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## THE BLEACHING OF COTTON WITH EMULSIONS OF HYDROGEN PEROXIDE IN A PERCHLOR-ETHYLENE MEDIUM

*by*

N. J. J. VAN RENSBURG

SOUTH AFRICAN WOOL TEXTILE RESEARCH INSTITUTE
P.O. BOX 1124
PORT ELIZABETH

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# THE BLEACHING OF COTTON WITH EMULSIONS OF HYDROGEN PEROXIDE IN A PERCHLORETHYLENE MEDIUM

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## ABSTRACT

*Cotton can be successfully bleached with emulsions of hydrogen peroxide in perchlorethylene by single step batch application. The effect of peroxide concentration, reaction time and reaction temperature on the degree of whiteness and the fluidity of cotton bleached in perchlorethylene has been studied.*

## KEY WORDS

Cotton – bleaching – hydrogen peroxide – emulsion – perchlorethylene.

## INTRODUCTION

The fundamental aspects of the dyeing and finishing of textiles in organic solvents have been investigated by a large number of scientists[1, 2, 3]. Their results indicated that the treatment of textiles in non-aqueous solvents offered several advantages over conventional aqueous processes. The dyeing and finishing of textiles in organic solvents, however, only became feasible when the manufacturers of textile equipment started developing and producing special solvent finishing machines[4].

Research workers at the South African Wool and Textile Research Institute (SAWTRI) have been active in the solvent processing field and several papers on the treatment of wool in perchlorethylene have been published[5, 6, 7, 8].

The scouring of cotton and various cotton blends in trichlorethylene and perchlorethylene is in commercial operation in several countries. This was recently followed by the development of continuous solvent scouring, desizing and bleaching processes, such as the Markal process[9, 10]. In the Markal process scouring is combined with bleaching and desizing by treatment of the cotton in emulsions of aqueous hydrogen peroxide and enzymes in a trichlorethylene medium, followed by steaming and subsequent aqueous washing-off. In this process, which employs a solvent as well as an aqueous step, only a half bleach is obtained. Laboratory trials carried out by Gordon[11] showed that an improved bleaching effect could be obtained for poor quality cotton fabrics when the samples were subjected to steaming after treatment with emulsions of hydrogen peroxide.

Most of the work done on the solvent bleaching of cotton, involved two-step processes. As yet, very little information is available on the effect of a one-step solvent bleaching process on the degree of whiteness, the mechanical properties, etc. of cotton and consequently the present investigation was carried out.

## EXPERIMENTAL

A plain weave, unbleached calico fabric (density 159 $g/m^2$) and a plain weave, greige cotton fabric (density 210 $g/m^2$) were used in the bleaching experiments. All the chemicals used were of laboratory grade. Hydrogen peroxide (100 vols.) was used as the bleaching agent. Lapotex PC (Laporte Chemicals) was used as a stabilizing agent in some of the bleaching experiments carried out in perchlorethylene. In the case of the bleaching trials performed in aqueous solution, a conventional silicate stabilizing system was employed. Commercial emulsifying agents* such as Aerosol OT[a] (American Cyanamid) or a mixture of Nansa HS[b] (Marchon Ltd.) and Empilan CME[c] (Marchon Ltd.) were used to obtain stable emulsions of hydrogen peroxide in perchlorethylene. The emulsions of peroxide in perchlorethylene were prepared in the following way: the emulsifying agent was dissolved in the perchlorethylene and the aqueous peroxide (in some cases containing the dissolved stabilizing agent) was slowly added to the solution while stirring mechanically. The aqueous bleaching solution was prepared as follows: 7 g sodium silicate, 0,5 g sodium hydroxide, 1,8 g sodium carbonate, 1 ml Tergitol Speedwet, $x$ ml hydrogen peroxide, made up to 1 litre with water.

Bleaching was carried out in an Ahiba laboratory dyeing apparatus, using a wool to liquor ratio of 1:40. Industrial scale experiments were carried out in a LMF-12 Permac Böwe apparatus. The degrees of whiteness of the fabrics were determined with a Zeiss Elrepho apparatus using the formula suggested by Berger[12]. The cuprammonium fluidities of the samples were determined by the method recommended by the Shirley Institute[13]. The abrasion resistance of the samples was determined on a Martindale Abrasion Tester. Breaking strengths of the fabrics were determined in the usual manner.

## RESULTS AND DISCUSSION

Table I shows the composition of emulsions of hydrogen peroxide in perchlorethylene which were found to be reasonably stable. Emulsion system A was used in most of the cases. The emulsions were normally used within five minutes after having been made up.

It is generally accepted that the treatment of cotton with hydrogen peroxide alone does not have a significant bleaching effect on the cellulosic fibres[14]. Hydrogen peroxide is normally stabilized with acids and to increase the reactivity of the peroxide, it is activated with alkali. It is, however, necessary to control the rate of decomposition of hydrogen peroxide during a bleaching process and consequently cotton is normally bleached in an alkaline medium in the presence of

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*a = dioctyl sodium sulphosuccinate
b = sodium dodecylbenzenesulphonate
c = lauryl monoethanolamide

## TABLE I

## COMPOSITION OF EMULSIONS OF HYDROGEN PEROXIDE IN PERCHLORETHYLENE

| | SYSTEM A | SYSTEM B |
|---|---|---|
| Perchlorethylene | 1000 ml | 1000 ml |
| Hydrogen peroxide | 0 – 5 ml | 0 – 5 ml |
| Lapotex PC* | 0 – x g | 0 – x g |
| Aerosol OT | 10 g | – |
| Nansa HS | | 6 g |
| Empilan CME | | 4 g |

*The ratio of Lapotex PC to hydrogen peroxide was 1 : 1000

stabilizing agents, such as silicates. In order that the effect of stabilizing agents on the bleaching of cotton with emulsions of peroxide in perchlorethylene might be determined, a number of bleaching experiments employing peroxide and a stabilizing agent under various conditions were carried out. The results obtained are given in Table II. Surprisingly enough, it was found that a good bleaching effect could be obtained by using peroxide as bleaching agent in perchlorethylene without

## TABLE II

## THE DEGREE OF WHITENESS OF COTTON BLEACHED WITH HYDROGEN PEROXIDE IN WATER AND PERCHLORETHYLENE IN THE PRESENCE OF VARIOUS STABILIZING AGENTS

| Treatment* | Whiteness W |
|---|---|
| Perchlorethylene | 17,6 |
| Perchlorethylene, 0,1% Lapotex PC, pH 1,6 | 17,8 |
| Perchlorethylene, 0,1% Lapotex PC, pH 7,0 | 18,1 |
| Water, 0,1% Lapotex PC, pH 10,0 | 17,3 |
| Water, silicate buffer | 19,3 |
| Untreated | 14,4 |

*Bleaching was carried out with 1 ml $H_2O_2/l$ for 30 minutes at 60°C the addition of alkali or stabilizing agent. The addition of a stabilizing agent to the peroxide had very little effect on the degree of whiteness of the cotton fabrics. The pH of a 0,1% solution of Lapotex PC in hydrogen peroxide was 1,6. This solution was neutralised with sodium hydroxide to pH 7,0 prior to bleaching the cotton. Once again it was found that neutralisation of the peroxide had very little effect on the degree of whiteness of the fabrics. It was, unfortunately, not possible to increase the pH of the solution any further, since the peroxide started to decompose when the pH exceeded 8,0. Since no significant increase in the degree of whiteness of the cotton was obtained when bleached with peroxide in the presence of the stabilizing agent under various conditions, it was decided to use hydrogen peroxide as bleaching agent without adding alkali or a stabilizing agent.

Perchlorethylene and trichlorethylene were compared as a medium for the bleaching of cotton by hydrogen peroxide. The results obtained appear in Table III.

**TABLE III**

**THE DEGREE OF WHITENESS OF COTTON BLEACHED* IN PERCHLORETHYLENE AND IN TRICHLORETHYLENE**

| CONCENTRATION OF HYDROGEN PEROXIDE (ml/*l*) | DEGREE OF WHITENESS | |
|---|---|---|
| | TRICHLORETHYLENE | PERCHLORETHYLENE |
| 0,5 | 16,8 | 16,9 |
| 1,0 | 18,8 | 18,2 |
| 2,0 | 20,9 | 18,9 |

*Bleached for 30 minutes at 60°C

It can be seen that the fabrics which had been bleached in trichlorethylene were slightly whiter than those bleached in perchlorethylene, when more than 1,0 ml/*l* of hydrogen peroxide were used. No differences were found between the two solvents when low concentrations of peroxide (0,5 ml/*l*) were used. In the industrial scale solvent dyeing apparatus, which was to be employed in some studies, a perchlorethylene system was used and consequently it was decided to do all the preliminary studies in perchlorethylene.

The effect of the time of reaction on the degree of whiteness of cotton fabrics bleached with hydrogen peroxide in water and perchlorethylene respectively, is shown in Figure I. It can be seen that cotton can be bleached to a high degree of whiteness in perchlorethylene, but it was, however, not possible to obtain the same degree of whiteness in this medium as that obtained in water. This is in contrast to the solvent bleaching of wool, where it was found that the non-aqueous bleaching process gave a whiter fabric more rapidly than did the conventional aqueous

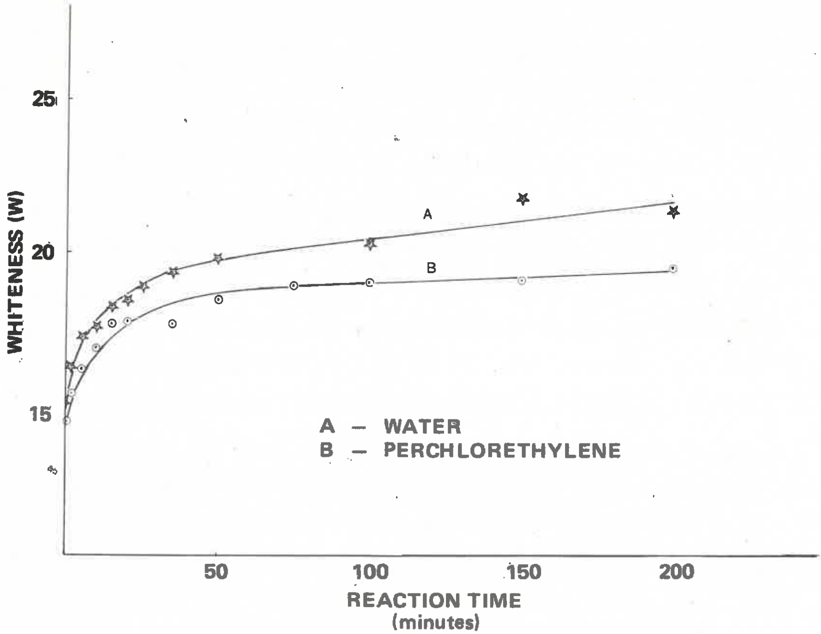


**FIGURE I**

**The effect of the reaction time on the degree of whiteness of cotton bleached in water and perchlorethylene, respectively**

process[7]. The handle of cotton fabrics which had been bleached in perchlorethylene was, however, superior to the handle of fabrics bleached in water.

Figure II shows the effect of the concentration of hydrogen peroxide on the degree of whiteness of cotton fabrics bleached in water and in perchlorethylene. It can be seen that the degree of whiteness of the fabrics increased when treated with increasing concentrations of hydrogen peroxide. There was little difference between the perchlorethylene and the aqueous bleaching processes when low concentrations of hydrogen peroxide were used (less than 0,5 ml $H_2O_2/l$). When more than 0,5 ml $H_2O_2/l$ was used, bleaching in water gave whiter fabrics than those obtained by bleaching with the same quantity of peroxide in perchlorethylene.

The effect of reaction temperature on the degree of whiteness of cotton bleached in water and in perchlorethylene respectively, is shown in Figure III. The graphs show an increase in the degree of whiteness with increasing reaction temperatures. There was an increase in the degree of whiteness of cotton bleached in perchlorethylene with increasing reaction temperatures up to a temperature of 70°C, after which the whiteness decreased slightly up to boiling point. In the case

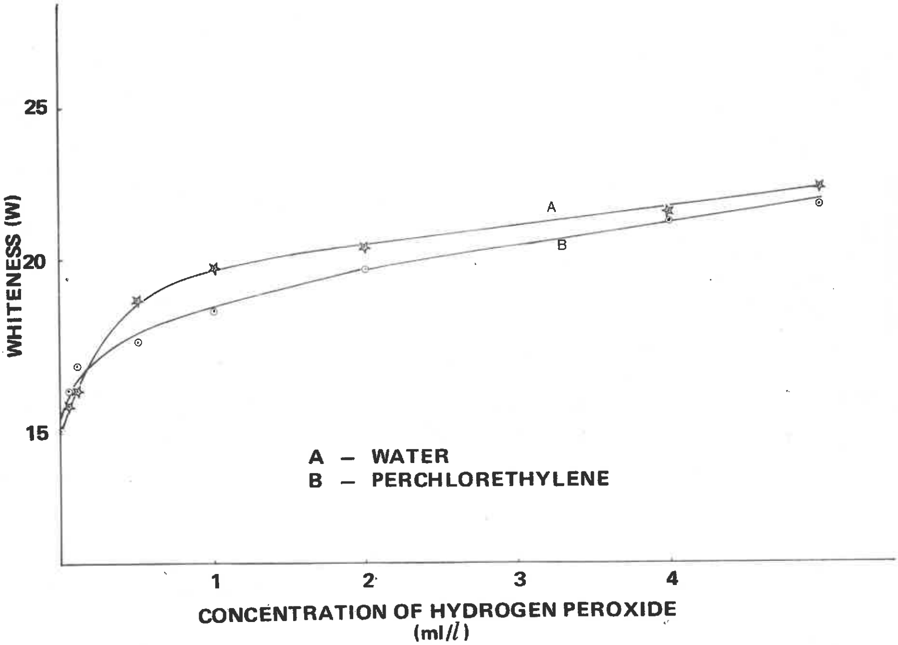


**FIGURE II**

**The effect of the concentration of hydrogen peroxide on the degree of whiteness of cotton bleached in water and perchlorethylene, respectively**

of the aqueous bleaching process there was a gradual increase in the degree of whiteness up to boiling point. A comparison of the aqueous and non-aqueous processes shows that at relatively low and high reaction temperatures the samples treated in water were whiter than those treated in perchlorethylene. When the fabrics were bleached at intermediate temperatures (60–70°C), no differences were found between the two processes.

It was first thought that the fact that the bleaching of cotton in water generally produced slightly whiter fabrics than did bleaching in perchlorethylene, could have been due to the extraction of water-soluble coloured compounds by the water from the fibres. This was, however, shown not to be the case since the same effect was observed when the fabrics were washed for 45 minutes at 40°C in a conventional washing machine and dried at room temperature, prior to bleaching.

The difference in the physical state of the fibres in the aqueous and non-aqueous media could have been the reason for the observed differences in bleachability of the cotton. Aqueous bleaching was carried out in a strong alkaline medium which could have increased the swelling of the fibres, thereby facilitating

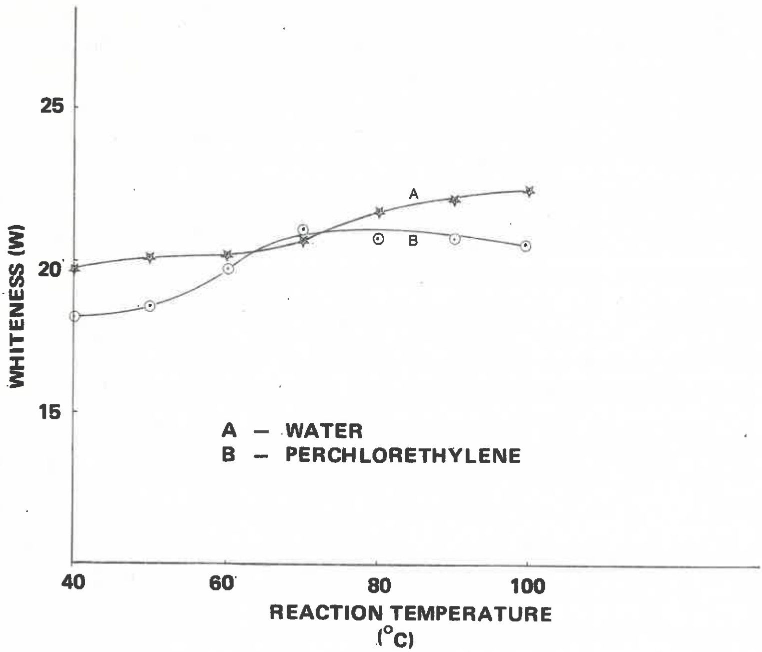


FIGURE III

**The effect of the reaction temperature on the degree of whiteness of cotton bleached in water and perchlorethylene, respectively**

the bleaching process. It was, therefore, decided to mercerise the cotton before bleaching the samples but even in the case of mercerised cotton the aqueous bleaching process yielded slightly whiter fabrics than did the perchlorethylene bleaching process.

The emulsifying agent in the perchlorethylene could have yellowed the cotton and some experiments were therefore carried out to investigate this possibility. Cotton was treated at various temperatures up to the boil in perchlorethylene and perchlorethylene plus emulsifying agent, respectively. It was found, however, that neither the perchlorethylene nor the combination of perchlorethylene and emulsifying agent had a significant effect on the degree of whiteness of the cotton fabrics.

The extent to which the cellulosic fibres were chemically damaged by the peroxide bleaching processes was also determined. In this study the conventional cuprammonium fluidity determination was used as a measure of the chemical degradation. Cotton having a fluidity of below 3 rhes is normally classified[15] as "mildly scoured and bleached" while samples having fluidities of from 3 to 7 rhes are classified as being "normally scoured and bleached". Samples with fluidities above 10 rhes are regarded as being over-bleached. Figure IV shows the effect of concentration of peroxide on the fluidity of cotton bleached in water and perchlorethylene. It can be seen that the fluidities of the samples treated in perchlorethylene were significantly higher than those of the samples treated in water. Furthermore the fluidities of the samples treated in perchlorethylene increased rapidly with increasing concentrations of peroxide. In fact, in the cases studied, no aqueous bleaching process caused overbleaching of the cotton, while the use of more than 3,0 ml/*l* $H_2O_2$ in perchlorethylene caused some overbleaching of the fabrics. The most critical factor in the solvent bleaching process proved to be the concentration of the hydrogen peroxide. Reaction time and temperature affected the fluidity to a lesser extent. For a study of the effect of time of reaction and reaction temperature on the fluidity of cotton, fairly low concentrations of peroxide (e.g. 1,0 ml/*l*) were used. The fluidity of cotton bleached in perchlorethylene for 15 minutes (at 60°C), was increased by 0,9 units upon extending the reaction time to 200 minutes, while the increase in fluidity in the case of samples

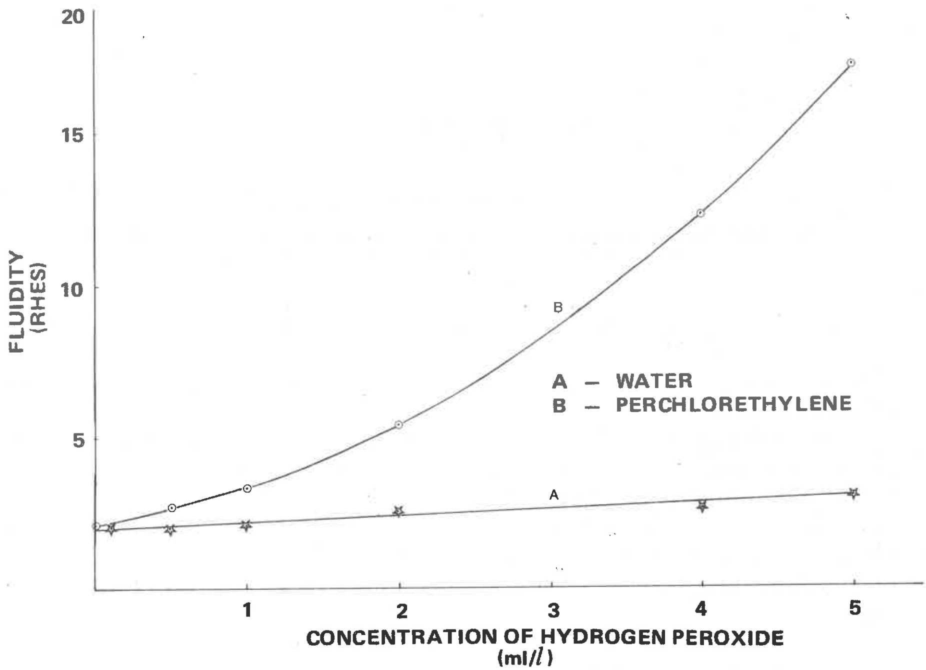


**FIGURE IV**

**The effect of the concentration of hydrogen peroxide on the fluidity of cotton bleached in water and perchlorethylene, respectively**

## TABLE IV

### THE BREAKING STRENGTH, BREAKING EXTENSION AND ABRASION RESISTANCE OF COTTON BLEACHED IN WATER AND IN PERCHLORETHYLENE

| TREATMENT* | WARP BREAKING STRENGTH (Kg) | WARP BREAKING EXTENSION (%) | CYCLES TO ENDPOINT |
|---|---|---|---|
| Water, 60°C | 91,4 | 24,8 | 12 000 |
| Water, 80°C | 90,4 | 25,0 | 16 000 |
| Perchlorethylene, 60°C | 87,6 | 23,5 | 14 000 |
| Perchlorethylene, 80°C | 90,0 | 25,7 | 16 000 |

*Bleached with 1,0 ml $H_2O_2/l$ for 30 minutes

bleached in water was 0,3 units. Bleaching of the cotton for 30 minutes at various temperatures resulted in the fluidity increasing from approximately 3,0 rhes at 50°C to 6,7 rhes at boiling point for perchlorethylene-treated samples and to 4,7 rhes at boiling point for water-bleached samples.

Finally, certain of the mechanical properties of the bleached samples were studied. The results obtained are given in Table IV. The results show little difference between the two bleaching processes and confirm the fluidity results, namely that there is little difference between the aqueous and non-aqueous

## TABLE V

### THE EFFECT OF THE CONCENTRATION OF PEROXIDE AND REACTION TEMPERATURE ON SOME PROPERTIES OF COTTON FABRICS BLEACHED IN AN LMF-12 BÖWE MACHINE

| TREATMENT* | DEGREE OF WHITENESS | FLUIDITY (rhes) | Breaking Strength (Kg) | |
|---|---|---|---|---|
| | | | Warp | Weft |
| 0,5 ml $H_2O_2/l$ 60°C | 17,8 | 3,00 | 64,6 | 48,2 |
| 1,0 ml $H_2O_2/l$ 60°C | 19,6 | 3,39 | 75,7 | 50,6 |
| 0,5 ml $H_2O_2/l$ 80°C | 19,0 | 3,98 | 76,3 | 45,9 |
| Untreated | 14,4 | 1,93 | 80,3 | 38,5 |

*2,5 Kg cotton was treated for 30 minutes in 50*l* perchlorethylene containing 170 g Nansa HS and 113 g Empilan CME. The rotation speed of the drum was 2,5 r.p.m.

bleaching processes, provided that the concentration of peroxide is kept relatively low.

After completing the laboratory scale experiments, some bleaching trials were carried out on an industrial scale in a Permac Böwe machine using perchlorethylene as the solvent. The results obtained are given in Table V and show that cotton fabrics could be bleached to a high degree of whiteness with emulsions of hydrogen peroxide in this machine. The degree of whiteness increased with increasing concentrations of peroxide and with an increase in reaction temperature, confirming the results obtained on a laboratory scale. A slight increase in the fluidities of the samples was observed when the concentration of peroxide or the reaction temperature was increased, but no overbleaching of the fabrics was noticed under the conditions studied. The breaking strengths of the samples also showed that the bleaching treatments caused no significant deterioration of fabric strength. It can therefore be concluded that cotton can be successfully bleached in a one step process by batch application in a LMF-12 Permac Böwe machine.

## CONCLUSIONS

It has been found that cotton could be successfully bleached with emulsions of hydrogen peroxide in perchlorethylene. A high degree of whiteness could be obtained without adding any stabilizing or activating agent to the peroxide. The degree of whiteness obtained depended on the concentration of peroxide, the reaction time and the reaction temperature. It was found that the bleaching of cotton in water produced a whiter fabric than did bleaching in perchlorethylene under identical conditions. The handle of the fabrics bleached in perchlorethylene was, however, superior to the handle of the fabrics bleached in water.

The cuprammonium fluidity of cotton bleached in perchlorethylene was higher than that of samples bleached in water. However, as long as the concentration of peroxide was kept fairly low, no overbleaching was observed in the case of the samples bleached in perchlorethylene. It was furthermore found that the mechanical properties of the fibres were not impaired to a larger extent by the perchlorethylene-bleaching process than by the water-bleaching process.

Finally, it was found that batches of cotton fabrics could be successfully bleached with peroxide in perchlorethylene on an industrial scale in a LMF-12 Permac Böwe solvent dyeing apparatus.

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