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MICROSCOPICAL INVESTIGATION OF
RESIN TREATED COTTON FIBERS

BY

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ABSTRACT:

Trials have been made to make use of a light microscope and swelling solution to explain the reduction of strength and breaking extension after resin treatment. The results obtained are in full agreement with earlier postulation. The latter ascribed the loss in strength properties to stress concentration by crosslinking and cellulose degradation by the catalyst used in resin treatment.

INTRODUCTION:

Synthetic fibers possess beneficial properties which is unique, that is, thermoplasticity. They can be heat set to any required shape. The competition between synthetic and natural fibers has led to modification of the natural fiber to enable the latter to compete with the synthetics. One of the most successful modifications is cross-linking of cotton using N-methylol finishing agents commonly known as resins.

However, resin treatment of cotton reduces its strength and breaking extension considerably (1-4) which in turn affects the durability. The explanation of the reduction in strength and extension can be related to the fine structure of cotton (2-4). Cotton is two phase structure, crystalline and non-crystalline regions. Crystalline region is highly ordered structure and represents about 66%. The less ordered structure (noncrystalline) acts as areas in which slippage can occur, when stress is applied, allowing transfer and relief of stress. After treatment, cross-links between cotton molecules in the noncrystalline region take place. They do not

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allow transfer of stress causing a stress concentration and hence drop in strength and breaking extension.

Another reason explaining the drop in strength has been suggested^(1,3) that part of the loss in strength in formaldehyde treatment may be due to the degradation of cotton cellulose via acid hydrolysis resulting from the low pH required to effect reaction.

The object of the present work is firstly to show the location and the shape and secondly to identify the resin treated cotton fibers at different conditions of treatment by simple microscopical investigation. This identification is believed to help in controlling the process of resin treated cotton goods in the industry. Another useful application for identification that in the fabric analysis area.

EXPERIMENTAL:

Materials:

Giza 70 cotton in fiber form has been used. Resin under the trade name "Cassurit PV" produced by CASSELLA company in liquid form has been used. Magnesium chloride with fixed amount of 15 g/l as a catalyst was employed and a commercial wetting agent was added to help resin penetration inside the cotton fibers during the resin treatment.

Resin solution was prepared by adding the resin, wetting agent, and finally the catalyst.

Scouring:

Before chemical treatment with resin solution, the cotton fibers have been scoured for one hour at the boil in an aqueous solution containing 2% sodium hydroxide. This stage was done to remove wax and impurities in order to improve absorpency and hence resin penetration when treating with the resin solution. The cotton fibers then squeezed and left to dry at ambient conditions.

Resin Treatment:

After scouring and drying the cotton fibers were immersed in the resin solution bath for 10 minutes, then squeezed to a liquor pick-up of 150%. After that the fibers were dried in electrical oven at 70°C for 2 hours.

Percentage resin pick-up was controlled by varying the resin concentration and keeping the liquor pick-up constant. The range of concentration was 80-160 g/l.

The curing has been carried out in electrical oven. The range of curing temperature was 120-200°C, and curing time was 1-5 minutes.

Microscopic Examination:

a) A light microscope with attached camera and heating disc has been used. The temperature was controlled by connecting the heating disc with "Universal Incubator Type U₃(Kovo)".

b) A solution consisting of Zn Cl₂ (100 g), KI(32 g), distilled water (34 g) and I₂ till saturation * was used as swelling agent during the microscopic examination at 80°C for raw and treated cotton fibers.

Testing:

Fiber bundle strength and breaking extension percent were measured by using a stelometer, Model 154. The gauge length used was 1/8 inch bundle length gripped in the jaws.

RESULTS AND DISCUSSION:

Three factors have been studied namely, resin concentration, curing temperature, and curing time. The experiments covered a wide range of these three factors including the conditions recommended by the industry.

* W.Bobeth U.A.Mally; Formel Faserstoffe Fertigungsinformation (1/79), S-1-19.

Resin Concentration:

Fig. 1 shows the resin concentration versus fiber bundle strength and breaking extension. Loss in strength percent and in breaking extension percent based on the untreated strength and breaking extension of the untreated fibers are shown in two separate scales. The bundle strength and breaking extension of the untreated fibers are 30 g/tex and 7% respectively. The fiber strength and breaking extension, as it appear from Fig. 1, consistantly decrease with percent resin concentration.

Plates 1,3-6 show the longitudinal views of treated fibers of those shown in Fig. 1.

Plate-1 shows the longitudinal view of swollen raw cotton fibers. It is seen that the secondary wall is highly swollen in a direction perpendicular to the lumen of the mature fibers and appears pale blue in colour, whilst the layer of the primary wall is much less swollen than the inner layer and appears as deep blue bundles as well as zig-zag or ring form tightly surrounding the swollen cellulose of the primary wall.

Plate-3 shows the longitudinal view of resin treated fibers at 80 g/l concentration. Swelling of the secondary wall of cellulose is observed in the direction perpendicular to the lumen along the mature fibers. It is further seen that for the same fiber, some parts are swollen while the others are not.

Plate-4 shows longitudinal view of cotton fibers treated with 100 g/l. It is observed: (a) that some fibers exhibit nearly same degree of swelling, (b) that with some fibers swelling is not uniform along the fiber and, (c) that there are fibers which strongly resist swelling. The resistance to swelling is unequivocally due to the crosslinking of cotton fibers via reaction with the finishing agent. It is understandable that the crosslinks impede diffusion of the swelling

agent inside the cellulose structure thereby decreasing swelling.

Plate-5 shows longitudinal view of cotton fibers treated with 120 g/l resin concentration. Fibers of high resistance to swelling are seen. Natural convolutions of the mature fibers are lost. The fibers exhibit a blue colour. Fibers of high swelling properties also exist.

Plate-6 shows longitudinal view for fibers treated with 140 g/l resin concentration. It shows a collection of swelling behaviours similar to those mentioned above.

It is clear from Fig. 1 and plates 1,3-6 that the loss in strength and in breaking extension with increasing the resin concentration can be explained as mentioned earlier in this paper. The increase in concentration increases the number of crosslinks formed between molecules in the noncrystalline region. The more the crosslinks the less stress transfer and hence less strength will result. The results show that a range of 80 g/l-160 g/l reduce the strength and extension, 27% - 48% and 13% - 32% respectively.

Curing Temperature:

Fig. 2 shows the effect of curing temperature on fiber bundle strength and breaking extension. Here again both strength and breaking extension decrease sharply with curing temperature.

Plates 5, 7, 8 show the longitudinal views of three samples out of the five samples of Fig. 2.

Plate-5 is described in the last section.

Plate-7 represents a part of longitudinal view of cotton fiber showing variation in swelling between conventional swelling behaviour of secondary wall bound with primary wall and

the inability of some parts of fiber to swell efficiently.

Plate-8 shows most of the fibers resist swelling to a considerable degree though some tend to swell horizontally.

The effect of setting temperature on bundle strength and breaking extension as seen from Fig. 2 and plates 5, 7, 8 that increasing temperature initiate the reaction between the cotton molecules and the resin and hence more crosslinks formed. The more crosslinks the more hindrance for stress transfer causing stress concentration and reduce strength. Another reason causing the reduction in strength and breaking extension by temperature increase that is high temperature may result in deterioration of cellulose molecules. Moreover, very high temperature (180-200°C) may cause over-curing of the resin which cause embrittlement to the whole structure.

Curing Time:

Fig. 3 illustrates the effect of curing time on fiber bundle strength and breaking extension. It is seen that the longer the curing time the lower the strength and breaking extension.

Plates 9-12 show the longitudinal views of those samples of Fig. 3.

Plates 9,10-show cotton fibers treated with 120 g/l at 160°C for 1 and 2 minutes respectively. It is seen that swelling is just similar to the aforementioned cases.

Plates 11, 12-show cotton fibers treated with 120 g/l resin concentration at 160°C for 4 and 5 minutes respectively. In plate 11 swelling appears as beads at different distances along the length of one of the fibers. It is generally observed that some fibers have become susceptible to swelling by allowing penetration of swelling agent most probably due to

cleavage of crosslinks. In plate 12 swelling appears nonuniform in some fibers; other fibers resist swelling. Again the reduction in both strength and extension can be explained by the fact that the longer the setting time the more the number of crosslinks and less stress transfer resulting in stress concentration. In addition the longer the curing time the more chemical deterioration of cellulose chains. Moreover, at very long curing times (5 minutes) embrittlement and hence weak structure may occur.

Contribution of Different Treatment Stages:

It is important to analyse the mechanism of the reduction in the strength and breaking extension of resin treated cotton fibers and investigate the contribution of each treatment stage. To do this it is necessary to break down the treatment stages.

Figs. 4, 5 show the effect of each treatment stage on fiber bundle strength and breaking extension respectively. They show five different samples each treated at different treatment stage. The first sample (Plate 1) is untreated sample. The second sample was treated with catalyst. The third sample was treated with resin and catalyst. The fourth sample was treated with catalyst and heating. The fifth sample was treated with resin and catalyst followed by curing (Plate 5).

From Figs. 4, 5 it is clear that each stage causes reduction in both strength and extension.

The effect of catalyst is a reduction of 10% and 8% in strength and breaking extension respectively. The effect of the treatment with resin and catalyst is a reduction of 12% in strength and 12% in breaking extension. This means that the effect of treatment with the resin alone is reduction 2%

in strength and 4% in breaking extension (difference between Column 3 and 2).

The chemical deterioration can be deduced from columns 2 and 4. Column 2 represents a sample treated with catalyst and column 4 represents a sample treated with catalyst and heating.

The difference between column 5 and 4 represents the effect of the crosslinks on the reduction in strength (13 %) and breaking extension (10%).

The results of Figs. 4, 5 support strongly the reasons mentioned earlier in this paper, that is the reduction in the strength and breaking extension of resin treated cotton is due to firstly the cross-links hinder the transfer of stress causing stress concentration, secondly the chemical deterioration resulting from acid hydrolysis of the cellulose chain.

SUMMARY:

It has been shown that the reduction in strength and breaking extension of resin treated cotton is due to:

1- Stress concentration resulting from the crosslinks formed, the more the crosslinks the more stress concentration and hence reduction in strength and breaking extension. The three factors studied namely resin concentration, curing temperature, and curing time reduce strength and extension.

2- Chemical deterioration due to acid hydrolysis resulting from low pH required to effect reaction.

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Longitudinal view of swollen raw and treated cotton fibers swelling agent consists of 100 g. Zn Cl₂+32 g. KI +34 ml. distilled water .
Microscopic examination performed at 80°c.

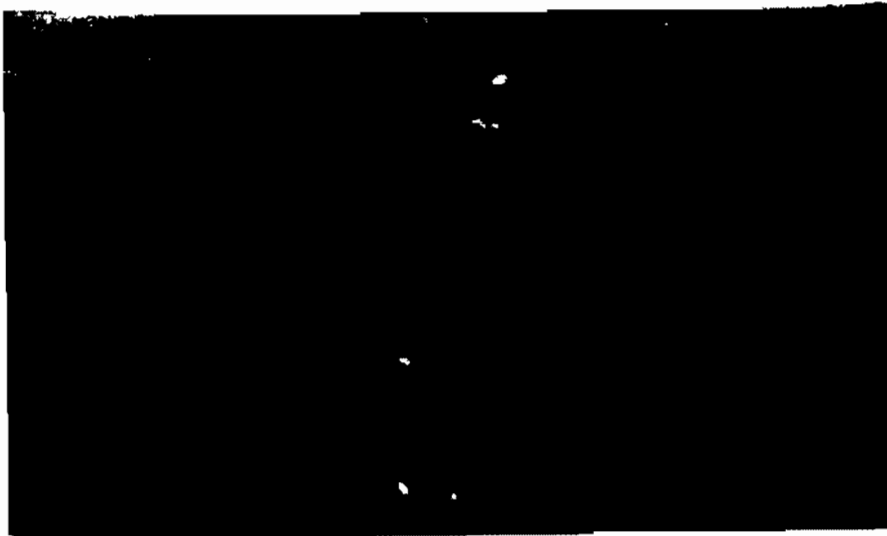


Plate 1: Raw cotton fibers



Plate 2: Treated cotton fibers (washed and heated with catalyst at high temp. in absence of finishing agent.)

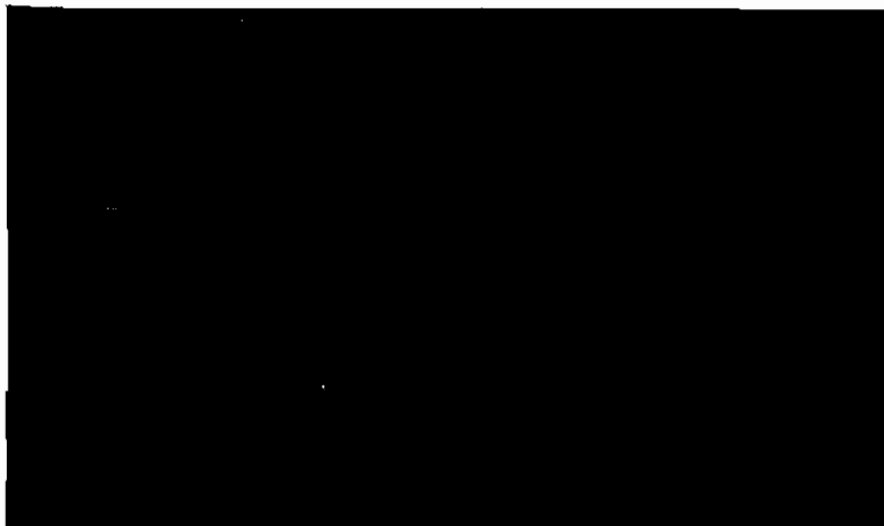


Plate 3: Cotton fibers treated with finishing agent (80 g/l) for 3 min. at 160°C.



Plate 4: Cotton fibers treated with finishing agent (100 g/l) for 3min. at 160°C.



Plate 5: Cotton fibers treated with finishing agent (120 g/l) for 3 min. at 160°C.

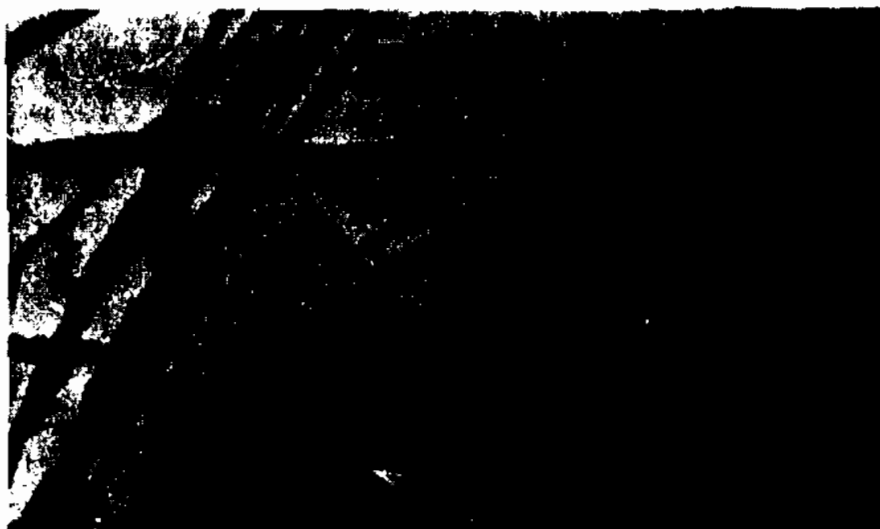


Plate 6: Cotton fibers treated with finishing agent (140 g/l) for 3 min. at 160°C.

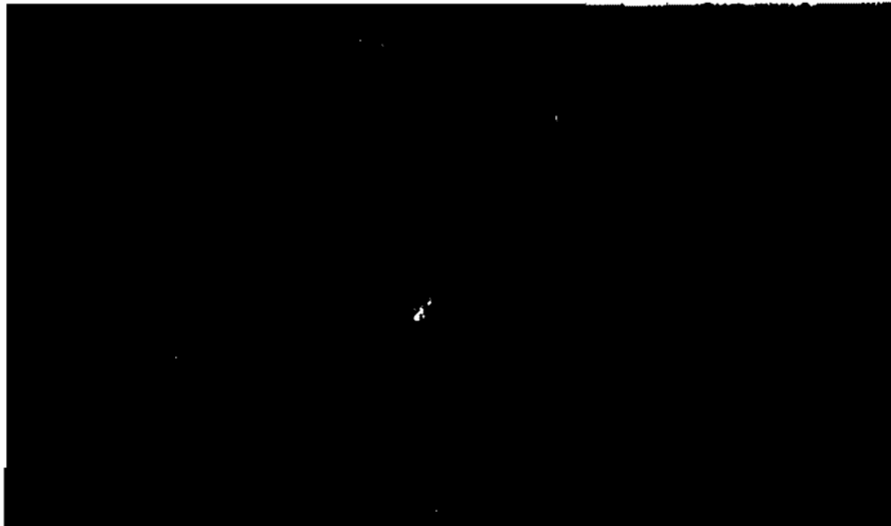


Plate 7: Cotton fibers treated with finishing agent (120 g/l) for 3 min. at 180°C.



Plate 8: Cotton fibers treated with finishing agent (120 g/l) for 3 min. at 200°C.

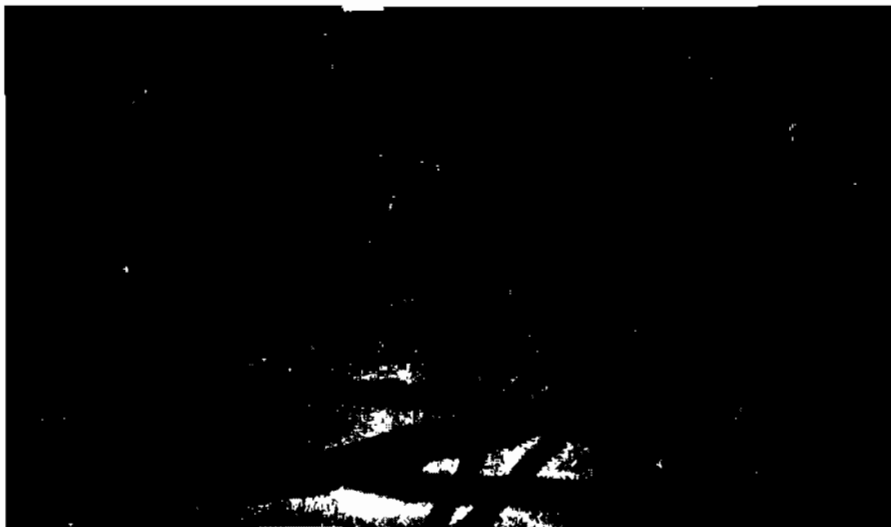


Plate 9: Cotton fibers treated with finishing agent (120 g/l) for 1 min. at 160°C.

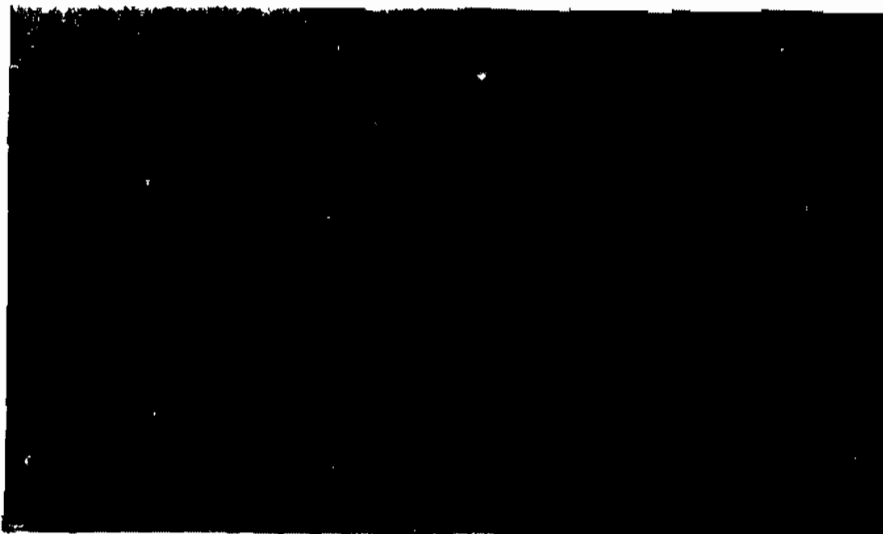


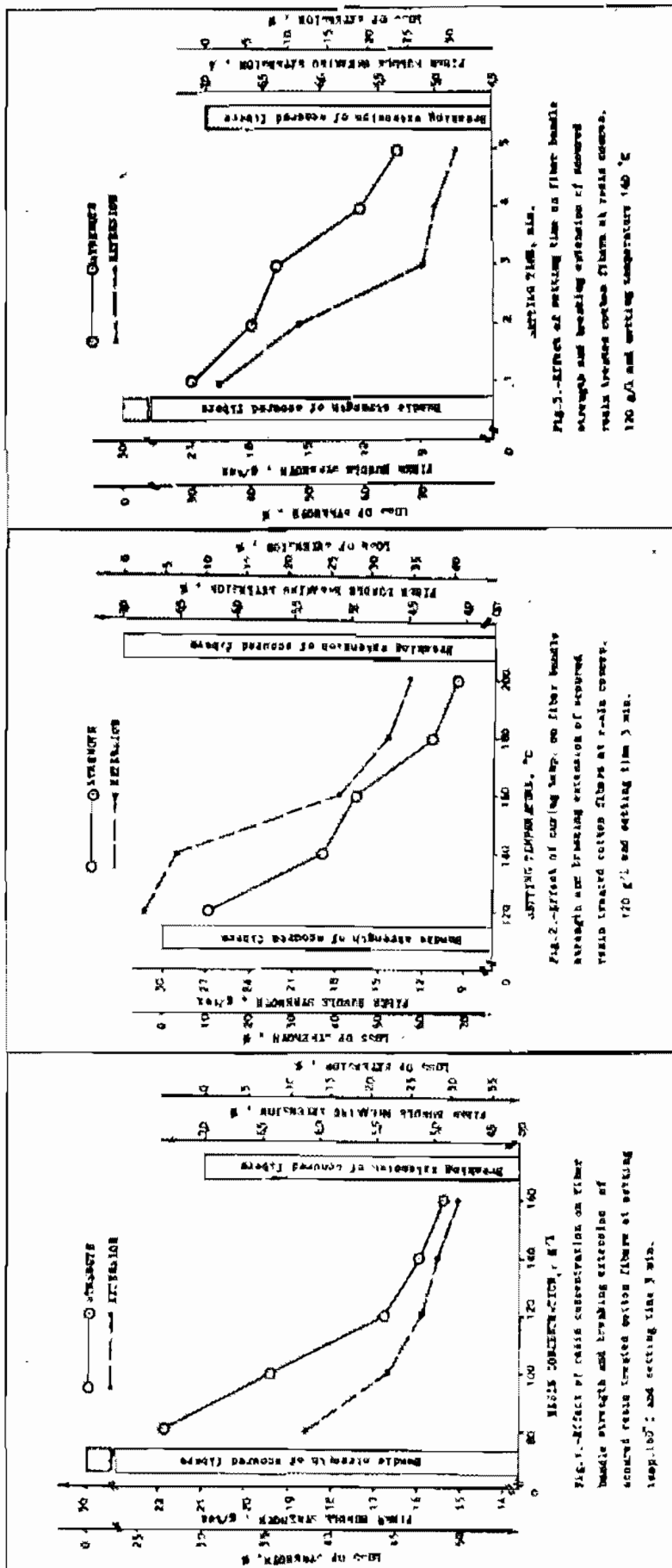
Plate 10: Cotton fibers treated with finishing agent (120 g/l) for 2 min. at 160°C.



Plate 11: Cotton fibers treated with finishing agent (120 g/l) for 4 min. at 160°C.



Plate 12: Cotton fibers treated with finishing agent (120 g/l) for 5 min. at 160°C.



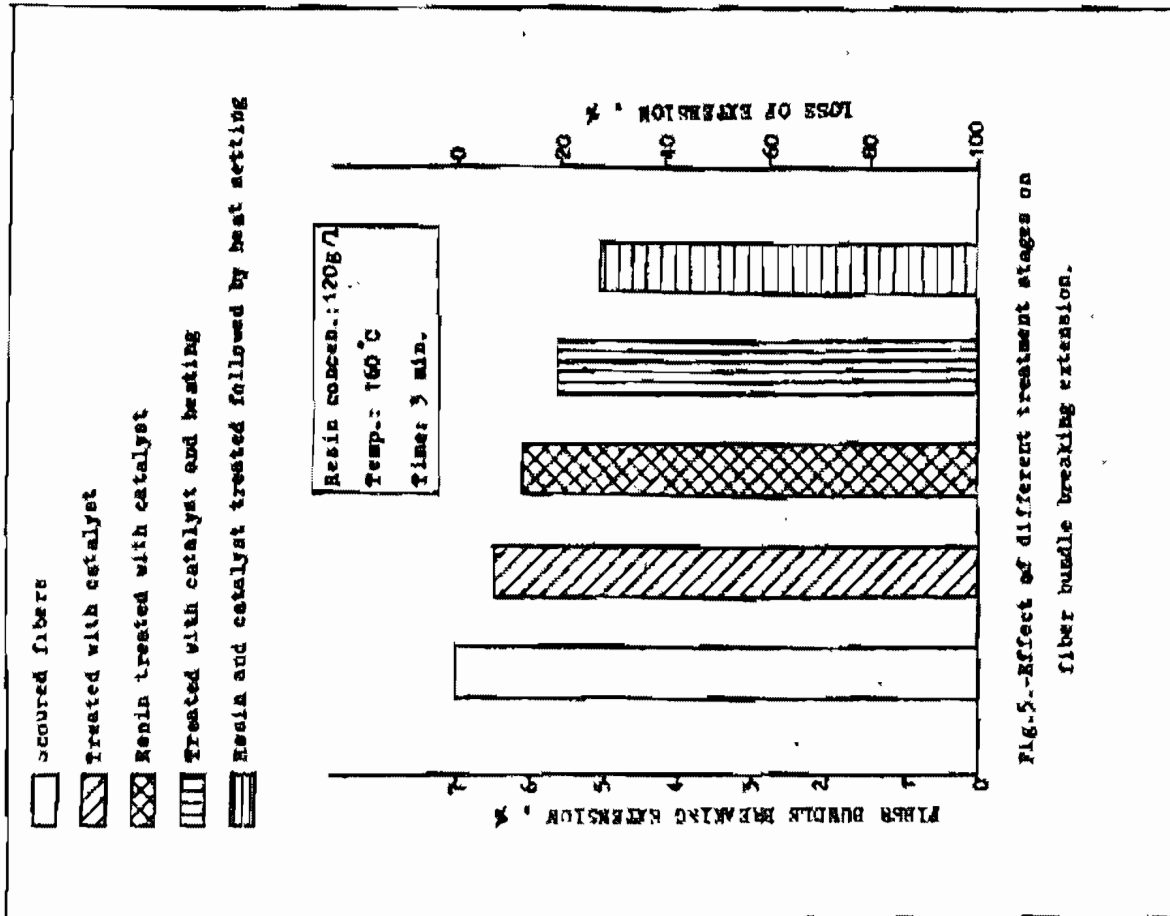


Fig.5.-Effect of different treatment stages on fiber bundle breaking extension.

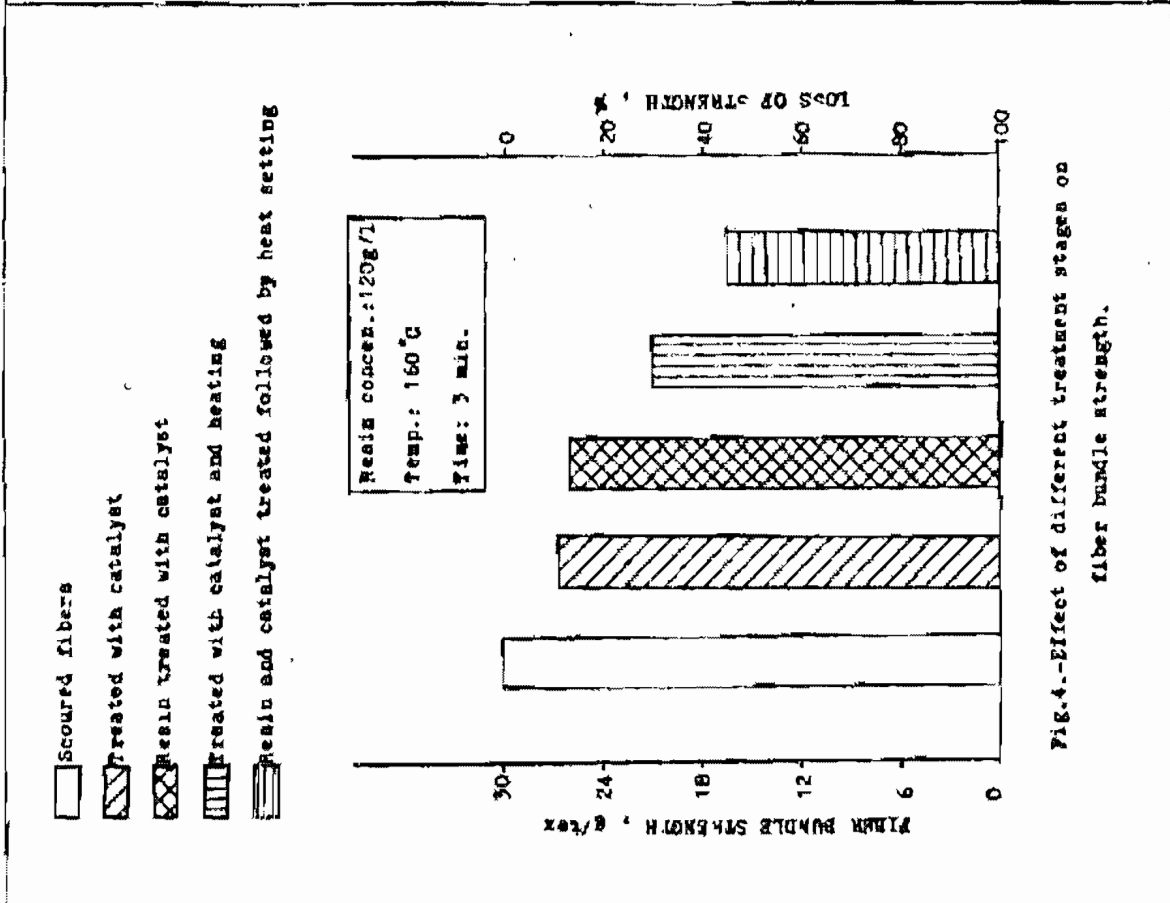


Fig.4.-Effect of different treatment stages on fiber bundle strength.