

NOVEL APPROACH FOR PREDICTING
DEGRADATION OF COTTON FABRICS

مدخل جديد لتقدير تلف الاقمشة القطنية

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الخلاصة

أثبتت التجارب العملية أن الزيادة في النسبة المئوية لتلف الاقمشة القطنية المختبرة تكون متسوية بانخفاض في معامل قياس درجة البلمرة المقترحة.

ثبت أن أعلى قيم تلف للاقمشة القطنية المجهزة سجلت في مرحلتى التبييض والطبءة فحين أقلها سجل في القماش الخام والمنسول.

الطريقة الميكروسكوبية الجديدة سهلة وموفرة للجهد والوقت وثبت ارتباطها بالطرق الكيماوية المعروفة لتقدير تلف الاقمشة القطنية.

ABSTRACT

The results indicated that the increase of percentage of damage of tested cotton fabrics was accompanied by a drop in suggested degree of polymerization (DP). For finished fabrics, the maximum increase in percentage of damage was higher with bleached and printed fabrics than that of grey and scoured fabrics. It was found that the higher the percentage of damage, the lower the suggested (DP.) value of tested cotton fabrics and vice-versa.

Also it has been found that, microscopically the percentage of damage could be used in the degradation measurements instead of copper number, fluidity, and methylene-blue absorption test, both gave results that are strongly correlated to each other. The novel method is easy and saves much time and effort.

1. INTRODUCTION

The textile industry plays a major role in the economic development of Egypt. This type of industry faces various problems in its production systems. These problems should be taken by scientific approaches to achieve practical results that would help improve the technical as well as the economical aspects of the manufacturing processes.

The textile industry in Egypt is looking forward for more production, but this could not be achieved just by increasing the speeds of the machines, because the fibre may not respond to the mechanical strains imposed on it due to the high speeds. Also it is required to improve the properties of the fibre through the chemical treatments.

This and the above could not be achieved unless enough knowledge is available about the structure of the fibre and its behaviour when subjected to mechanical or chemical actions.

Lots of discussions were made among specialists in the Egyptian textile industry about damage of cotton fibres. It is propable that some degradation is caused by the standard cleaning, spinning, weaving, and finishing processes. But

generally it is known that the chemical treatments in the finishing processes really affect the fibre. The chances of serious deterioration are much greater.

It is evident from the experience in the industry that under certain circumstances cotton fibres are exposed to both physical and chemical damage, unless operations are carefully controlled.

1.1 Purpose

The purpose of the present investigation is to investigate the possibility of using the light microscope with conjunction with iodo-zinc chloride swelling agent in predicting the degree of polymerization and percentage of damage of cotton fibre, when the cotton fabrics are subjected to pretreatment and dyeing processes.

The ultimate objective is to establish a new method for assessing the damage that may occur to the fibre by following up the behaviour of the fibre when placed in the swelling agent iodo-zinc chloride, especially the behaviour of both the primary wall and secondary wall of the fibre

1.2 Scope

The method of test in this paper covers the determination of the percentage of damage and suggested degree of polymerization value of a sample of loose cotton fibres whether raw, processed, or obtained from cotton fabrics.

2. GENERAL DESCRIPTION OF COTTON FIBRES.

Figure 1, shows schematic diagram of the cotton fibre given by Summer, showing major structure features and round shape of fibres before and after drying [1].

It is clear that the fibre consists of cuticle, primary wall, winding layer, secondary wall and lumen.

The primary wall is the outer skin of the cotton fibre. It consists of a network of cellulose microfibrils randomly interlaced and encrusted with noncellulosic materials. Chemical analysis of the primary wall shows about 50% cellulose and contains about 10% pectic material, 10% fatty material, 15% proteinaceous material [1].

Upon swelling in chemical reagents the net effect of this phenomenon is shrinkage of the primary wall sleeve into a constricting casing around the fibre.

The first layer of the secondary wall deposited inside the netlike primary wall of the cotton fibre is referred to as S-1 layer or winding layer of the fibre wall.

The secondary wall is considered to be the main body of the cotton fibre.

In Ref. [2] more about the secondary wall is given. According to this reference and as may be seen from Fig. 1 the bulk of the fibre is contained in the secondary wall S-2. In this layer the fibrils and the molecules are oriented helically and the angle of the helix is of the order of 30°. Generally it is believed that the angle remains constant at intervals along the fibre, the sense of the helix reverses.

2.1. General Longitudinal Appearance of Cotton Fibres [3].

Cotton fibre is a single cell and when examined under the microscope appears as an irregularly twisted and collapsed, flattened tube, with a central, or lumen through its length (Fig. 2a).

The Base: The basically, a cotton fibre is an elongated epidermal cell, the basal portion of which extends into one layer of cells on the surface of the seed coat.

The Body : The body of the fibre is characterized by its thickened wall and central canal. The spiral or convolutions are distinct twists about the longitudinal axis of the fibre, and the direction of the helix is frequently reversed. The body of the fibre constitutes the greatest part of its length.

The Tip : The distinguishing features of the tip convolutions. It is further differentiated by a tapered rodlike end.

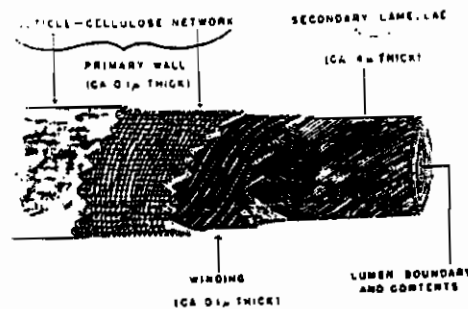


Figure 1. Schematic diagram of the cotton fibre given by Summer, showing major structure features and round shape of fibres before collapse and drying.

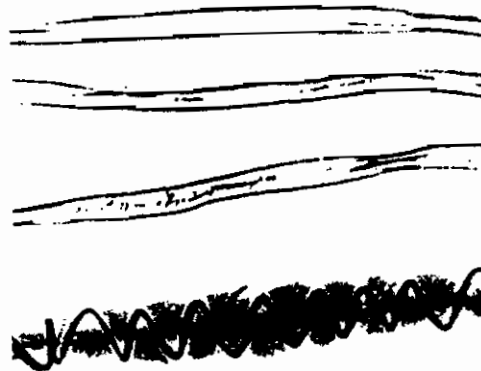


Figure 2. Longitudinal view of cotton fibre before and after swelling in iodo zinc chloride swelling agent. according to Barghash [10]

2.2 Swelling

According to Katz [4] , a solid is said to swell when it takes up a liquid whilst at the same time,

- a) it does not lose its apparent homogeneity,
- b) its dimensions are enlarged, and
- c) its cohesion is diminished.

The swelling phenomenon of cellulose are suitably subdivided into the following way :-

- i) intermicellar swelling involving limited and unlimited swelling,
- ii) intramicellar swelling involving limited and unlimited swelling.

The changes in nature and properties of native cellulose, which swelling treatment can cause include :-

- 1) reduction in fibre length,
- 2) better circular cross-section of fibre,
- 3) higher area of cross-section,
- 4) higher dye affinity,
- 5) higher moisture sorption,
- 6) higher chemical reactivity,
- 7) higher elongation at break,
- 8) lower density, and
- 9) reduction in weak places due to removal of structural reversals.

2.2.1. Swelling of cotton fibres.

Once the cotton fibre has been swollen by such solutions, in general, it never returns to the former compact state, and the swelling is permanent. In iodo-zinc chloride swelling agent complete swelling occurs to the cotton fibre. In practice there is also usually a certain amount of degradation, due to a proportion of the chains becoming broken, resulting in a lowering of the mean chain length. Swollen cotton behaves much the same as ordinary cotton, but chemical reactions such as hydrolysis, oxidation, and esterification processed faster, and physical reaction such as moisture absorption (regain), dyeing, staining with reagents, and absorption of inorganic salts processed farther [5].

3- BASIC METHODS OF FIBRE DAMAGE

The purpose of the present part is to cover some of the various methods used [2,5, and 6] to estimate the nature and the amount of damage that is likely to be suffered by cotton during mill processing, during pretreatments and finishing processes.

There are many qualitative tests in the literature to assess the damage of cellulosic fibres but not all of them are reliable nor more reliable than to be reviewed in some details.

3.1 Quantitative tests [5]

3.1.1. Fluidity in cuprammonium solution.

It is known for many reasons that the viscosity of solutions of long chain compounds is related to the mean chain length, long chains giving high viscosity, and short chains low viscosity. The viscosity of solutions in cuprammonium is therefore a measure of chemical damage.

Fluidity is the reciprocal of viscosity, and as it is desirable to have a high number representing great damage and a low number for small damage.

Viscosity is the resistance of a liquid to flow, and is measured in dynes per square centimetre per cm. per second - i.e. in "poises". If V = viscosity in poises, and F = fluidity in reciprocal poises, then

$$F = \frac{1}{V} \quad (1)$$

3.1.2. Copper number [5]

The copper number is the weight of copper reduced by 100 gms. of a sample of cellulose fibre from the cupric to the cuprous state.

It is determined by a quantitative modification of Fehling's test. The original Fehling's solution was modified as follows:-

Solution A. 100 gms $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ per litre, Solution B. 50 gms. Na_2HCO_3 , 350 gms. Na_2CO_3 , $10\text{H}_2\text{O}$ to 1 litre.

Solution C. 100 gms iron alum, 140 c.c. conc. H_2SO_4 to 1 litre. The procedure of carrying this test is given in Ref. [5].

3.1.3. Methylene-blue absorption [5]

The amount of methylene-blue which is absorbed by a sample of cotton under the standard conditions is proportional to either (a) the percentage of COOH groups presents after alkaline oxidation, or (b) by residual mineral acids which have produced hydrocellulose by drying in. Pure cotton, bleached to remove protein and pectin, and washed to remove salts, does not absorb methylene-blue. Absorption indicates pectin, protein, alkali, oxycellulose, certain type of cotton, and soape residues. A low absorption figure may be given by heavily damaged cotton.

The disadvantages of these methods are:-

- 1- They are tedious and lengthy,
- 2- All the methods should be carried out to asses deterioration and degradation,
- 3- None of these methods can give reliable informations about the location of damage in the fibre,
- 4- Non of these methods can detect the kind of degradation whether chemical or mechanical.

Thus it is obvious that a rapid and simple method for assessing damage of cotton fabrics is badly needed. The present investigation was undertaken to fill this gap.

4. EXPERIMENTAL PART

4.1 Experimental Methods

4.1.1 Cotton fabric construction

The testing cotton fabric is a woven fabric from single yarns spun from cotton fibres. The fabric has 18 threads/Cm. in the warp direction and 25 threds/ Cm. in the weft direction. The warp count is 23/1, and the weft count is 14.7/1 (English count). Fabric structure is plain weave 1/1.

Wet Processing : were carried out as follows:-

4.1.2 Treatment with sodium hydroxide

The above mensioned cotton fabric was treated at the boil at 90°C for one hour. With an aqueous solution of sodium hydroxide at a concentration of 20 g/l. The fabric was then thoroughly washed, neutralized with dilute acetic acid, thoroughly washed, and finally dried at ambient condition.

4.1.3. Hypochlorite treatment

Treatment of scoured cotton fabric with sodium hypochlorite was carried at 90°C , and PH. 7, for one hour. At the end of this period the fabric wash with drawn and thoroughly washed.

4.1.4. Printing treatment

Fabric was padded with an aqueous liquor containing the reactive dye urea and sodium carbonate. This liquor was kept cold to secure its stability. After padding fabric was batched and then stored at room temp. for 24 hours. The finished cotton fabric has a commercial name of "Ribs El-Nasr".

4.2. Determination the percentage of damage for cotton fabrics.

4.2.1. The random sample method

This method employs the same conditions and technique for fabric tensile properties as those prescribed in the standard method of test for fabric strength. Ten samples of 10x2.5 Cm. were cut from grey cotton fabric in diagonal direction. Both warp and weft threads were separated from these samples using a needle, and then collected together to form laboratory test sample.

4.2.2. Test specimen (fibres in form of powder).

The test specimen consists of approximately 1200 fibres divided among six slides, each slide containing approximately 200 fibres. Fibres can be prepared from laboratory test sample, using simple handmicrtome and blade.

4.2.3. Carrying the test

The simplest method of carrying out an examination is to clamp two blades together and make a double cut across a bundle of threads (warp and weft together). Cut sections of these threads gives loose cotton fibres (powder), about 170-200 μ m. long. The whole operation may be carried out on the microscope slide, using simple handmicrtome and a blade.

Cover the cut fibres with a cover glass slide and apply a drop of iodo-zinc chloride swelling agent at one corner of the cover glass slide while holding it down at the opposite corner. Tap the cover gently or remove air bubbles and excess solution.

Place the slide over heating disc for (3 min.) (approximately temp. is 62 °C), then classify the swollen cotton fibres as explained in Ref. [7, 8, and 9].

The characteristic appearance of a very short length of cut cotton fibres which is undamaged in the primary wall (n_1) is that of a "Dumbell". The fibre itself does not swell in width, but its contents extrude at the ends (see Fig. 3a).

If, however, the primary wall has been damaged by sever chemical action, it is too weak to resist the swelling action, and is disrupted more or less evenly over its whole surface, so that the fibre swells in diameter, but there is no extrusion (n_2), as shown in Fig.(3_c).

While medium swollen fibres (half-damaged) are intermediate between well swollen and unswollen cotton fibres in appearance (n_2), as shown in Fig.(3_b).

This procedure of preparing specime test sample (fibrs powder) was repeated again for scoured, bleached, and printed cotton fabrics.

4.2.4. Preparation of swelling agent [10].

The swelling agent (iodo-zinc chloride) used consists of $ZnCl_2$ (100 g), KI (32 g), 34 ml. water, and Iodid till saturation

4.2.5. New "Dumbell" test

The "Dumbell" test described in Ref. [6, 7, 8, 9, and 11] was used to assesses the percentage of damage and/or suggested degree of polymerization value has

occurred to the cotton fibres, using iodo-zinc chloride swelling agent instead of caustic soda 15%.

4.2.6. Microscopic examination

A light microscope, with heating disc was used. All measurements were conducted at constant slide temperature of 62 °C [12].

4.2.7. Suggested degree of polymerization (DP).

According to Ref. [7 and 13] the microscopic degree of polymerization DP, value may be calculated from the following equation :

$$DP = \frac{2500 n_1 + 800 n_2 + 500 n_3}{n_1 + n_2 + n_3} \quad (2)$$

where

n_1 = no of well swollen fibres (undamaged fibers),

n_2 = no. of medium swollen fibres (half-damaged fibres),

n_3 = no. of unswollen fibres (damaged fibres),

2500, 800, and 500 = DP. values for n_1 , n_2 , and n_3 respectively according to Ref. [13]

4.2.8. Percentage of damage.

This test may be made quantitative by counting the number of unswollen cotton fibres (damaged fibres - n_3) and expressing them as a percentage of the total number of tested fibres (n). At least 1200 fibres should be counted for this purpose, but the counting is very rapid with the aid of an automatic counter of the type which clicks over by pressure with the finger.

The percentage of fibre damage (%D) was calculated from the equation:

$$\%D = \frac{n_3}{n} \times 100 \quad (3)$$

where

n = total number of examined cotton fibres, i.e. $n_1 + n_2 + n_3$.

4.3. Calculation and report.

The recording and calculation of data are facilitated by the use of a worksheet for each test specimen as shown in sheet (1). Calculate the percentage of fabric damage and/or microscopic degree of polymerization value as follows:-

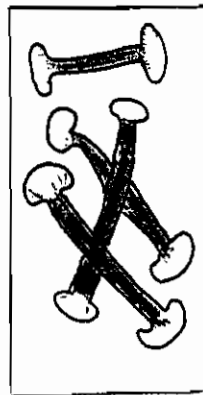
$$\% D = \frac{n_3}{n} \times 100 \quad (3.1)$$

$$DP = \frac{2500 n_1 + 800 n_2 + 500 n_3}{n} \quad (2.1)$$

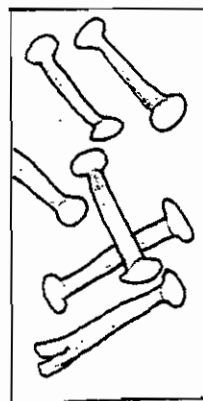
where

n_1 = total number of undamaged fibre (column 2),

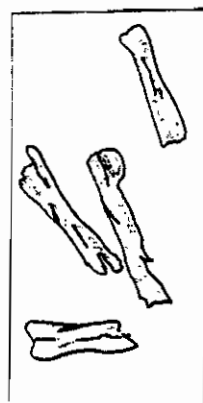
n_2 = total number of half-damaged fibre (column 3),



3_a, DP = 2500 [13]



3_b, DP = 800 [13]



3_c, DP = 500 [13]

Figure 3. Forms of Cotton in new "Dumbell" Test "Fibres are immersed in iodo-zinich chloride swelling agent [7 and 11].

3_a. Type 1 (n_1) secondary wall bulges remarkably in both sides of the fibres (undamaged cotton fibres).

3_b. Type 2 (n_2) secondary wall shows less bulging in both sides of the fibres (half, damaged cotton fibres), and

3_c. Type 3 (n_3) No bulging of the secondary wall (damaged cotton fibres).

TABLE I
RESULTS OF CHEMICAL TESTS

Type of test	Type of Fabrics			
	Grey Fabric	Scoured Fabric	Bleached Fabric	Printed Fabric
Fluidity	8.3	12	12.6	—
Copper number	—	0.2	0.25	0.2
Methylene - blue	- (ive)	- (ive)	- (ive)	- (ive)

TABLE II
TESTED COTTON FABRIC SPECIFICATIONS.

Type of test	Type of Fabrics			
	Grey Fabric	Scoured Fabric	Bleached Fabric	Printed Fabric
Tensile strength in warp direction(kg)	52	40	46	43
Tensile strength in weft direction (kg)	22	30	18	32
Average fabric tensile strength	27	35	32	37.5
No. of end / Cm.	25	28	28	28
No. of picks/Cm.	18	19	16	18

n_3 = total number of damaged fibre (column 4),
 %D = Percentage of fabric damage (column 5), and
 DP = Suggested DP value

SHEET 1. SUGGESTED FORM FOR REPORTING TEST DATA ON DAMAGE OF COTTON FABRICS.

Test No. Date :

Sample Marking and Description

Slide No.	n_1	n_2	n_3	$n = n_1 + n_2 + n_3$	$\% D_i = \frac{n_3}{n}$
1					
2					
.					
..					
.					
.					
.					
.					
.					
.					
n					
	n_1	n_2	n_3	n	D

5. RESULTS AND DISCUSSION

For the mentioned cotton fabrics the general trend is that average fabric tensile strength tends to increase with the increasing in number of ends and picks per cm. (relative to that measured for grey fabric).

Results of the chemical tests are given in Table 1. It was found that fluidity test can not give the viscosity value for printed cotton fabric, also its value effected by sizing and printing materials.

Using copper number test, the value of it for scoured, bleached, and printed cotton fabrics are 0.2, 0.25, and 0.2 respectively. It is clear that the differences between them are very small. Also it was found that the copper number test can not give information about the damage may be occurred to grey fabric.

Using methylene-blue test we find that grey, scoured, bleached, and printed cotton fabrics have the same value.

The microscopic degree of polymerization (DP) ranges between 2316 for grey fabric and 2110 which has been obtained when the scoured and bleached cotton fabric was printed. It is evident from Fig. 5 that generally high values of (DP) are associated with low values of fluidity (F) and vice-versa

Plotted in Fig. 4 are the values of fluidity (F) versus percentage of damage

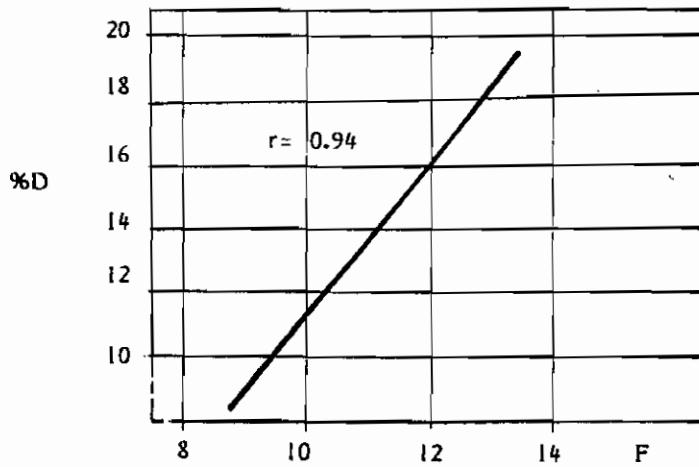


Figure 4. Shows values of percentage of fibre damage (%D) versus fabric fluidity values.

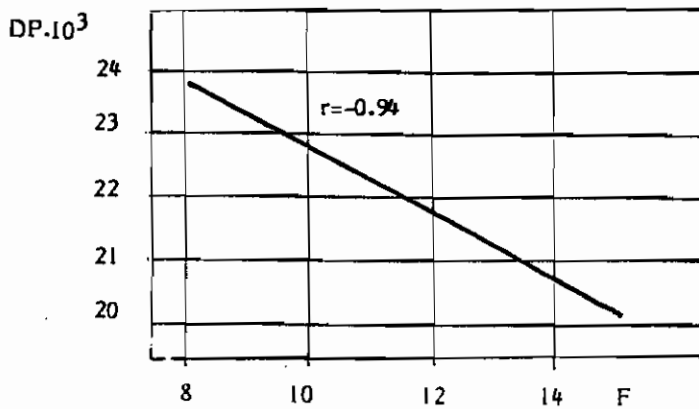


Figure 5. Shows values of suggested degree of polymerization versus fabric fluidity values.

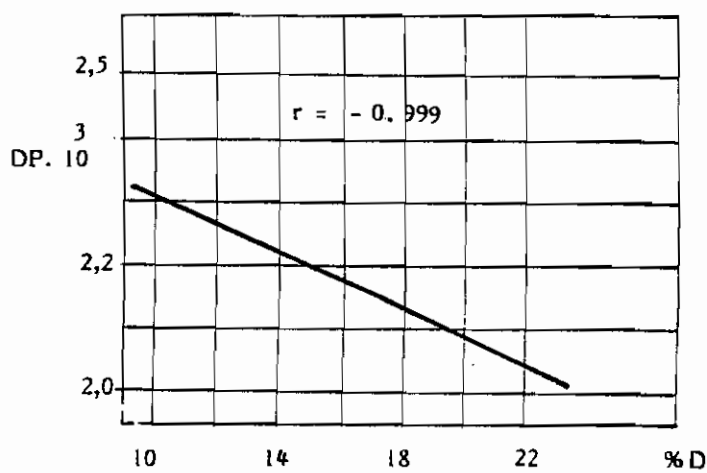


Figure 6. Shows values of suggested degree of polymerization versus fabric percentage of damage.

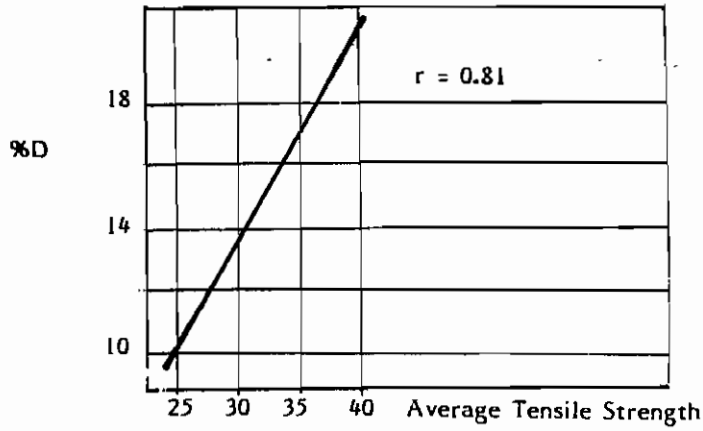


Figure 7. Shows values of average fabric tensile strength versus fabric percentage of damage.

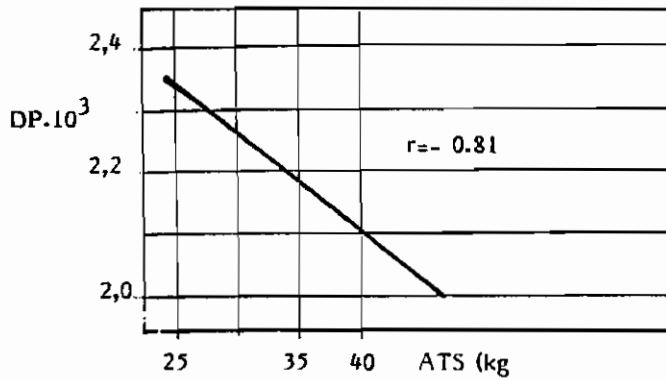


Figure 8. Shows values of average fabric tensile strength versus values of microscopic degree of polymerization

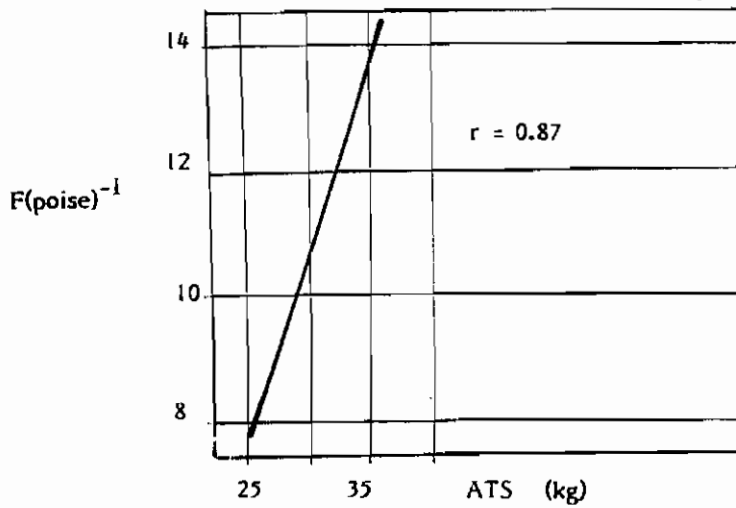


Figure 9. Shows values of average fabric tensile strength versus fabric fluidity values.

(%D). Statistical analysis has shown that percentage of damage is positively correlated with the fluidity values which is basically determined from chemical tests of fabrics. The correlation coefficient (r) is 0.94 and significant at the 5% level.

It was found from Fig. 5 that results of fibre damage (%D) and microscopical degree of polymerization values, gives a high ranking correlation coefficient ($R = 1$), which is not un-expected since both measurements describe the same character of cotton fabric.

Plotted in Fig. 6 the values of the %D versus values of DP, interesting to observe that generally the %D tends to increase with the decrease of DP values. Statistical analysis has shown that DP is negatively correlated with %D, which is basically determined from microscopical examination of fibres. The correlation coefficient (r) is -0.999. This result and the result obtained between DP and fluidity and/or between %D and fluidity, indicated that a relationship would exist between the two microscopical determinations, i.e. the DP and %D.

In fact this result pointed to the suitability of using the %D and/or DP as a quick measure for fabric damage, since only one type of fibres (n_3) is counted under the microscope instead of counting each type n_1 , n_2 , and n_3 as in case of DP value. This will save time and effort but the microscopic degree of polymerization, (DP) value has the advantage of considering all types of fibres in tested sample, which corresponds to the results obtained for fabric average tensile strength (AFS), as shown in Figures 7 and 8.

Plotted in Fig. 9 are the values of average fabric strength (AFS) versus fabric fluidity values, interesting that (F) values are in good correlation with (AFS) values. The correlation coefficient (r) is 0.87 and high significant at 5% level.

6. CONCLUSIONS AND REMARKS

It will be helpful in interpreting results of examinations by provided the following points :-

- a) The microscopic degree of polymerization (DP) is reduced in both bleached and printed cotton fabric, and average tensile strength and viscosity of fabrics are lowered.
- b) The microscopic degree of polymerization may not be reduced in pretreatment processes, such as desizing and scouring. The tensile strength may therefore not be reduced, but the viscosity in cuprammonium is reduced because of the effect of the alkali in the solvent.
- c) The microscopic degree of polymerization after bleach process and especially in dyed fabrics is reduced and there is therefore a loss of tensile strength and reduction in viscosity.
- d) The percentage of damage (%D) of tested cotton fabric ranges between 9.2% to 19.5%. The higher the %D, the lower the DP, and vice-versa.
- e) It was found that every 1% of %D values is equal to approximately 20 units of DP., and equal to four units of fluidity test values.

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