn von der Art der Konstruktion aus möglich (Einrollen der Kanten), auch aufgeschnitten erfolgen. Beim Arbeiten in Schlauchform kann es in der Schlauchinnenseite zu einer verstärkten Ansammlung von Faserstaub und Kurzfasern kommen.

Während aller Prozeßschritte in der Strangform muß ein geeigneter Lauffaltenverhinderer verwendet werden.

Bei allen Naßprozessen in Strangform ist zu beachten, daß Behandlungstemperaturen unter 50°C vermieden werden sollen (auch bei den diversen Spülprozessen). Temperaturen darunter bewirken eine erhöhte Gefahr der Bildung von Scheuerstellen und Falten.

„klassische" Veredlungsroute:

Vorwäsche wenn notwendig

Primärfibrillation: 4-6g/l Soda kalziniert, 60-90 min bei 90°C. Wenn die Primärfibrillation nicht sehr intensiv ausgelöst werden muß, kann diese gleichzeitig mit einer Färbung mit Reaktivfarbstoffen (Heißfärbern) erfolgen.

Färbung: Üblicherweise wird Lenzing Lyocell® mit Reaktivfarbstoffen gefärbt (Echtheiten, Brillianz etc.) Um allzu große Farbton- und Farbtiefenänderungen bei der anschließenden Defibrillierung zu vermeiden, müssen entsprechend echte Farbstoffe ausgewählt werden (Beständigkeit gegenüber essigsäurehältige Bäder). Auch auf einen Einfluß der Farbstoffe auf die Aktivität der Cellulasen ist zu achten.

Defibrillierung für Maschenware ist, im Vergleich zu Geweben, ein um 30-50 % erhöhte Einsatzmengen an Cellulase notwendig. Bisher zeigen nur saure Cellulasen eine entsprechende Aktivität. Bei Mischungen von Lenzing Lyocell® mit natürlichen Cellulosefasern sind Formulierungen mit einem erhöhten endo-Cellulase-Gehalt vorzuziehen

> **Reinigung:** Falls Faserablagerungen (Staub) auf der Textiloberfläche als störend empfunden werden, können diese durch ein Waschen der offenen Ware auf Breitwaschmaschinen (z.B. „Aquajet" von Santex) oder bei Schlauchware durch Wenden der Ware und trockenem Tumbeln auf einem diskontinuierlich arbeiteten Tumbler z.B. Thies roto-Tumbler entfernt werden.

> **Hochveredlung:** Um ein Nachfibrillieren während Haushaltswäschen zu vermeiden, muß die aufgeschnittene Ware mit einem Reaktantharz hochveredelt werden. Als am wirksamsten haben sich dabei Formulierungen mit veretherten DHDMEU erwiesen.

Diese Veredlungsroute beruht auf der Methode der Entfernung aller schlecht eingebundenen Fasern. Wenn alle diese Fasern entfernt sind, wird das Auftreten einer „unerwünschten" Fibrillation = Verflusen vermieden.

Problematisch dabei ist die Staubentwicklung nach der Enzymbehandlung.

Da bei einem Fibrillation / Defibrillationszyklus natürlich nicht alle schlecht eingebundenen Fasern zu entfernen sind, können bei anschließenden Haushaltswäschen weitere Fasern herausgelöst werden und zu einer Verflusung führen. Daher müssen Lenzing Lyocell Maschenwaren hochveredelt werden um eine entsprechende Waschbarkeit zu erlangen. Die Hochveredlung mit Reaktantharzen führt zu einer Vernetzung der Fasern. Daher werden diese Fasern bei einer Wäsche nicht mehr fibrilliert und können sich somit nicht mehr verhaken und Flusen bilden. Weiters wird eine Aufhellung der Farbe aufgrund einer Lichtstreuung an den Fibrillen verhindert.

AE 4425:

Viele Veredler scheuen die Enzymbehandlung aus prozeß- und/oder kostentechnischen Gründen. Weiters geht bei Maschenwaren der Trend hin zur kunstharzfreien Ausrüstung, aus Gründen der Kosten, dem Warengriff und der Formaldehydproblematik, insbesondere bei Textilien für Kinder und Kleinkinder,

als auch bei Textilien welche am Körper oder körpernah getragen werden.

Daher bietet sich für die Herstellung eine Maschenware aus Lenzing Lyocell® mit klare Warenoberfläche folgender Veredlungscyklus an, welcher zur Zeit in Betriebsversuchen optimiert wird:

> **Vorwäsche** und gleichzeitiger Einsatz von **AE 4425**

> **Färben** mit Reaktivfarbstoffen

> **Avivieren**

Durch die Behandlung mit AE 4425 während der Vorwäsche wird die Fibrillierneigung der Faser so weit herabsetzt, daß bei den anschließend durchgeführten Veredlungsprozessen in Strangform, eine Verflusung und Vergrauung der Oberfläche des Textils nicht auftritt. Abstehende und herausgearbeitete Fasern werden somit nicht fibrilliert und bilden daher keine Flusen. Eine Säuberung der Oberfläche mittels Cellulasebehandlung ist nicht notwendig. Weiters wird ein Nachfibrillieren während der Haushaltswäsche unterbunden.
Die färberischen Eigenschaften werden dadurch kaum beeinträchtigt.
Um eine Sekundärfibrillation auszulösen ist je nach Einsatzmenge des AE 4425 die Mechanik entsprechend zu erhöhen.
Der Einsatz von AE 4425 wird üblicherweise im Ausziehverfahren erfolgen. Dies kann wie geschildert während der Vorwäsche von textilen Flächen oder auch gleichzeitig mit einer Garnfärbung mit Reaktivfarbstoffen erfolgen.
Da die Fasern nicht fibrilliert werden, kann auch keine Aufhellung der Farbe durch Lichtstreuung auftreten.

Vorteile der neuen Veredlungsroute gegenüber der klassischen Variante:
Applikation im Ausziehverfahren und somit für die Behandlung von Schlauchware geeignet.
Verkürzte und somit kostengünstigere Verfahrenroute kein Einsatz von Kunstharz nicht toxisches Produkt, kann auch während der Färbung eingesetzt werden (z.B Garnfärbung) stark verminderte Staubentwicklung

LYOCELL-ERZEUGNISSE MIT EINGEBAUTEN FUNKTIONALEN EIGENSCHAFTEN

F. Meister, D. Vorbach, Ch. Michels, R. Maron, K. Berghof und E. Taeger, TITK, Deutschland

Polysaccharides are belonging to those natural products which had developed a great number of functional diversify. The development of more than one of these properties in the very same product requires a simultaneous dissolving and shaping process. In the case of the corresponding combination of variously functional polysaccharides or of other, even not soluble additions. and shaping according to the Lyocell process, cellulose products with novel qualities become possible.

The contribution describes the possibilities for the occlusion of polyelectrolytes, selected polysaccharides or polysaccharide derivates and higher concentrations of carbonblack. The resultant influences on the textile physical level and the made effects of produced fibres and filaments are represented from the point of view of technical application. Here the paper will focuse on fungicide qualities, modified behavior toward water as well as humidity dependent changes of the electric resistance of conductive cellulose filaments.

Polysaccharide gehören zu jenen Naturstoffen, die über eine Vielzahl funktioneller Eigenschaften verfügen. Die Erschließung von mehr als einer dieser Eigenschaften in ein und demselben Erzeugnis erfordert einen simultanen Auflöse- und Verformungsprozeß. Bei entsprechender Kombination verschieden funktioneller Polysaccharide bzw. anderer, sogar nicht löslicher Zusätze und Verformung nach dem Lyocell-Prozeß werden Celluloseerzeugnisse mit neuartigen Eigenschaften zugänglich. Der Beitrag stellt die Möglichkeiten zur Einlagerung von Polyelektrolyten, ausgewählter Polysaccharide bzw. Polysaccharidderivate und höherer Konzentrationen von Ruß dar. Die resultierenden Einflüsse auf das textilphysikalische Faserniveau und die erzeugten Effekte werden aus anwendungstechnischer Sicht dargestellt. Hierbei stehen fungizide Eigenschaften, modifiziertes Verhalten gegenüber Wasser sowie feuchteabhängige Änderungen des elektrischen Widerstandes leitfähiger Cellulosefilamente im Vordergrund.

Einleitung

Die makromolekularen Polysaccharide wurden im Prozeß der Organismenevolution zu Trägern unterschiedlichster Leistungen entwickelt. Beispielhaft seien dafür kräfteaufnehmende Gerüst- und Stutzfunktionen, die Rolle als Speicher- und Reservestoffe, wasserbindende Quellfunktionen, Permeations- und Stofftrennaufgaben, selektive Adsorptionsfunktionen sowie bakterizide und fungizide Abwehraufgaben genannt.

Die Verschiedenartigkeit der Leistungen, ihre gute Veredlbarkeit sowie Mannigfaltigkeit, gepaart mit einer für die Umwelt gefahrlosen Entsorgungsmöglichkeit, trugen zu einer großen industriellen Nutzung dieser Klasse nachwachsender Rohstoffe bei. Dabei standen jedoch im wesentlichen Anwendungen im Vordergrund, die lediglich die Funktion eines bestimmten Polysaccharides ausnutzen.

Die Tatsache einer weitgehend monofunktionaten Nutzung ist darauf zurückzuführen, daß Polysaccharide zu den nicht schmelzbaren Makromolekülen gehören. Das Erschließen einer kombinierten Nutzung verschiedener Polysaccharidfunktionen setzt einen simultanen Auflöse- und Verformungsprozeß voraus. Auf Grund der unterschiedlichen Struktur und Reaktivität verschiedener Polysaccharide sind all jene Technologien ungeeignet, die eine Verformung von semistabilen Derivaten zum Inhalt haben.

Seit Anbeginn der großtechnischen Viskosefaser-Produktion wurden deshalb Anstrengungen dazu unternommen, alternative Verfahren zu entwickeln. Idealerweise sollte ein zu entwickelnder Prozeß nur ein Lösungsmittel erfordern, das nach dem Verformungsprozeß technologisch einfach und möglichst quantitativ wiederaufbereitet werden kann.

Zwischenzeitlich ist ein solches Verfahren ausgearbeitet und mit dem Lyocell-Prozeß zur Realität geworden. Das für die Celluloseauflösung verwendete Lösungsmittel - N-Methylmorpholin-N-oxid Monohydrat (NMMNO) erfüllt in weitgehender Annäherung die erwähnten Idealforderungen.

Mitarbeiter des Thüringischen Institutes für Textil- und Kunststoff-Forschung beschäftigen sich seit Anfang der 80-er Jahre mit der Entwicklung einer eigenständigen Verfahrensvariante, dem ALCERU"-Prozeß. Frühzeitig wurde dabei die Aufmerksamkeit auch auf die Herstellung technisch anwendbarer Materialien gerichtet.

Die Celluloseauflösung in einem salzfreien, ungiftigen und in jedem Verhältnis mit Wasser mischbaren Einkomponentenlösungsmittel sowie die augenblickliche Koagulation in Wasser gestatten dabei viele technisch-technologische Varianten.

Variationsmöglichkeiten beim Lyocell-Verfahren

Abbildung 1 gibt einen Überblick über wesentliche Variations- und Verwendungsmöglichkeiten von Cellulose-NMMNO-Lösungen

Variationsmöglichkeiten der Nutzung van
Cellulose-NMMNO-Losungen
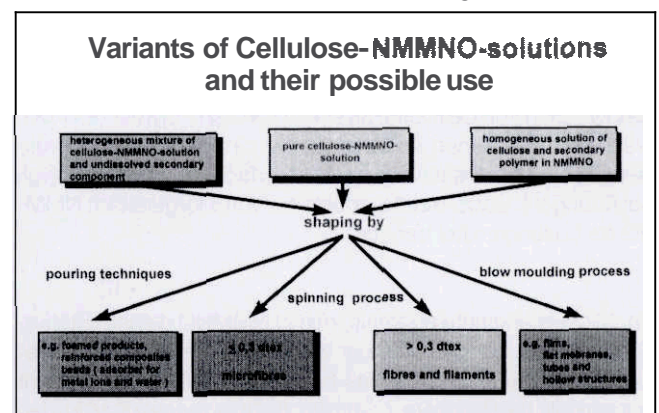
im Vordergrund steht natürlich die Fadenbildung im Düsenloch. In der textilen Verwendung richtet sich das Interesse auf die besonderen Vorzüge der Lyocellfaser:

- hohes Festigkeitsniveau mit Werten zwischen denen von Baumwolle und nahe Polyester,
- Schrumpffreiheit bei Beibehaltung aller Gebrauchsvorteile von Viskosefasern
- Nutzung der spezifischen Naßfribrillierbarkeit zur Erzeugung neuer textiler Oberflächeneffekte.

Hier lassen sich aber auch die verfahrensimmanenten Möglichkeiten zum Erspinnen von im Querschnitt profilierten sowie hohlen Fäden nutzen. Typische Einsatzfälle wären Filtermaterialien mit der großen Oberfläche eines beispielsweise sternförmigen Querschnittes, Hohlfasern für Polstermaterialien und Trennprozesse sowie bändchenförmige Filamente für Verpackungsanwendungen.

Außerdem lassen sich durch das Legieren der Celluloselösungen mit homogen eingemischten Zweitpolymeren Spezialfasern herstellen.

Das Verformen der reinen Celluloselösung oder auch von Blends mit homogen löslichen Zweitpolymeren über Blasverfahren kann für die Herstellung von Schläuchen, Folien und Hohlkörpern genutzt werden.

Gießverfahren ohne Verzug lassen sich für das Tränken von Vliesen für Faserverbundwerkstoffe mit chemisch unveränderter Cellulose als Matrix verwenden.

In Wasser koagulierte Tropfen zeigen nach dem Herauslösen des Lösungsmittels poröse Strukturen und eignen sich beispielsweise beim irreversiblen Einmischen von Chelat-bildenden Polymeren als Adsorbermaterialien für Metallionen.

Die gegebenenfalls durch ein zweites Lösungsmittel auf Erstarrungspunkte ≤ 20 °C eingestellten Celluloselösung läßt sich durch Eintragen in ein zweites, mit NMMNO nicht mischbares Fällungsmittel so verformen, daß sie unter dem Einfluß eines Scherfeldes zu Strömungsfäden verzogen und in dieser Form koaguliert werden kann. Über den Prozeß und die entstehenden Cellulosemikrofasern, die eine Feinheit von ≤ 0,3 dtex, einen Durchmesser < 5 mm und Faserlängen bis zu wenigen Millimetern haben, wurden von B. Riedel / 1 / aus unserer Arbeitsgruppe veröffentlicht.

Lyocell-Erzeugnisse mit eingebauten funktionalen Eigenschaften

Das Mischen von Cellulose mit einem zweiten Polymer über den Weg einer simultanen Auflösung ist ein in verschiedenen wissenschaftlichen Untersuchungen mehrfach geübter Weg (vgl. Abbildung 2). Dabei wurde jedoch nur von Morgenstern NMMNO als Lösungsmittel benutzt.

Ch. Michels erkannte erstmals, daß in NMMNO gelöste Stärke-Cellulose-Mischungen offenbar wegen der schlagartigen Fixierung des Cellulosenetzwerkes beim Fällbadeintritt auch leicht wasserlösliche Polymere praktisch nicht auswaschbar in den Cellulosefaden einbinden / 2 /.
Inzwischen haben wir begonnen, diesen Effekt systematisch unter Verwendung anderer wasserlöslicher Polysaccharide, Polysaccharidderivate sowie wasserlöslicher synthetischer Polymere zu studieren.

Cellulose-Zweitpolymer-Blends aus homogenen Lösungen

Secondary component	solvent	Reference
Polyacrylonitrile	DMF/NO₂ DMAc/LiCl	A.H. Jolan u.a.; J. Appl. Pol. Sci. 22 (1978), 2533 Y. Nishio u.a.; Polymer 28 (1987), 1385 W. Berger u.a.; Mell. Textilber. 72 (1991), 399
Polyvinylalcohol	DMSO/PF DMAC/LiCl DMSO/TEAC	Y. Nishio u.a.; Macromolecules 21 (1988), 1270 Y. Nishio u.a.; Macromolecules 22 (1989), 2547 M. Shibayama u.a.; Polymer 32 (1991), 1010
Polyvinylpyrrolidone	DMSO/PF	J.F. Masson u.a.; Macromolecules 24 (1991), 6670
Poly-4-vinylpyridine	DMSO/PF	J.F. Masson u.a.; Macromolecules 24 (1991), 5914
Polyethylenterephthalate	TFE	N.D. Fields u.a.; J. Polym. Sci. Phys. Ed. 22 (1984), 101
Poly-ε-caprolactone	DMAc/LiCl	Y. Nishio u.a.; J. Polym. Eng. Sci. 30 (1990), 71
Polyamide - 6	DMAc/LiCl	Y. Nishio u.a.; J. Polym. Eng. Sci. 30 (1990), 71
Polyethylenoxide	DMAc/LiCl	Y. Nishio u.a.; Polym. J. 21 (1989), 347
Aramide	DMAc/LiCl	
Poly-2,6-dichlor-1,4-phenylene terephthalic acid amide	NMMNO-MH	B. Morgenstern, Contribution to the 1. Rudolstädter Symposium „ Alternative Celluloseverformung" , September 1994
Polyimide	NMMNO-MH	
Chitosane	TFE	A. Isogai u.a.; Carbohydr. Polym. 19 (1992), 25 M. Hasagawa u.a.; J. Appl. Pol. Sci. 45 (1992), 1873

Abb. 2

Die Abbildung 3 faßt einige der in diesem Zusammenhang verwendete Zweitkomponenten sowie die im Lyocell-Erzeugnis angestrebten funktionalen Eigenschaften zusammen.

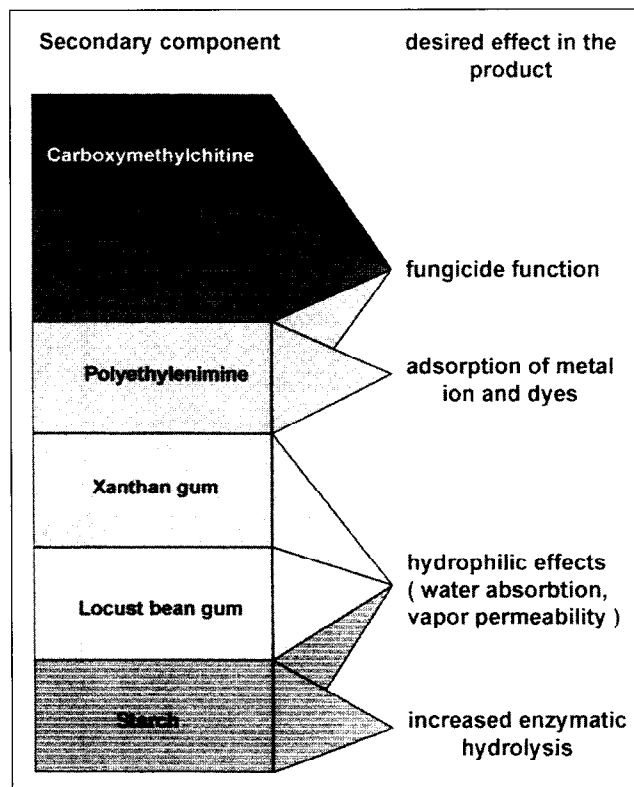## Wasser- und NMMNO-lösliche Blendkomponenten für Cellulose

Abb. 3

Cellulose-Polyelektrolyt-Blends

Carboxymethylchitin (CMCH) und Polyethylenimin (PEI) wurden simultan mit Cellulose homogen in NMMNO gelöst, Chitosan nach Umfällen aus Essigsäure bzw. nach Naßmahlung heterogen in die NMMNO-Lösung eingearbeitet. Die Ergebnisse der textilphysikalischen Prüfung der so hergestellten Fasern sind in Abbildung 4 dargestellt.

Der Abfall der Festigkeitswerte ist bei Zusätzen bis zu 20 % so moderat, daß das Niveau noch über dem von konventionellen Viskosefasern liegt. In Bezug auf die angestrebten, fungiziden Effekte bietet sich ein differenziertes Bild. Nichtpathogene Prüfpilze, die selber Cellulasen bzw. Proteasen produzieren, wachsen verstärkt in Gegenwart der funktionalisierten Cellulosefasern. In die Untersuchungen einbezogene pathogene Pilze, die in ihren Zellwänden weder Cellulose noch Chitin enthalten, werden in ihrem Wachstum gehemmt. Die Inhibierung erfolgt dabei bis unmittelbar an die Phasengrenze des textilen Formkörpers, was dafür spricht, daß selbst das extrem gut wasserlösliche Polyethylenimin auswaschresistent im Blendfaden verankert ist.

Das PEI stabilisiert in besonderem Maß die Spinnsicherheit beim Lyocell-Verfahren. Dies betrifft seine Wirkung auf das System Cellulose / NMMNO / Wasser, indem die den NMMNO-Zerfall katalysierenden Schwermetallkationen durch Chelatkomplexbildung gebunden werden. Darüber hinaus wird eine verbesserte Anfärbbarkeit der Cellulosefasern durch saure Farbstoffe erreicht / 3 /.

Cellulose : Carboxymethylchitosan (CMCH) $A = (100 - A) : A$
Cellulose : Polyethylenimin (PEI) $A = (100 - A) : A$
Cellulose : Chitosan $A = (100 - A) : A$

Textilphysikalisches Eigenschaftsniveau von Cellulose-Polyelektrolyt-Blends
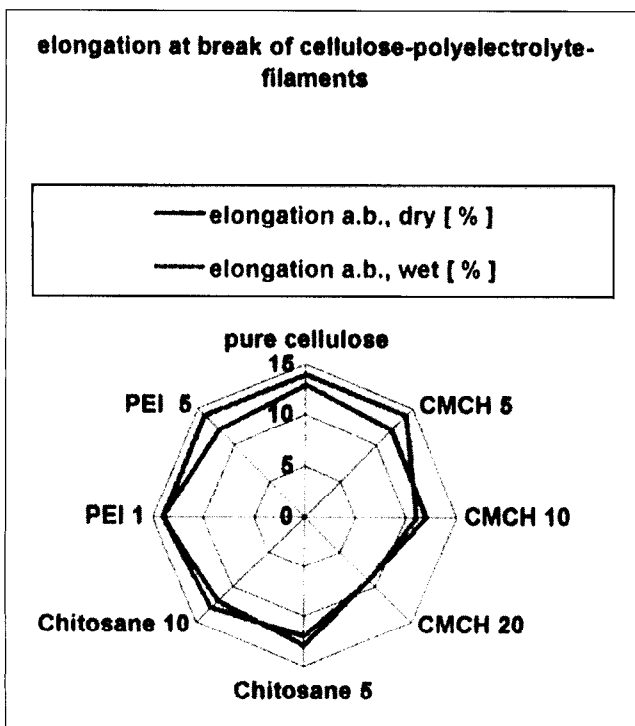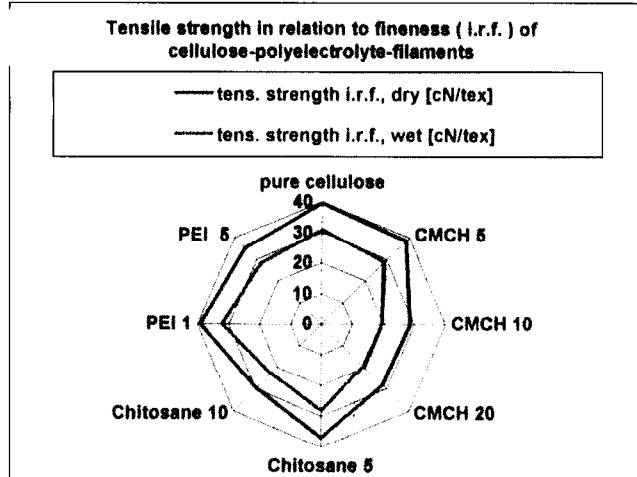

Abb. 4

Abb. 4a

Stärke und Heteropolysaccharide als Zweitkomponenten

Stärke und die genannten Heteropolysaccharide sollten in eine Mischung mit Cellulose mit dem Ziel eingebracht werden, stärker hydrophile Formkörper herstellen zu können. Es wurden verschiedene Mischungen mit am Markt erhältlichen Stärken bzw. Stärkederivaten formuliert und untersucht. Die Ergebnisse in Abbildung 5 zeigen, daß die verschiedenen Cokomponenten einen unterschiedlichen Einfluß auf Reißfestigkeit und Reißdehnung der resultierenden Fasern haben.

Eine zunehmende Stärkekonzentration des gleichen Stärketyps in der Mischung führt entsprechend der zunehmenden Celluloseverdünnung zu einer stetigen Absenkung der Faserfestigkeiten. Wachsende Amylosegehalte nativer Stärken schwächen den festigkeitssenkenden Einfluß ab.

Mischungen unter Verwendung chemisch modifizierter Stärken zeigen ein differenziertes Bild. Die Hydroxypropylierung von amylopektinreichen Typen führt dazu, daß ein auf chemisch nicht veränderte Stärke gleichen Typs bezogen, verbessertes Festigkeitsniveau erreicht wird. Dies sollte nach unserer Auffassung durch eine Verbesserung der Löslichkeit im System NMMNO / Wasser verursacht werden. Bei amylosereichen Typen hat diese chemische Modifizierung dagegen einen umgekehrten Effekt. Hier sollte die im Verlauf der chemischen Modifizierung veränderte Struktur, insbesondere die Erniedrigung der mittleren Molekularmassen ein absinkendes Festigkeitsniveau erklären lassen.

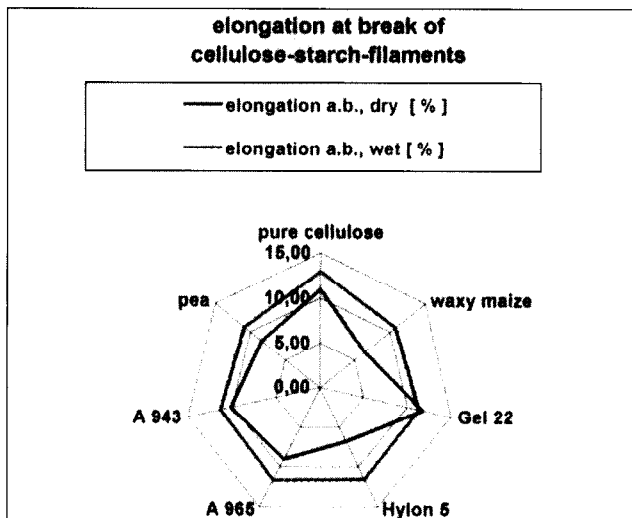**Textilphysikalisches Eigenschaftsniveau verschiedener Cellulose-Stärke-Blends**
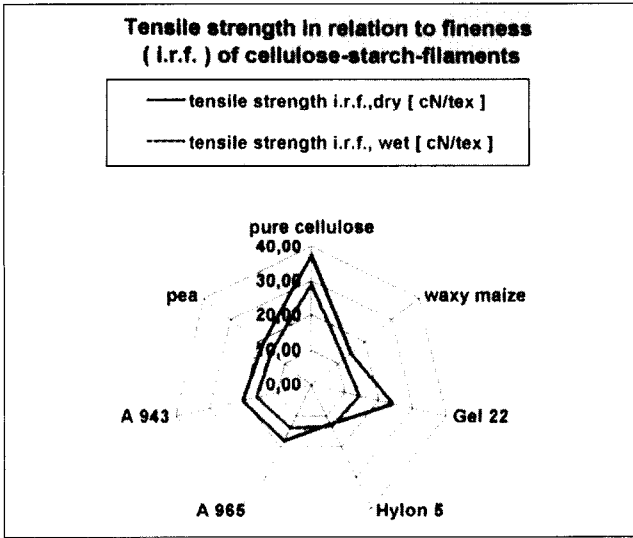

Abb. 5

Tensile strength in relation to fineness (i.r.f.) of cellulose-starch-filaments

——tensile strength i.r.f.,dry [cN/tex]

—— tensile strength i.r.f., wet [cN/tex]

pure cellulose

Abb. 5a

Wachsmais - Wachsmaisstärke (0 % Amylose)
Gel 22 - Maisstärke, hydroxypropyliert (45)
Hylon 5 - Maisstärke (45)
A 965 - Maisstärke, hydroxypropyliert (65)
A 943 - Maisstärke (65)
Markerbse - Markerbsenstärke (>95)

Betrachtet man den Einfluß der unterschiedlichen Blendkomponenten auf das Wasserrückhaltevermögen (vgl. Abbildung 6) als Bewertungsmaßstab sich ändernder hydrophiler Eigenschaften, so findet man erwartungsgemäß eine monoton steigende Abhängigkeit zwischen Amylopektingehalt und Erhöhung des Wasserrückhaltevermögens der resultierenden Cellulose-Stärke-Filamente.
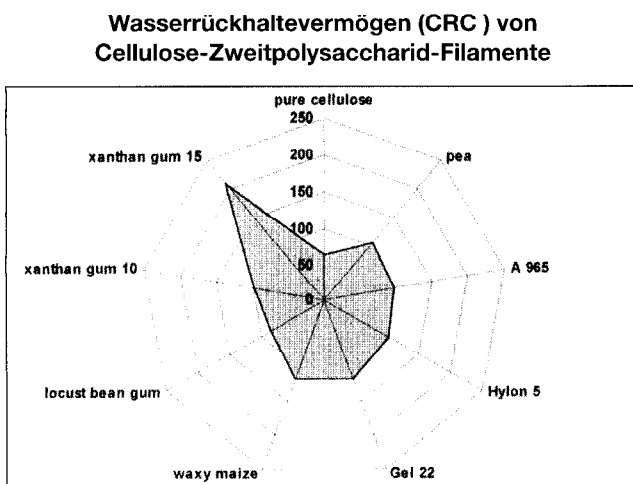
Wasserrückhaltevermögen (CRC) von Cellulose-Zweitpolysaccharid-Filamente

Abb. 6

Cellulose : Stärken = 70 : 30
Cellulose : Johannisbrotkernmehl (LBG) 10 = 90 : 10
Cellulose : Xanthan (XG) 10 = 90 : 10
Cellulose : XG 15 = 85 : 15

Die graduelle Abstufung in Abhängigkeit von der Modifizierung fällt bei niedrigeren Amylosegehalten entsprechend stärker ins Gewicht. Auch dieser Trend läßt den Einfluß einer verbesserten relativen Löslichkeit modifizierter amylosearmer Typen erkennen. Bemerkenswert ist das vergleichsweise hohe textilphysikalische Niveau bei Cellulosefasern mit Heteropolysaccharidzusätzen.

Beim Einmischen von bis zu 10 % kommt es nur zu einem moderaten Abfall der Reißkraftwerte entsprechender Filamente (Abbildung 7).

Textilphysikalisches Eigenschaftsniveau verschiedener Cellulose-Heteropolysaccharid-Blends im Vergleich zu Gel 22 und Wachsmais
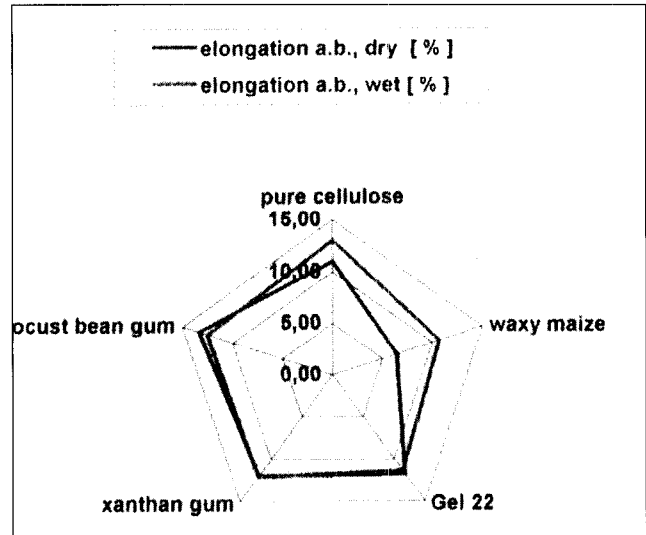
——elongation a.b., dry [%]

——elongation a.b., wet [%]

pure cellulose

Abb. 7

——tens. strength i.r.f.,dry [cN/tex]
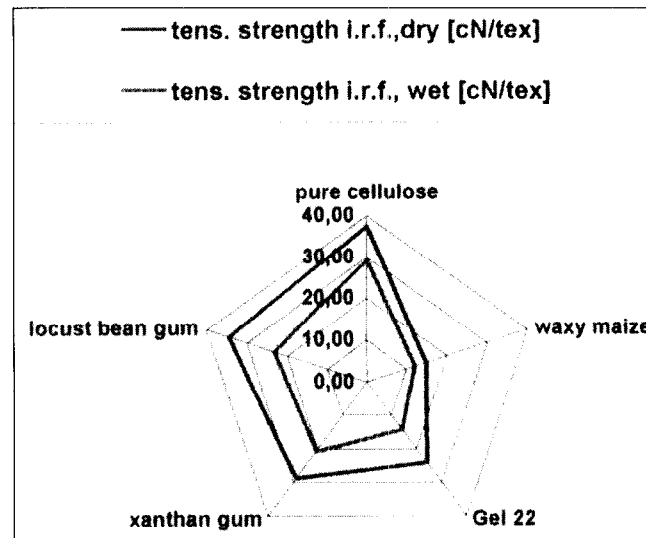
——tens. strength i.r.f., wet [cN/tex]

pure cellulose

Abb. 7a

Der gleichzeitige Anstieg der Dehnung deutet darauf hin, daß die höheren Molekulargewichte Ursache des Effektes sein könnten. Das Wasserrückhaltevermögen (CRC) von Filamenten steigt bei der Zumischung von bis zu 15 % Xanthan (XG) auf ca. 220 %.

Leitfähige Cellulosefilamente

Interessante Produkteigenschaften lassen sich auch beim Einmischen von in der Cellulose-NMMNO-Lösung unlösliche Substanzen erzielen.

Die hohe Verdünnung der Cellulose in der NMMNO-Lösung erlaubt, bezogen auf Cellulose, Zusätze bis zu 200 und mehr Prozent, ohne wesentliche Verschlechterung des Spinnverhaltens.

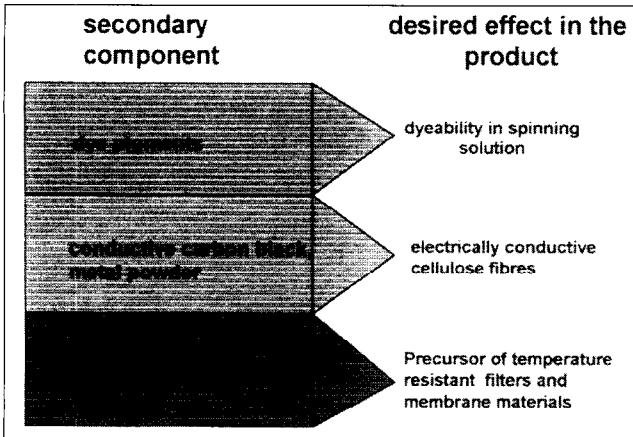## Wasser- und NMMNO-unlösliche Zweitkomponenten

Abb. 8

Die elektrischen sowie textilphysikalischen Eigenschaften entsprechender, Ruß gefüllter Filamente zeigt Abbildung 9. Die mittlere Teilchengröße der Rußpartikel betrug ca. 20 nm. Werte für den spezifischen Ohm'schen Widerstand der so modifizierten Cellulosefäden lassen erkennen, daß hier eine anwendungstechnisch vielseitige Kombination von textiler Verarbeitung und elektrischer Leitfähigkeit erreicht wird.

Eigenschaften elektrisch leitfähiger Cellulosefilamente
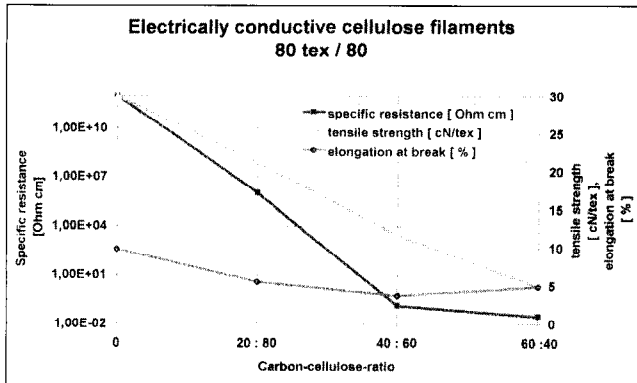

Abb. 9

Eine interessante Modifizierung des Prozesses ergibt sich nach den Arbeiten von D. Vorbach aus unserer Arbeitsgruppe durch die Anwendung des Bikomponenten-Spinnens / 4 /.

Dabei werden Kern-Mantel-Strukturen erhalten, die im Fadeninneren eine Seele aus hoch leitfähiger Cellulose sowie einen cellulosischen Mantel, der als mechanisch tragende Schicht eine hohe Festigkeit aufweist, besitzen (Abbildung 10). Eine Umkehrung der Anordnung ist ebenso möglich.

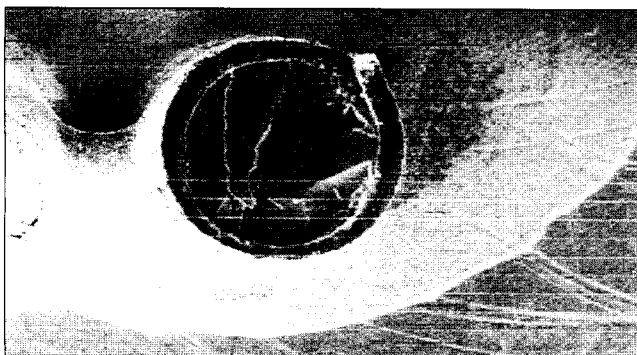## Kern-Mantel-Struktur eines leitfähigen Cellulosefadens

Abb. 10

Eine weitere Eigenschaft der mit Rußanteilen im Bereich der Perkolationsschwelle gefüllten Cellulosefilamente folgt aus der Empfindlichkeit des spezifischen Widerstandes gegenüber quellenden Substanzen. Die Abhängigkeit des Widerstandes von der relativen Luftfeuchte bei verschiedenen Umgebungstemperaturen zeigt die Abbildung 11.

Es wird deutlich, daß mit zunehmender Luftfeuchte die elektrische Leitfähigkeit sinkt. Dieser Effekt beruht offenbar auf der Trennung der Kohlenstoffpartikel infolge Aufquellung durch die aufgenommene Feuchte. Die Meßwerte weisen eine geringe Temperaturabhängigkeit bei gegebener Luftfeuchte sowie eine gute Reproduzierbarkeit nach vielen Meßzyklen auf. Untersuchungen konnten nachweisen, daß ein ähnlicher Effekt bei Zug- bzw. Druckbeanspruchung induziert werden kann.

Widerstand von Feuchtesensoren in Abhängigkeit von der relativen Luftfeuchte
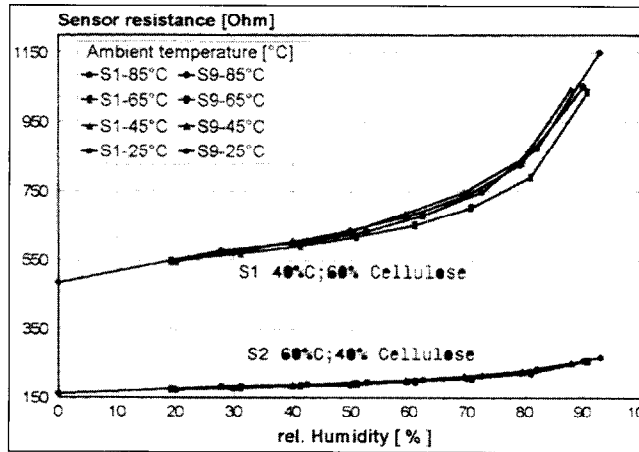
Abb. 11

Die Sensorwirkung erstreckt sich aber auch auf die Konzentrationsbestimmung in anderen organischen Flüssigkeiten (vgl. Abbildung 12).

Widerstandsänderung eines Sensorfadens in der Dampf- bzw. Flüssigkeitsphase bei verschiedenen Lösungsmitteln

Abb. 12

Messungen bei 25 °C und Normaldruck bewiesen einen zwar abgeschwächte aber deutlich meßbare Konzentrationsabhängigkeit. Die Höhe der Widerstandsänderung beim Übergang gasförmig zu flüssig korreliert mit der Polarität der Flüssigkeit.

Literatur

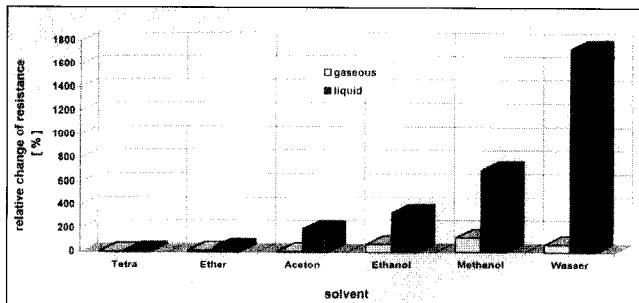1 B. Riedel;"Neue Wege zur Herstellung von Cellulosemikrofaservliesen", Vortrag zur 35. Internationalen Chemiefasertagung Dornbirn, 1996

2 Ch. Michels; Fachlicher Schlußbericht zum Vorhaben "Strukturmodifizierte Cellulosefaserstoffe", FKZ: 5 / 93

3 Ch. Michels; Fachlicher Schlußbericht zum Vorhaben "Biologisch aktive Cellulosefaserstoffe", FKZ: 275 / 94

4 D. Vorbach und E. Taeger; "Eigenschaften von kohlenstoffgefüllten Cellulosefasern", Vortrag auf dem 8. Techtextil-Symposium Frankfurt/Main, Mai 1997

TECHNICAL APPLICATIONS OF COURTAULDS LYOCELL®

Calvin Woodings, Courtaulds Lyocell, UK

Introduction

Cellulosic fibres made via the direct dissolution of wood-pulp in an organic solvent are now known generically as lyocell fibres. The development of the lyocell process has been described in many technical papers and articles in the 10 years since Courtaulds built the plant from which the first products were commercialised. Fibre and fabric properties, as they apply to the Tencel® brand of fashion apparel products were discussed. Courtaulds Lyocell® (abbreviated to Cly in the rest of this paper) is the brand covering fibre types developed for performance products, targeting technical markets such as nonwovens, special papers and technical textiles. This paper reviews the development of applications in each of these sectors.

Fibre Attributes

Fibre properties of relevance to technical applications can be summarised as follows. Where comparisons are implied they are made against other cellulosic fibres:

- Higher Tensile strength especially when wet
- Lower extensibility wet and dry
- Higher modulus wet and dry
- Higher radial swelling on wetting
- No longitudinal swelling or shrinkage
- Higher water imbibition than cotton
- Can be fibrillated to micro-fibres
- Fibrillated fibres self-bond (100% Cly papers possible)
- More resistant to wet-collapse (higher total absorbencies possible)
- Dyes readily
- Does not melt and is more durable at high temperature
- Can be oxidised and carbonised
- Readily biodegradable aerobically and anaerobically
- Blends easily with synthetics and cotton

Applications in Technical Textiles

The sector has characteristics which differentiate it from the other textile sectors :

- New fibres are judged on technical merit.
- Cost/performance ratio is the key selling point.
- New product lead times tend to be long.
- Changes of raw-material are not undertaken lightly.
- Markets are less volatile than fashion markets.
- Barriers to entry can be high, but so can barriers to exit.
- They are driven by technological change, not fashion.

Cly confers a soft silky handle with exceptional drape and fluidity when finished for apparel applications by the methods recommended for the Tencel® branding programme. These characteristics are not valued in most technical textiles. Toughness and durability are often more desirable, and these attributes are not often associated with cellulosic fibres.
Cotton is however widely used in technical textiles, the required

end-use performance being defined by the details of the fabric construction and finishing processes. Cotton, alone or blended with synthetics, appears in many performance fabrics from belting to bandages, and workwear to waterproofs. Cly, made in precise deniers and long staple-lengths can outperform long-fibre cottons to make finer, stronger, more absorbent yarns alone or in blends. Fabrics therefrom are inherently more comfortable to wear and when correctly constructed and finished they also demonstrate higher tear resistance, freedom from progressive shrinkage, better appearance retention, and better softness retention through repeated industrial laundering.

While the abrasion resistance of any cellulosic fibre is poor compared with the synthetics, and while Cly's fibrillation tendency can cause laundry-frosting of improperly finished fabrics, blends with synthetics and the correct resin treatment can overcome these apparent defects.

The development efforts of the last few years are now yielding some commercial products. Many more are still invisible and "in the pipeline".

- In the high performance workwear sector a joint development with Carrington Career and Workwear has led to the launch this Autumn of the "Fury" protective suit for the North Sea oil market. This capitalises on Cly's advantages over cotton as indicated above, and additionally illustrates the favourable interaction between the new fibre and the established flame-resist finishing techniques.
- In the ladies workwear sector, a joint development with Klopman International has led to a new range of garments for nurses which exhibits some of the characteristics of the Tencel® fashion fabrics while demonstrating the sort of appearance retention and durability required to maximise longevity in industrial laundering.
- In sewing threads, the high strength and high-speed sewability of Cly yarns coupled with enzyme resistance and a dye uptake comparable to the base fabrics have led several producers to introduce Cly threads to their range for use in cellulosic garments to be dyed and/or finished in garment form.
- In coating bases, Cly's high strength and modulus linked to the ability to make smooth surfaced fabrics which bond well to the coatings have led several producers to introduce Cly fabrics to their range.

Applications in Nonwovens and Special Papers

Cly, being made as a continuous tow which is then mechanically crimped and cut, gives a precision crimped staple fibre for easy opening, carding and blending. It has been converted into a wide range of nonwoven products and papers which exhibit the following general benefits. Here any implied comparisons are against viscose fibres processed in a comparable manner.

- Twice the dry strength
- Nearly three times the wet strength
- Higher wet-cohesion especially when fibrillated
- Stronger bonding with latexes
- Stronger thermal bonding with synthetics
- Lighter fabrics/papers possible
- Reduced shrinkage in drying/curing
- Better dimensional stability
- Higher absorbency (rate and capacity) especially when fibrillated

Laying processes

Dealing with the potential for Cly in the numerous conversion routes:

Carding

Cly is easy to blend, easy to open and gives regular webs. Cohesion levels can be manipulated by adjustments to fibre finish, crimp and moisture content during tow production. Unlike viscose, the tow is converted to staple after drying and the fibre presentation in the bale is therefore similar to synthetic fibres.

Carding speeds possible with Cly have been increased dramatically by moving away from the original finishes which were designed for viscose. On the main card in Courtaulds Fibre Processing Unit the fibre can now be run at 200 metres/minute, and it has exceeded 140 metres/min on full size nonwoven cards. Further advances appear possible.

Wet Laying/Papermaking

The parallel tow presentation gives more precise short-cutting and therefore cleaner wet-laid webs. The higher modulus gives better dispersion at longer fibre length and the higher wet cohesion gives easier wet-web transfers prior to bonding. The lower water imbibition gives easier drying and the high wet stability gives lower shrinkage losses. If necessary, the average fibre size can be reduced by fibrillation. This can be achieved by vigorous mixing of a Cly slurry in a hydrapulper. After fibrillation the stock can be processed as a paper stock, with care being taken to avoid agglomeration of the finer fibres.

Cly can also be fibrillated in refiners or beaters to give fine round fibrils of much smaller diameters than obtainable from natural pulps. The resulting papers have an unexpectedly beneficial opacity/strength relationship, an unusually high porosity, and very good particle retention in filtration tests. While tensile properties are usually lower than those obtainable from the best pulps, tear strengths can be enhanced by the inclusion of Cly.

A new fast-fibrillating version of Cly has been introduced for companies needing the highest levels of fibrillation in the shortest possible time.

Pulp Air-laying

The Kroyer and Dan-web air-layers being used to make wet-wipes, subfacings and absorbent cores work particularly well with short-cut Cly. Cly enhances fluid wicking and improves the strength and texture of these majority-woodpulp products. Multi-head machines give the opportunity to utilise the Cly predominantly in the surface layers to maximise the softness of wet-wipes.

Fibre Air-laying

The entanglement-free low web cohesion obtainable with the tow-based Cly production route are particularly advantageous in the newer long-fibre air-layers. These systems appear ideal for making the truly random webs needed by the new generation of entanglement lines now being installed.

Bonding Processes

Hydroentanglement (HE)

Cly gives stronger, less extensible and more stable fabrics. As with the other bonding systems these features can be used directly to improve the fabric, or indirectly to reduce costs by basis weight reduction. Many low pressure systems use some secondary bonding (latex or thermal) to achieve improved wet strengths. When Cly is used, this additional bonding can be reduced or eliminated. In aperturing processes very precise patterning is possible giving more textile-like appearance than is possible with other fibres.

HE trialists are inclined to treat Cly with the highest possible water pressure to see if their machine can fibrillate it. Most machines making lightweight fabrics can't, but the high pressures give all the expected strength benefits. They also give firm fabrics with excellent surface integrity and a rather less "soft" handle than viscose. If softness is required, the water pressures need to be reduced below those used for viscose until the fabric strengths are the same as viscose. If a really silky-touch product is required, and CD extensibility is not critical, a more parallel laid web will give best results. If a silky appearance is required as well, the lustrous bright form of the fibre will give it.

Raw material cost-savings can be obtained by reducing the basis weight. It has been possible to halve the basis weight c.f. viscose when seeking to make the lightest possible covering material with adequate strength.

The strength benefits obtainable from Cly use in low pressure HE are enhanced at higher pressures up to the point where fibrillation occurs. At higher pressures still the strength plateaus and ultimately declines, while the fabric opacity and firmness increases dramatically. The silky aesthetics are lost, but the toughness of the fabrics makes semi-durable and durable end-uses possible. High pressure hydroentanglement of Cly fibre has enabled the production of nonwovens which are stronger than the equivalent weight of woven cotton. Effects on HE water filters are minimal.

For applications where the fibrillation is essential, a new fast fibrillation variant of Cly has been introduced. This yields stiffer, less extensible more cotton-like nonwovens with higher absorbencies - both rate and capacity. At the highest levels of fibrillation the fabric tensile strengths are reduced.

Finally there is an excellent fit between Cly's attributes and those HE machines which use wet-lay web forming. Here the ability to disperse the easily entangled Cly fibres at longer lengths (up to 15 mm) than other cellulosics enables this wet route to outperform dry routes using 38 mm viscose.

Needling

In needlefelt technology Cly fibre strengths are efficiently translated into fabric properties with three-fold wet strength advantage over viscose fabrics. In addition to attaining higher strength specifications this performance can be used to make lighter weight products or reduce the levels of non-absorbing binding material which can impair fabric handle.

Cly tends to form more open and bulkier needled structures than viscose. Coupled with its higher resiliency this leads to increased total absorbent capacity. 1.7 dtex lyocell fabrics are commercially feasible (high fibre strength) and show only half the wet collapse of 3.3 dtex rayon structures yielding the expected increase in total capacity.

Latex Bonding

Cly gives much stronger fabrics than viscose especially in the wet state. This feature can be used directly to meet tougher customer specifications, or indirectly to reduce raw material usage. (Lower fabric weights through less fibre and/or less binder.) Cly bonded with half the latex level needed for viscose gives much improved fabric aesthetics and absorbency, allowing this venerable but still important nonwoven route to enter new territory. Cly also gives low-shrink, high stability fabrics during drying/curing which make it possible for the latex bonder to increase the area productivity of his lines by 10-15%.

Thermal Bonding

Cly bonds better to some types of polypropylene and gives stronger thermal blends than viscose especially in the wet state. This allows the thermal bonder to incorporate a cellulosic at higher concentrations, or to make 50/50 blends at lower basis weights.

These features will be particularly relevant if coverstock producers ever attempt to increase the skin-comfort characteristics of their currently all-synthetic product. Coverstocks can be made by plain-bonding fabrics containing 70% Cly. These materials used over the latest diaper or sanpro cores give adequate surface dryness (no compromise in wet-comfort) while substantially improving dry comfort.

Papermaking (Hydrogen Bonding)

Fibrillation of Cly can be achieved in many ways (dyeing, finishing, suedeing, hydroentanglement, brushing, ultrasonic treatment), but it is in the papermaking processes that fibrillation can be carried out most efficiently and controllably.

The ability to fibrillate Cly in beating, refining or hydrapulping allows the manipulation of sheet properties. For example the following can be altered dramatically by fibrillation:

* handle/aesthetics
* barrier properties
* opacity/cover
* moisture absorbency/transport
* tear and tensile strength
* air permeability
* particle capture

The last two properties are particularly important in applications such as filtration.

Paper strength depends upon hydrogen bonding, which increases as more fibrillation is generated. Paper tensile strength improves in proportion, but tear strength goes through an optimum at moderate levels of fibrillation. Air resistance of the sheet goes up proportionately, but the permeability of lyocell sheets is still higher than that of equivalent woodpulp papers, due to the fine circular nature of the lyocell fibrils.

Pore size is also affected - increased fibrillation giving smaller average pores. It is therefore possible to manipulate the mean pore and distribution of pore sizes by controlling the level of fi-

brillation produced. Cly papers exhibit a similar pore size/permeability relationship to the more costly microglass fibre which is commonly used in filtration papers. Pulp sheets show lower permeability.

Applications in Nonwoven/Paper Products

Cly, at current prices, is not an obvious substitute for any of the commodity raw materials used in this sector and most of the successes to date have arisen from developing new fabrics which utilise the strengths of Cly to best effect.

To date the wet processes (hydroentanglement, wet-laid nonwovens and special papers) consume the majority of sales but needlefelting applications are "on the move".

The following table summarises the key applications along with some information as to why Cly has been preferred over other fibres:

Application	Benefit
Wipes - Domestic - Food service - Paint Shop - Printing Industry - Clean Room	Improved strength for durability Lower binder levels for increased absorbency. Increased wet resiliency. Enhanced wipe to dryness from fibrillation. Increased (cellulosic) chemical resistance. Low Lint Purity
Artificial Wash Leathers	Leather-like durability and wet texture. Smear-free wiping. (Fibrillation effects)
Wet wipes	High CD wet strength. Softness and bulk. Odour-free.
Coating substrates	High strength. Absorbency and comfort. Microfibre surface.
Medical swabs and gauzes	Cleaner processing (than cotton.) Higher strength (including post-sterilisation). Low linting. Clean, precise aperturing. Aesthetic control. FDA 510K.
Filters - Medical - automotive - HEPA/ULPA - Food/Drink - Smoke (Cigarette) - Vacuum bag	Sub-micron fibril sizes Particle Retention Permeability Purity/Biodegradability Tar retention Efficiency
Biocomposites	High Strength High Modulus Biodegradability Tow for pultrusion
Battery Separators	Purity. Burst Strength Electrolyte absorbency. Extended battery lifetimes.
Durable clothing	Strength. Attractive aesthetics. Dyeable.
Disposable clothing	Strength and comfort Clean aperturing

Future Possibilities

Developments have to date been made against a background of limited availability of fibre from the US plants which have been fully stretched meeting the fashion-led demand for the Tencel® branded variants. The special deniers, lengths and finishes required by many technical applications have only been available from the original Grimsby line - now 10 years old.

This year we are seeing a major increase in European capacity with both Courtaulds and Lenzing starting up the first of several large lines using the latest lyocell technology. Availability problems are behind us, economies of scale begin to take effect and the development of the technical market can move into higher gear.

Courtaulds Lyocell® will move from its premium priced "new product - new market" positioning into the mainstream where it will begin to make inroads into markets currently dominated by other types of fibre. Its status as the best available technology for converting abundant and renewable natural cellulose into fibre will ensure its place amongst the leading raw materials of the next century.

C R Woodings

Copies of the slides used to illustrate this talk can be obtained by faxing the author at Courtaulds Fibres, Coventry UK. - Fax number +44 (0)1203 583455 - and quoting Dornbirn 97.

THE APPLICATION OF CELLULOSIC FIBERS IN NONWOVENS

G. Sames, Lenzing AG, Austria

The nonwoven industry undergoes a turbulent phase. Therefore, the steady increase of the market volume implies an increase of requirements in several production technologies. Furthermore, new technologies on the one hand and innovations of existing technologies on the other hand have been developed to marketable installations. In the middle of this century the process that led to marketability quite often lasted ten to twenty years. Nowadays we are used to time periods of up to five years,

some branches of industry have reached cycles of even less than a year.
While once the spunlace technology was thought to be rather difficult to applicate economically, the last years manifestly taught us that this process has found its fixed position among all the other technologies. Moreover, a growing necessity of enhancing capacities can be noticed for methods that have already been on the market for a considerable period of time.

Cellulosic Fibers

The resulting capacities as well as the performance of end-products are eminently influenced by the choice of raw materials. The following report focusses on cellulosic staple fibers and their blends with polypropylene and polyester in nonwovens. These three are the main raw materials used in this application.

All cellulosic fibers have a macromolecular chain, with cellulose as an elementary common basis (Fig.2). Although consisting of the same chemical core, their morphological and physical properties differ characteristically.

Viscose in Drylaid Nonwovens (Western Europe)

Source: Edana

45,2   46,6   49,2   52,5
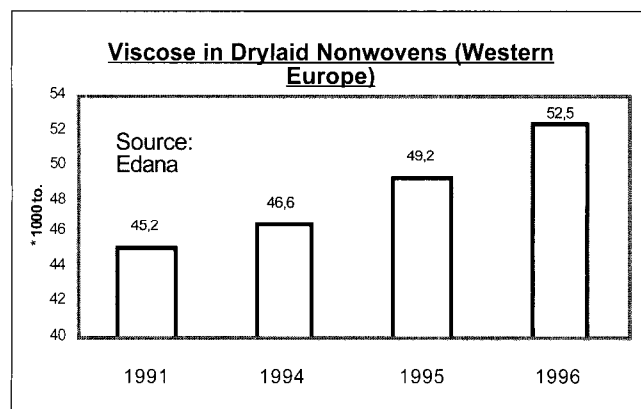
1991   1994   1995   1996

*1000 to.

Fig. 1

Viscose fibers have been in use for a long period of time in more or less classical products. Modal fibers, providing higher tenacities, mainly in wet conditions, equally offer a broad field of applications. The most recent products in this context are lyocell fibers, which have attracted a lot of interest so far.

Fiber modifications in physico-mechanical or chemical ways make it possible to implement additional advantagable properties in the fiber, such as increased hydrophilicity. Such fibers have already been tested and show the same processabilities as viscose.

Hydroentangling Cellulosic Fibers

At present hydroentanglement is the latest technology in this field. By their nature cellulosic fibers hold a steady place in the fields of hygienic, body care as well as medical applications. This fact has been even more manifested through the optional use of totally chlorine free bleached Viscose by Lenzing. Cellulosic fibres

do not cause any irritation of the skin or mucous membranes. Besides that, another big advantage can be seen in its complete biodegradability. The biological degradation is due to an aerobic deterioration of the organic fiber structure caused by natural microorganisms, such as bacteria, yeasts or moulds. The fiber is decomposed into the components CO_2 and H_2O.

- Parameters

We will now have a closer look at carded, cross-lapped and spunlaced nonwovens of the mentioned fiber types - all with a 40 mm cut length -namely viscose, modal, lyocell and a viscose/polypropylene blend. Hydrophilic C is a chemically modified type of viscose fiber (Tab.1). Web weights of 40, 60, 80 and 120 g/m^2 include the biggest part of industrial applications.

Glucose   Cellobiose

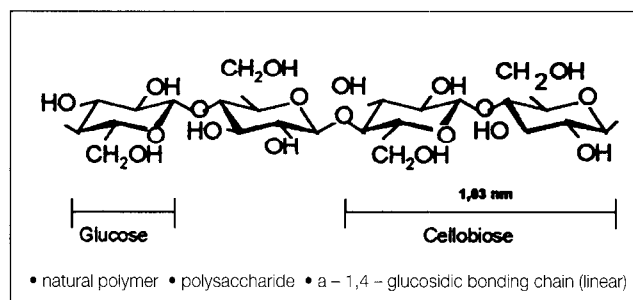• natural polymer • polysaccharide • a – 1,4 – glucosidic bonding chain (linear)

Fig. 2

Fiber types used
Viscose by Lenzing 1,7 dtex/40 mm. . .1,3 dtex/40 mm
Modal by Lenzing 1,3 dtex/40 mm. . .1,0 dtex/40 mm
Lyocell by Lenzing 1,7 dtex/40 mm. . .1,3 dtex/40 mm

70% Viscose 1,7 dtex/40 mm – 30% PP 1,7 dtex/40 mm
50% Viscose 1,7 dtex/40 mm – 50% PP 1,7 dtex/40 mm
Hydrophilic C 1,7 dtex/40 mm

Tab. 1

- Tenacities

The sum of tenacities in machine- and cross direction reveals the highest values for lyocell. The viscose types and Hydrophilic C show the lowest tenacities. The modal types and polypropylene blends range between these groups. This is true for both conditioned and wet state results.
Additionally, higher tenacities can be evaluated for finer fiber titers. Almost every single material provides a higher tensile strength with the finer titer (Fig.3).
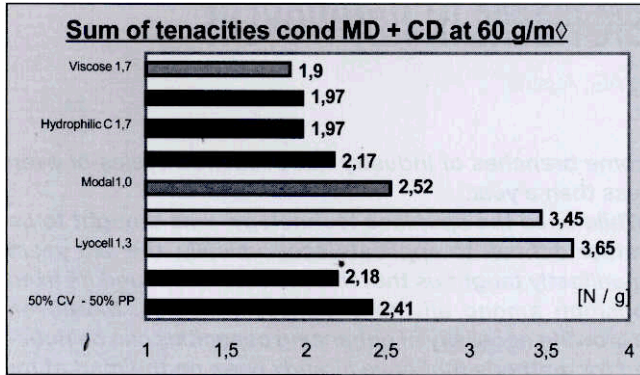
Sum of tenacities cond MD + CD at 60 g/m◊

Material	Value
Viscose 1,7	1,9
	1,97
Hydrophilic C 1,7	1,97
	2,17
Modal1,0	2,52
	3,45
Lyocell 1,3	3,65
	2,18
50% CV - 50% PP	2,41

[N / g]

Fig. 3

- Elongations

Comparing all elongations, the viscose-polypropylene blends clearly illustrate the highest values in cross- and m machine direction, followed by the two viscose samples. Elongations for lyocell and modal are the lowest ones (Fig.4).
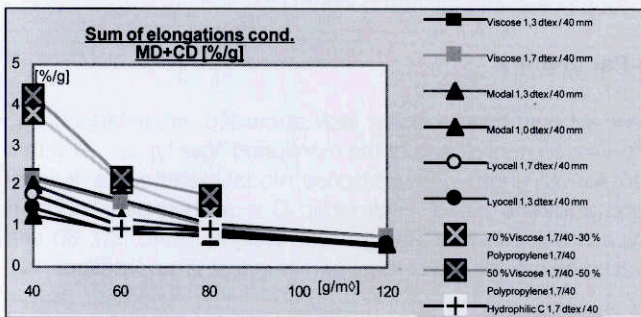
Sum of elongations cond. MD+CD [%/g]

Legend:
- Viscose 1,3 dtex / 40 mm
- Viscose 1,7 dtex / 40 mm
- Modal 1,3 dtex / 40 mm
- Modal 1,0 dtex / 40 mm
- Lyocell 1,7 dtex / 40 mm
- Lyocell 1,3 dtex / 40 mm
- 70 % Viscose 1,7/40 -30 % Polypropylene 1,7/40
- 50 % Viscose 1,7/40 -50 % Polypropylene 1,7/40
- Hydrophilic C 1,7 dtex / 40

Fig. 4

- Densities, air permeability

Due to the lower density of polypropylene as a raw material (0,90 g/cm³), the lowest densities can be found among the blends containing polypropylene, whereas the rest of the materials are on a rather uniform level. The densitiy of cellulose is 1,53 g/cm³. Hence, the polypropylene blends show the highest air permeabilities.

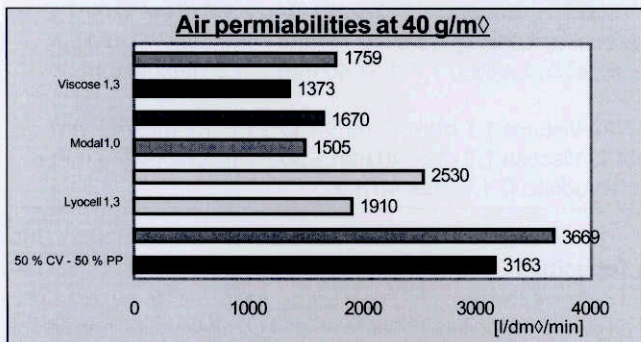Moreover, lower air permeabilities have been evaluated for finer fiber titers (Fig.5).

Air permiabilities at 40 g/m◊

Material	Value
Viscose 1,3	1759
	1373
	1670
Modal1,0	1505
	2530
Lyocell 1,3	1910
	3669
50 % CV - 50 % PP	3163

[l/dm◊/min]

Fig. 5

- Varying energy inputs

Energy inputs have been varied between 0,5 and 1,1 kW.h/kg. This has resulted in increasing various properties without actually changing the relations between the single testing materials.

To conclude, the results merely undergo a parallel shift within certain ranges of energy inputs when changing these.

- Samples vs. marketable products

The samples have been compared to market-established ones in order to determine their possible marketabilities - in terms of sums of tenacities cond. in MD and CD.
Both end-products and samples have been split into two groups. Firstly, wipes based on 100 % cellulosic fibers have been compared to each other. The samples' properties are equivalent to those of the market products (Fig.6).

Comparison of samples of 100 % cellulosic fibre with marketable products in 100 % Viscose

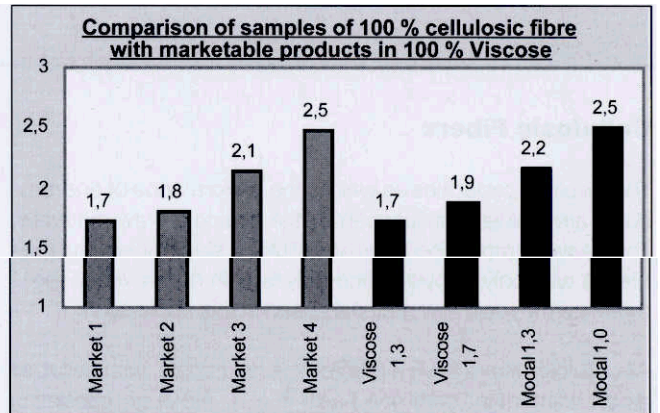| Sample	Value
Market 1	1,7
Market 2	1,8
Market 3	2,1
Market 4	2,5
Viscose 1,3	1,7
Viscose 1,7	1,9
Modal 1,3	2,2
Modal 1,0	2,5

Fig. 6

Secondly, two samples made of viscose/polypropylene blends have been compared to five different endproducts made out of blends with cotton or viscose and polypropylene or polyester. In this case likewise, the properties of the investigated samples do not differ from those of market brands (Fig.7).
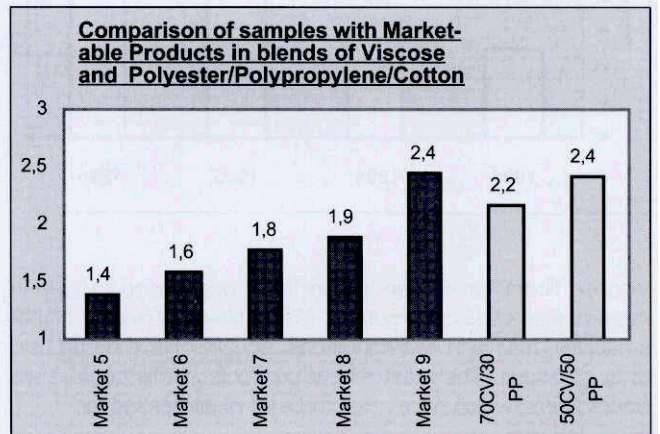
Comparison of samples with Marketable Products in blends of Viscose and Polyester/Polypropylene/Cotton

Sample	Value
Market 5	1,4
Market 6	1,6
Market 7	1,8
Market 8	1,9
Market 9	2,4
70CV/30 PP	2,2
50CV/50 PP	2,4

Fig. 7

Binary Blends - Spunlacing vs. Needlepunching

- Parameters

Two different nonwoven types have been produced on the ternary basis of
- Lyocell 1,7 dtex/ 40 mm
- Viscose 1,7 dtex/ 40 mm,
- Polyester 1,7 dtex/ 40 mm
out of a series of bilateral blends as well as of these three single materials.

Web forming of both has been done on the same equipment - with different parameters - from blending to carding and cross-lapping. From then on two different bonding technologies have been used, hydroentanglement and needlepunching.

- Purpose

Although needlepunched products hardly ever find their way into a medical or hygienic end-use, a comparison between hydroentangled and needlepunched samples is of highest interest. However, there is no comparison of the absolute outcomes, but of the properties depending on the various blending types. Firstly, we look at two different bonding techniques and therefore should not get these results mixed up.
Secondly, as marketable samples have been produced, the weights of the two sample types had to be completely different. Moreover, one can hardly imagine any realistic end-use for needlepunched samples below around 100 g/m². They have therefore been produced at a weight of 160 g/m².
Spunlaced samples, on the other hand, need to be a lot lighter. Thus, they have been manufactured at 45 g/m².
The purpose is to find out the relationship between all the single blends depending on the different bonding techniques used. Therefore, the tenacity in conditioned and wet state, the thickness, the density and the air permeability of the numerous above mentioned blends have been investigated.

- Hydroentangled webs

- Tenacities cond.

The highest tenacities in conditioned state have been evident for the Polyester samples followed by the Lyocell samples.
The lowest tenacities cond. have been evaluated for 100 % viscose samples. The more Lyocell or Polyester has been added, the more the tenacity values have increased.

- Tenacities wet

Generally, wet tenacities show no major changes considering tenacities cond. The results are quite comparable (Fig.8).
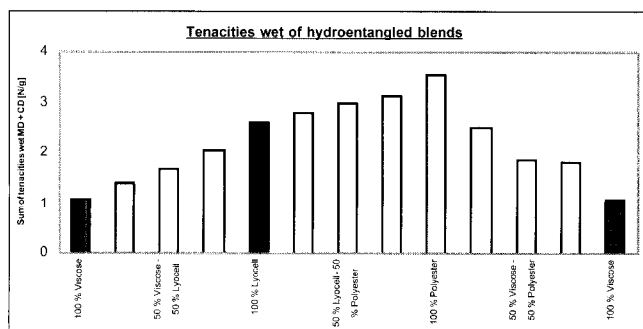

Fig. 8

- Thickness, density, air permeability

As expected, the samples containing polyester manifest the lowest densities, therefore imply the highest thicknesses. On the other hand, the lowest thicknesses and highest densities can be found among the viscose containing samples. Concerning hydroentangling, viscose is the most compressible among these three materials. This leads to thinner end-products than those made of polyester.

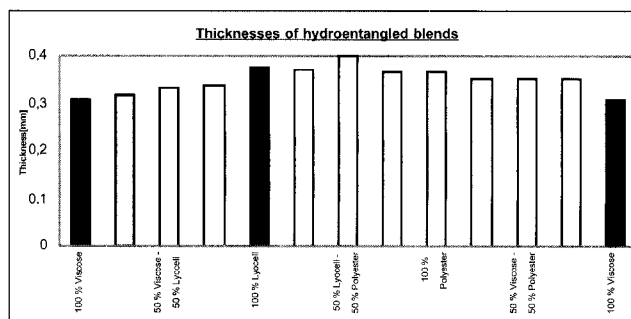The air permeability is higher, the more the amount of polyester is increased (Fig.9).

Fig. 9

- Needlepunched webs

- Tenacities cond.

The properties of the needlepunched samples can be characterized less continuously than those of the hydroentangled ones. Again, the highest tenacity in conditioned state appears when 100 % polyester is used. The differences between the strongest and the weakest materials are much lower than among the spunlaced samples. The tenacity cond. of polyester decreases by 51 % from 3,9 N/g for hydroentanglement to 1,9 N/g for needlepunching, that of viscose, however, decreases only by 6% from 1,6 N/g for hydroentanglement to 1,5 N/g for needlepunching (Fig.10).
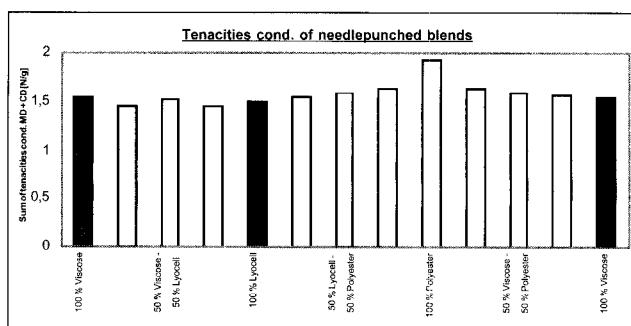

Fig. 10

- Tenacities wet

The wet tenacities of the needlepunched samples equally depend on the blend or fiber type as it is the case for the spunlaced samples.
The loss of tensile strength in wet conditions of viscose is much higher among the needlepunched samples than among the spunlaced ones. Whereas the loss of tenacity in wet state - in comparison to conditioned state - for spunlaced samples is only 38%, it reaches 90% for the needlepunched ones.
Considering the end-products, it is obvious that no hydroentangled nonwoven would ever pass any end-use approbation, if the tenacity in wet state were that much lower than the tenacity in conditioned state as it is true of the needlepunched samples.
Due to their high wet tenacity rates, spunlaced nonwovens on a viscose fiber basis are frequently found when contact with any kind of liquid must be expected such as body fluids (sweat, blood, wounds, etc.)

- Thickness, density, air permeability

Needlepunching causes far less compression to the web than hydroentanglement.

Thus, all polyester containing samples show thicknesses at a uniformly high level which are about 2,5 times higher than those of the other samples.

Hence, the densities of the materials containing polyester are at a uniformly low level. The situation concerning the air permeability is the same as for spunlaced materials. Higher polyester amounts lead to a higher air permeability.

Conclusion

Considering viscose fibers, tenacities are positively influenced by adding modified cellulosic fibers and synthetic fibers as well as by using finer fiber titers. Relations between viscose and lyocell are similar for tenacities in conditioned and in wet state when examining hydroentangled products. Needlepunched products show rather similar tenacities in conditioned state for viscose and lyocell, whereas there are differences between them in tenacities in wet state, which are equivalent to those of spunlaced samples.

Elongations are increased by adding synthetic fibers. Viscose fibers have shown higher elongations than modified cellulosic fibers.

Higher opacities can be reached by finer fiber titers and higher degrees of dullness.

Furthermore, air permeabilities are risen by using synthetic fibers and thicker titers.

Densities of the end-products result from the raw materials' densities. The density itself directly influences the thickness, which influences the air permeability.

Literature

[1] G. Sames; Production, Properties and Users' Application Habits of Nonwoven Hygienic Products Based on Cellulosic Fibers; Thesis Report - Lenzing AG (1997)