Practical aspects of the ammonia treatment of natural cellulose textiles

The liquid ammonia treatment of natural cellulose fibre woven fabrics is unique in terms of its effect, and is not to be replaced by mercerizing treatment. Particular advances in quality are produced by subsequent chemical finishing, which results in excellent wear and easy care properties with cotton and bast fibres, e.g. with shirts, blouse fabrics and industrial clothing.

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The liquid ammonia treatment of natural cellulosic fibre materials was introduced in the seventies by the Sanforized Company at first under the "Sanfor-Set" name, and first of all only for the final finishing of denim fabric. Shortly after, this technology came to Germany, where it has been practised up to now by the Martini company. Since 1984, there has been another variant of the liquid ammonia treatment, the Veramtex (B) "Beau-Fixe" process *(fig. 1)*, which is also well established in practical finishing.

Differences between the two treatments

Today, both liquid ammonia treatments are used in the finishing cycle in Europe predominantly as a pretreatment for easy care finishes. The two ammonia treatment methods differ particularly in the method of ammonia removal following the swelling process, which has some effect on the characteristics of the treated fabric. After impregnation with liquid ammonia, contact with the swelling medium amounts to a few seconds only ("Sanfor-Set" 4 to 8 secs, "Beau-Fixe" 10 to 20 secs). In the "Sanfor-Set" process, the ammonia is largely removed dry over drying cylinders at 130 to 145 °C and by blowing out the residual NH_3 gas with superheated steam, approx. 0.1% or more of residual ammonia remaining in the woven fabric, and generally being removed by water action prior to chemical finishing. With the "Beau-Fixe" process, part of the ammonia is removed first by blowing out with NH_3 gas to approx. 80% residual ammonia, followed by washing out with water, first cold then hot, and drying on stenters and/or perforated cages at 100 to 120 °C. Practically no NH_3 residues are present in the material after this treatment.

Which products and characteristics are produced today?

Tab. I shows the important products which are produced today on the basis of liquid ammonia treatments.

In terms of quantity, 100% cotton shirting and blouse materials plus industrial clothing for various types of application have the widest distribution. The biggest quality advances have however been achieved in the industrial care finishing of 100% cotton industrial clothing and the easy-care, shrinkproof, low-crease finishing of woven bast fibre fabrics in the widest variety of application fields.

The result of the latter is that projects are being increasingly put for-

Table I: Liquid NH_3-based products on the market.

shirts – blouses	industrial clothing	leisurewear
in cotton, cotton/linen, cotton/ramie, cotton/viscose: non-iron and easy care	hospital laboratory clothing, official clothing, 100% cotton leasing and protective clothing suitable for industrial easy care	trousers – windjammers – skirts etc. in 100% linen, 100% cotton and cotton/linen blends: easy care – non-iron
household textiles	various	new developments
table and decor products in linen and cotton and their blends: wash-fast – easy care	public authority products, woven transport fabrics, jeans: polyester in cotton and cotton/polyester, hemp	– bast fibre yarns or rovings: increased weaving elongation, decreased finishing shrinkage – woven lyocell fibre fabric

ward at political and economic levels for changing agricultural areas over from foodstuff to bast fibre cultivation.

Test figures

Illustrated by hand from meaningful test figures, *tab. II* shows the positive wear and care properties which have been obtained with shirts, and which have been tried and tested in the interim on millions of washed items. With the finishing processes usually employed to date, neither the fastness values required in practice nor the fast to washing wear and care results are to be obtained in terms of the cotton qualities concerned, and this statement still applies even for 30 and more wear and care cycles.

Parallel statements can be made by virtue of the results for cotton industrial clothing. So it is absolutely possible today:

- to treat cotton industrial clothing in industrial care terms as productively as polyester/cotton clothing.
- Wear behaviour and comfort during wear are more better than with polyester/cotton, and the long-term durability comparison comes out in favour of the 100% cotton product.

Practical knowledge reveals that bast fibre and bast fibre/cotton blend products can be finished so that wear, wash and care effects, which were hitherto just wishful thinking, can now be obtained.

These brief product result statements confirm that new products and effects suitable for the market can be produced by liquid ammonia treatments.

As a conclusion to this section, it is interesting to know that in Europe, for approx. 90% of all ammonia treated products, the relevant treatment is carried out as the basis for subsequent chemical finishing, in contrast to the original intention of both the "Sanfor-Set" the "Beau-Fixe" inventors.

Essential prerequisites for attaining high quality levels

In order to be able to attain the above-mentioned products and quality levels, requirements must be met in finishing on the one hand, while, on the other, the peculiarities of the cellulose conversion due to liquid ammonia must be taken into consideration.

Prerequisites

1. Woven fabric pretreatments properly and effectively carried out.
2. Taking account of and controlling fabric widths during the entire finishing cycle.
3. Taking account of some relevant, specific properties of the converted cellulose.
4. Familiarity with and careful application of the necessary finishing processes.

the pretreatment process cycle *(Tab. III)*.

This results in the appearance on the fabric per litre of treatment liquor in an impregnation scour/bleach process of:

- mineral particles: 8–15 g. This equates with approx. 400 to 700 German degrees of hardness,
- sizes and other scouring substances of 70 to 90 g, "crushed + emulsified".

In addition, the liquor is still charged with the treatment chemicals used.

On the basis of a great deal of work and experience, the following pretreatment process stages have been tried and tested:

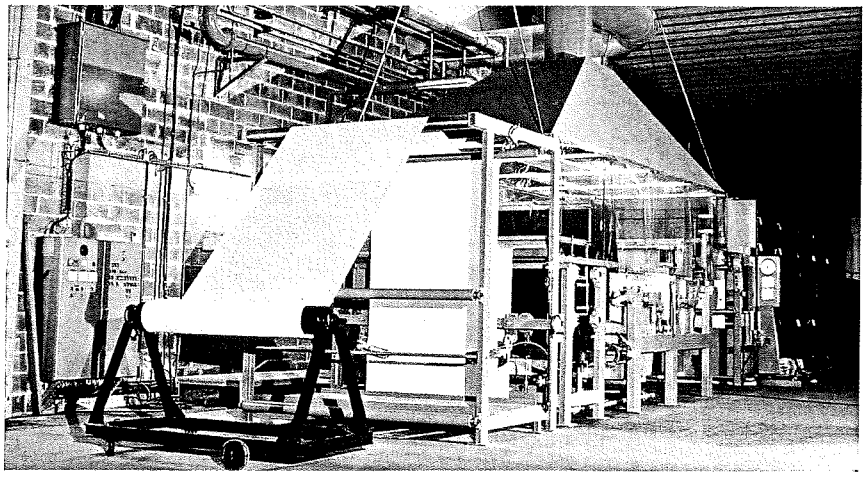

Fig. 1: Ammonia treatment plant. All photographs: Veramtex

Effective pretreatment

This is essential for obtaining optimum results, for it makes little sense to increase with liquid NH_3 access by dyestuffs and finishing chemicals to the fibre interior and to freely expose additional cellulose groups capable of reaction if these effects, which unfortunately is often the case, are utterly impaired by defective desizing, scouring and bleaching.

Very often, finely distributed impurities, usually mineral particles stemming from the grey fibres and sizes, remain on the fibre surface in

- acidic, complexed desizing/demineralisation with subsequent peroxide bleaches, which are well stabilized, and contain little alkali but good dispersing and dirt suspending agents; the target must be a material:
- with a residual ash content below 0.1%, and
- good absorbency, not due to a simulated rewetting agent.

Taking account of and controlling fabric widths

The liquid ammonia plants in current use are not capable of effec-

Table II: Poplin shirting: non-iron
Material: 100% cotton, weight: 110 to 115 g/m²

test criteria	pretreatment liquid NH_3 Veramtex	pretreatment NaOH mercerisation
tenacity weft [daN]	22–26	22–24
edge tear strength weft [cN]	1000–1200	500–600
% abrasion resistance loss, 3000T/90s	4–6	10–16
wash/wear appearance after 3 boil washes + tumbling	4–5	4
creasing angle total K + S dry wet	 280–310 250–280	 280–310 240–250
handle	soft/smooth, warm	harder/boardy, cold
fabric appearance/handle after 30 boil washes	soft clear	clear fluffy

tively countering the fabric width loss due to swelling. This width loss must be countered during the entire finishing cycle, otherwise troublesome finished product width, washing, and shrinkage values may result.

Tab. IV shows how this problem is solved successfully in practice. With woven bast fibre fabrics, it may be necessary to increase the loom reed width additionally by around 5–15% in order to obtain good dimensional stability in the wash.

Relevant properties of cellulose converted by liquid ammonia

The following must be taken into account for finishing processes following liquid NH_3 treatment:

- the increased and faster dye take-up,
- the alkali sensitivity of the transformed celluloses in the unwetted state in particular; it is worth mentioning here that this sensitivity is quite significantly lower with bast fibre celluloses than with cotton.

Some references to this sensitivity to alkalis:
- alkaline mercerisation or caustic soda treatment practically completely destroys the converting effects;
- dyeing for more than 15 minutes in strongly alkaline hot dyebaths in part reveals very considerable loss of effect.

Worth mentioning in connection with dyeing are:

- pad-steam dyeing, normally carried out, produces no significant loss of effect;
- indigosol/anthrasol dyeing with 60–70 ° sulphuric acid development does not have an adverse effect, and produces positive results in subsequent cross-linking;
- exhaust treatments or lengthy hot levelling out processes are to be avoided;
- the inconstant dyeing qualities more frequently appearing with alkaline mercerizing, caused by varying fibre dye absorption capacity, are greatly improved or avoided by liquid ammonia treatments;
- after liquid NH_3 treatment, an uneven, clearly visible yellowing, usually coupled with a residual ammonia content of 0.1% or more, often remains in the fabrics. These phenomena appear mainly with the "Sanfor-Set" process, and generally have to be corrected by subsequent washing out and/or bleaching, linked unfortunately in many cases with a reduction in positive ammonia effects.

Easy care finishing

Ammonia treatment results in poor minimum-iron properties, though it can create conditions for obtaining with a cross-linking finish good care properties combined with good wear and durability characteristics.

Interesting first of all is the fact that the improved crease recovery due to the ammonia treatment of woven cotton fabrics is also effective in chemical cross-linking, i.e. that fewer cross-linkers are required for obtaining a specific creasing angle level.

The markedly increased dry creasing angle level after "Sanfor-Set" treatment occurs to some extent only when the woven fabric is additionally hot water treated prior to cross-linking.

Detrimental easy care/strength relationships are produced by the classic chemical finishing of woven cotton fabric. This can be effectively countered by ammonia treatment. The improved fabric durability following this kind of treatment and finishing is particularly documented by much improved edge tear strength and abrasion resistance.

In order to guarantee the best possible finishing results, the liquid ammonia treatment should if possible be carried out in the finishing cycle immediately before the application processes. Liquid ammonia treatments generally produce

Table III: Impurities materializing in the pretreatment process (as a %).

	cotton	linen
minerals	0,8–1,4	0,6–1,2
pectins	3	3
fats/waxes	1–1,4	1,2–1,6
protein	2	3
hemicellulose	1,5–2	14–16
lignin	–	8

the required handle, wear and care effects only with selected chemical finishing operations.

As the cellulose conversion effected by liquid ammonia is also reversible by hot scouring liquors, the positive effects should be stabilized for all washable products at least by light cross-linking; this also applies to woven bast fibre fabrics.

How are the very positive product characteristics produced in practice?

There is an old prejudice, which needs correcting. Liquid ammonia treatments do not demand new chemical finishing processes, but produce improved or new product characteristics by the adaptation and extension of familiar operating cycles and formulations.

If however one wants to produce these improved characteristics, allowances must be made for the specific facts of the converted celluloses. The finishing cycles and formulations worked out must be very consistently implemented and monitored.

Many examples of the use of liquid ammonia reveal a relatively simple finishing process and simple dry cross-linking formulations, in which quite high cross-linker quantities can be used for obtaining good wear and care characteristics thanks to the effect of liquid NH_3 treatment.
Such a treatment cycle for 100% 115 g/m² multi-coloured/white cotton fabric for a formaldehyde-free easy care finish can be carried out as follows:

- singeing,
- acidic (complex former) desizing/scouring,
- produce ramie properties,
- liquid ammonia treatment,
- pre-softening for ramie properties (necessary depending on the soft handle required),
- chemical finish – for ramie properties,
- calendering,
- curing,
- Sanforising,
- final inspection/make-up.

Exacting chemical finish formulations (see box) are necessary in order to produce 100% cotton industrial twills not only to industrial easy care standards but also with wear comfort and a long service life.

Table IV: Width proportions of woven fabrics with the use of liquid ammonia.

treatment stages	poplin shirts non-iron	industrial twills
	cm	cm
grey fabric	160–162	162–166
after desizing/bleaching/ scouring/ramie	155–158*	*154–156
after liquid NH_3	144–148	144–155
after pre-softening and ramie	153–156*	*154
after chemical finishing and ramie/curing	153–156	152–153
after washing out/ following after-softening/ramie	152–153	152–153
after Sanfor final inspection	151–152	151–152

* tenter fabric to width with plenty of overfeed

Example of a chemical finish formulation for industrial twills dyed at pH 4–6

Wet cross-linking:	"Knittex FA conc."	100 g/l
	"Fixapret PH"	160 g/l
	"Dicrylan WK"	40 g /l
	"Ultratex FH" new	40 g/l
	"Turpex MA"	20 g/l
	Catalyst UMP	110 g/l
	Hydrochloric acid conc.	8 cm³/l
Dry at 65–70 °C		
Batch 38–40 °C	residual moisture 7–8%	
Reaction time 22–24 hrs at 30 to 34 °C		
After-softening (after washing out, wet on wet)		
	"Dicrylan WK"	40 g/l
	"Ultratex FH" new	30 g/l
	"Dicrylan cat. W"	5 g/l
	"Phoboton WS" conc.	5 g/l
	"Avivan MS"	15 g/l
	Acetic acid 80%	1 cm³/l
Dry at 110–120 °C gentle air supply		
Cure: 150 °C on woven fabric approx. 3–4 mins (after 24 h dwell period).		
Sanforising		

Future of liquid ammonia treatment

The use of this treatment will steadily increase, since better effects, new finishing methods and qualities can be achieved with it. In addition, a new field has been opened up for ammonia treatment by the increased use of bast fibre fabrics, in the clothing sector too. In addition, this process is relatively environment-friendly.

In plant development, it is to be expected that liquid NH_3 equipment will be improved. This applies in relation to better woven fabric dimensional stability, use for knitted fabrics, residual NH_3 removal with no reduction in effect, and for use in new fields of application like yarn treatment, and particularly bast fibre yarn, for example.

ONS-No. 8-466 ■