

TESTING OF COTTON FIBRES  
 USING MICROSCOPIC MEASUREMENTS  
 اختبار الشعيرات القطنية باستخدام  
 القياسات الميكروسكوبية  
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الخلاصة:

أستخدم الميكروسكوب الضوئي العادي ومحلول كلوريد الزنك اليودي كحلول انتفاخ في تقدير العديد من خواص الشعيرات القطنية قبل وبعد معالجتها كيميائيا أو ميكانيكيا. والقياسات الميكروسكوبية التي أمكن استخدام الاسلوب السابق في تقديرها هي: - تقدير نضوجة الشعيرات القطنية - متوسط عدد الالتواءات في وحدة الطول - النسبة المئوية للتلف - رقم الجودة للشعيرات المختبرة - متوسط عدد الانتفاخات في وحدة الطول وعلاقتها بمتانة الشد للشعيرات المختبرة. والاسلوب المقترح سهل وغير مكلف ويساعد على قيام دراسات أخرى حول تأثير العمليات السابقة لتجهيز الأقمشة القطنية على كل من النسبة المئوية للتلف وكذا رقم جودة الأقمشة المختبرة.

ABSTRACT: Light microscope in conjunction with iodo-zinc chloride swelling agent were used to monitor several properties of cotton fibre before and after the latter were subjected to degradation treatments. Among properties measured were maturity, convolution frequency, percentage of damage and quality number of cotton fibres. Hence it was possible to assess the total effect of mechanical and/or chemical treatments on the physical properties of cotton fibres. The technique adopted fast and inexpensive. It enables studies to be made of the effects of pretreatment and finishing processes on damage and quality of cotton fabrics.

KEYWORDS

Quality number, Maturity, Convolution frequency, % of damage, Tensile strength, Number of beads, and Compression modulus.

1. INTRODUCTION

Cotton is perhaps the most widely used textile fiber all over the world. This preeminence is due to a happy combination of properties such as abundance, fine cross section, high strength and durability, ability to absorb moisture, easy dyeability, etc.

Cotton fiber is the purest form of natural cellulose and has very little lignin or pectin compounded with the cellulose as flax, jute, hemp or wood. However it still contains several unwanted impurities (1). Nearly 6% of the fiber weight is impurities that need to be removed to prepare cotton for most textile uses. Beside the natural impurities, cotton fabrics in a loom state contains impurities such as oil and sizing material which have been added to cotton to facilitate spinning and weaving.

The need for removal of impurities is obvious to make grey fabrics white and absorbent and to prepare them for dyeing, printing or chemical finishing. In practice, the grey cotton fabrics after singeing are subject

to a number of wet processing viz; desizing, scouring (or kier boiling), mercerizing and bleaching, to remove impurities. Except mercerizing these processes involve chemical treatments at high temperature and considerable length of time. Also dyeing, printing and chemical finishing, the fabrics are exposed to several conditions of temperatures and chemicals.

The effect of these various treatments is to bring about deterioration and degradation of the cotton fiber in the fabric. This and the fact that these treatments are performed under different tensions on fabrics previously suffered from tension during spinning and weaving necessitate measurement of deterioration and degradation of cotton fabric.

Deterioration of the fiber is usually assessed by strength measurement whereas fiber degradation is assessed by determination of degree of polymerization (2), or fluidity (3), copper number and carboxyl group (4). The disadvantages of these methods are:

- 1- They are tedious and lengthy.
- 2- All the methods should preferably be carried out to assess deterioration and degradation.
- 3- Non of these methods can give reliable informations about the location of damage in the fiber.
- 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 degradation and deterioration of fibers is badly needed. The present work was undertaken to fill this gap.

### 1.1 Purpose

The purpose of the present investigation is to investigate the possibility of using the optical microscope in assessing and predicting the strength and/or quality of cotton fiber, when the fiber is subjected to chemical and/or mechanical action.

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

## 2. EXPERIMENTAL ARRANGEMENT AND TECHNIQUE.

### 2.1 Raw Material

Varieties of Egyptian and Sudanese cotton fibers which are commercially available, have been used. The commercial cotton fibers are Giza 70, Giza 45, Giza 75, Giza 77, Giza 74, Karnek, Barakat 1, Barakat 2, Gizera 1, Gizera 2, Barakat 5, and Up-land. In preparing test specimens for this investigation, the method described in Ref. (5) was used.

### 2.2 Microscopic Examination

A light microscope with heating disc was used. All the measurements were conducted at constant slide temperature of 62°C (6).

### 2.3 Maturity Percentage

The percent maturity values (M) were determined by the sodium hydroxide method according to ASTM D 1442-75T.

### 2.4 Fiber Bundle Strength

The tensile strength was measured using Pressley Strength Tester at zero gauge. The % drop in bundle strength "L%" was calculated from the equation:

$$L\% = \left(1 - \frac{(P.I.)_t}{(P.I.)_0}\right) \times 100 \quad \dots\dots(1)$$

where  $(P.I.)_t$  = fiber bundle strength of treated sample.

$(P.I.)_0$  = fiber bundle strength of untreated sample.

### 2.5 Preparation of Swelling Agent

The swelling agent used consists of  $ZnCl_2$ , (100g), KI (32g), (34 ml) distilled water, and  $I_2$  till saturation (7).

### 2.6 New Dumbell Test

The Dumbell test described in Ref. (8, 9, 4, and 10) was used to assess the damage that has been occurred to the fibers using iodo-zinc chloride swelling agent instead of caustic soda 15%.

## 3. RESULTS AND DISCUSSION

### 3.1 Mathematical Treatment For Assessing The Damage of Egyptian and Sudanese Cottons:

The purpose of the present part of this paper is to show how one could rely on light microscope for, assessing the damage that may occur to the cotton fiber at any stage of processing or when stored. The technique proposed here is simple and may prove it's suitability for the routine tests in the industry.

The technique is based on the fact that when well developed cotton fiber is placed in swelling agents, it forms beads or balloons. The number of these was found to be dependent on the type of cotton, but independent of tested fiber length for each type of cotton, (lengths of 2000  $\mu$  and above). It is understandable that the swelling agent used for this test was the zinc-chloride and iodine dissolved in water.

Based on the number of beads fibre damage may be calculated mathematically by using one of the two following equation:

$$\% \text{ Damage} = \frac{N_0 - N_t}{N_0} \times 100 \quad \dots\dots(2)$$

$$\text{and } \% \text{ Damage} = \frac{T_{so} - T_t}{T_{so}} \times 100 \quad \dots\dots(3)$$

where  $N_0$  = number of beads formed in the original untreated fiber.

$N_t$  = number of beads formed in the treated fiber,

$T_{so}$  = time of swelling for untreated fiber, and

$T_t$  = time of swelling for treated fiber.

One has to know that in Eq.(3) the fiber has to be observed under the microscope to follow up the formation of beads till it reaches it's maximum number. For each type of cotton this time was recorded and used. Eq.(2) would give different values for % damage if sample of fibers of lengths less than 1000  $\mu$ , is used, but fairly constant values for lengths of 2000  $\mu$ , and above this equation could be written in the form of:

$$\% \text{ Damage} = \frac{(\bar{N}_0/L) - (\bar{N}/L)}{\bar{N}_0/L} \times 100 \quad \dots\dots(4)$$

According to Eq.(4) when  $\bar{N}/L = \text{zero}$ , then the percentage damage is 100% which means full damage of the primary wall, and when  $\bar{N}_0/L$  is equal to  $\bar{N}/L$ , then the percentage damage is zero, i.e. the fiber shows no damage in the primary wall.

### 3.2 Location of Damage

The light microscope technique could be used in detecting the location of mechanical damage that may occur to the fiber when the fiber is exposed to mechanical strains. It is expected, that high swelling of the secondary wall occur in portions of the fibers which suffered most, while the impaired portions would show much less swelling. This would be reflected also on the number of beads formed. This could be seen from photograph (1).

In the case of chemical damage, it is very difficult to locate the damage, since the damage usually occurs in the entire fiber.

### 3.3 Kind of Damage

It is quite easy to distinguish between the chemical and mechanical damage by making use of longitudinal swelling views of cotton fiber, using the light microscope. As shown in Fig. 1 (photos 1a and 1b) the number of beads in mechanically degraded fibers is greater than those chemically degraded fibers.

### 3.4 Fiber Maturity

Maturity of cotton fiber is a determining factor in the overall quality of cotton. No commercial cotton has ever been found to be 100% mature. However, in common practice, a normal cotton with a maturity falls as low as 50-60% range, processing decreases as indicated by increased ends down in spinning, and decreased product quality are observed.

The measurement of fiber maturity is principally based on microscopical tests. It is known that mature fiber would show considerable swelling. Fig. 2 shows mature fibers which appear almost cylindrical when swelled, while the immature fiber are of corkscrew shape.

If a fiber is well developed it's maturity would be expected to be high, and hence would show high strength, and vice-versa. This would appear from strength measurements of fiber bundle strength as will be seen later.

### 3.4.1 Additional Parameter for Assessing maturity of cotton fibers:

The number of convolutions/unit length of fiber could be used as a measure of fiber maturity, especially for raw cottons. This is a purely microscopical test. In preparing test specimens for this test, the method described in Ref. 5 was used.

#### 3.4.1.1 Results of convolution frequency:

When a fiber is viewed through a microscope, the most obvious characteristic is, that generally flat band structure with corkscrew-like twisting. These twists are designated as convolutions. Convolution generation is intimately related to internal structure and secondary wall thickness. Very thin-walled cottons generally are lacking in convolutions. It was found, that the convolution frequency varies between 3.414, 3.244, 3.347, 2.876, and 2.762 per mm for Giza 45, Giza 75, Barakat 1, Barakat 2, and Up-land cottons respectively.

A linear regression analysis between the microscopically measured convolution frequency and per cent maturity was found in the form of:

$$\bar{n} = 0.027 M + 0.959 \quad \dots\dots(5)$$

Given in Table 1, are results of per cent maturity and convolution frequency, which give a high ranking correlation coefficient ( $R = 0.943$ ), which is not unexpected since both measurements describe the same character of cotton fiber.

Table 1: Relationship between fiber Maturity (M) and Convolution Frequency ( $\bar{n}$ ).

Type of cotton	Percent maturity(M)%	Convolution frequency( $\bar{n}$ )
Giza 45	91	3.414
Giza 75	89	3.244
Gizera 1	76	3.185
Barakat 1	81	3.347
Barakat 2	74	2.876
Up-land	72	2.762

#### 3.4.1.2 Results of Compression Modulus:

The compression modulus<sub>2</sub> was measured at twelve pressure ranging between 0.2 and 104.2 g/cm<sup>2</sup> using the Shirley Thickness Gauge with largest foot (area = 50 cm<sup>2</sup>) and fiber attachment (5).

The compression modulus could be determined according to the equation:

$$C.M. = \frac{t_1 (P_2 - P_1)}{t_1 - t_2} \quad \dots\dots(6)$$

where  $t_1$  and  $t_2$  are losses in cotton fiber thickness measured at two arbitrary pressure  $P_2$  and  $P_1$  respectively. According to Ref.(5) it was found that the ranking correlation coefficient between maturity percentage (%M) and C.M. is 0.58, i.e. the correlation between these two measurements is not strong as that found between %M and convolution frequency ( $\bar{n}$ ).

### 3.5 Relationship between Average Number of Beads and Tensile Strength:

The idea of the present work is based on a simple fact that when a cotton fiber is placed in a swelling agent the primary wall splits in different ways as shown in Ref. 11, and at the places at which bursting occurred the secondary wall bulges out and forms what we call beads. All attempts made are related to the number of beads formed and tensile strength of the fiber (actually in the form of a bundle).

Therefore by following the changes in the number of beads formed after swelling one could judge on the change of fiber strength. It is expected that as the number of beads decreases after any chemical or mechanical action, the fiber would show less strength. This is due to the fact that as the primary wall splits more, its continuity diminishes, and its share to tensile strength of the fiber decreases.

Also we are going to show that the number of beads formed and the tensile strength of a wide variety of Egyptian and Sudanese cottons are well related irrespective of the variety of cotton.

### 3.6 Effect of Pretreatment and Dyeing of Strength and Quality of Cotton/9/.

To examine the influence of chemical treatment on the strength of cotton, samples of Giza 75 cotton have been scoured, bleached, then dyed with 7 types of commercial dyes, that widely used in dyeing mills. The change (if any) because of these treatment have been followed microscopically and mechanically by measuring the bundle strength (Pressely Index, P.I.).

In microscopic tests the dumbbell test described in 2.6 of this investigation was used to assess the fiber damage. Microscopical investigations using the dumbbell test have shown that when the cotton fibers (dyed or not dyed) are placed in iodo zinc chloride solution they show the forms shown in Fig. 3, with different proportion in the specimen examined.

In the mathematical analysis of assessing the quality of the fibers at any chemical treatment, type 1 was counted in the sample examined as  $n_1$ , while type 2 was counted as  $n_2$ , and type 3 was counted as  $n_3$ , and the total number of fibers examined is  $n$ , where

$$n = n_1 + n_2 + n_3 \quad \dots\dots(7)$$

The quality number (Q.N.) which is used in the present work as a microscopic measure for quality is calculated by proportion from the following equation:

$$Q.N. = \left( \frac{n_1}{n} + \frac{n_2}{2n} + \frac{n_3}{3n} \right) \times 100 \quad \dots\dots(8)$$

In another mathematical analysis for the same test the % of damaged fibres, i.e. type 3 only in which no bulging of the secondary wall is observed was used as a measure of the quality of treated cotton under consideration. The % damaged fibres is calculated from the equation:

$$\% D = \frac{n_3 \text{ Damaged}}{n \text{ Total}} \times 100 \quad \dots\dots(9)$$

Plotted in Figure 4 are the values of strength (P.I.) versus quality number (Q.N.). Statistical analysis has shown that tensile strength is positively correlated with the quality number which is basically determined from microscopical examination of fibers. The correlation coefficient ( $r$ ) is 0.82.

Table 2: Values of Quality Number (Q.N.), % Damaged Fibres (%D) and Fibre Bundle Strength (P.I.) For Giza 75.

Treatment	Strength (P.I.)	% Damage (%)	Quality Number (Q.N. %)	% Drop (P.I.)	% Drop QN.	% Increase in damage % D
Raw ginned	9.9	4.6	93	0	0	0
Scoured	9.5	9.8	84	4.04	9.7	1.1
Bleached	9.4	17.5	70	5.05	24.7	2.8
Dyed with Levafix	9.1	33.6	65	8.08	30.1	6.3
Dyed with Besilen	8.8	32.0	62	11.11	33.3	5.9
Dyed with Procion	9.0	30.5	60	9.09	35.5	5.6
Dyed with Drimarene	8.9	31.0	59	10.10	36.6	5.7
Dyed with Direct	9.2	26.6	66	7.07	29.0	4.8
Dyed with Naphtol	9.2	31.7	64	7.07	31.2	5.9
Dyed with Vat	9.2	26.7	62	7.07	33.3	4.8

The Q.N. ranges between 93.0% (for raw ginned cotton) and 59% which has been obtained when the scoured and bleached Giza 75 cotton was dyed by Drimarene. (See Table 2). It is evident from Table 2 that generally high values of Q.N. are associated with low values of % damaged fibers and vice-versa. In fact this result pointed to the suitability of using the % damage as a quick measure for fiber damage, since only one type of fibers (type 3) is counted under the microscope, instead of counting each type. This will save time and effort, but the Q.N. has the advantage of considering all fibers in the tested sample, which corresponds to the result obtained for strength, where the average strength of fiber bundle is recorded.

### 3.7 Relation Between Fiber Bundle Strength, Fiber Maturity, Number of Convolution and Average Number of Beads for Raw Cottons:

To examine the relationship between fiber bundle strength and properties that were basically determined from microscopical tests, seven Egyptian and Sudanese cottons were selected and examined in the raw state. These are Giza 77, Giza 70, Giza 75, Dandara, Barakat (B1 and B2), and Gizera 1 (G1).

Given in Table 3 are the values of the average number of beads ( $\bar{N}/L$ ), number of convolution ( $\bar{n}$ ), maturity and fiber bundle strength (P.I.). The average number of beads ranges between  $10.25 \times 10^{-3}$  and  $15 \times 10^{-3}$ , and higher for Egyptian cottons than for Sudanese cottons.

Plotted in Fig. 5 are the values of the average number of beads  $\bar{N}/L$  and fiber bundle strength (P.I.) for Egyptian and Sudanese cottons. From the figure one may observe that the two values are positively correlated, i.e. fibers showing high number of beads are also showing higher strength and vice-versa, and the correlation coefficient ( $r$ ) is 0.91. The Egyptian cotton shows higher strength and higher number of beads compared with that of Sudanese cottons.

Plotted in Fig. 6 are the values of fiber maturity and the average number of beads ( $\bar{N}/L$ ) for Egyptian and Sudanese cottons. Generally fibers showing higher maturity also show higher average number of beads and vice-versa. The correlation coefficient ( $r$ ) is 0.70 and significant at the 5% level.

Table 3: Values of Number of Beads, Number of Convolutions, Maturity, and Fiber Bundle Strength for Some Egyptian and Sudanese Cottons (Raw Cottons).

Property	Egyptian Cottons				Sudanese Cotton			
	Type of cotton	G 77	G 70	G 75	D	B 1	B 5	G 1
Number of beads $\bar{N}/L \times 10^{-3}$		15.0	12.3	12.5	11.8	11.35	11.08	10.25
Number of Convolutions (in 2000 $\mu$ length)		9.14	8.76	9.30	10.1	8.53	8.07	8.25
Maturity (Caustic Soda Solution)		88.2	88.7	81.8	82.1	85.10	71.40	73.70
Fiber Bundle Strength (P.I.)		10.97	10.04	9.98	9.0	7.82	7.50	6.62

### CONCLUSION

The microscopic technique was used to monitor the maturity, convolution frequency, fibre damage, quality number, average number of beads, compression modulus, and fibre strength. It was proved to be simple, fast, inexpensive, and highly reproducible test method for determining the quality and damage cotton fibres either in the raw or treated state, provided that there is no complete damage to the primary wall. Fibres which were considered mature showed high convolution frequency and low compression modulus.

It was also found that the higher the quality number, the higher the percentage of undamaged fibres in the sample and vice-versa.

The average number of beads formed in Egyptian and Sudanese cottons was strongly correlated to fibre bundle strength and fibre maturity. Generally fibres of high maturity and strength showed high number of beads.

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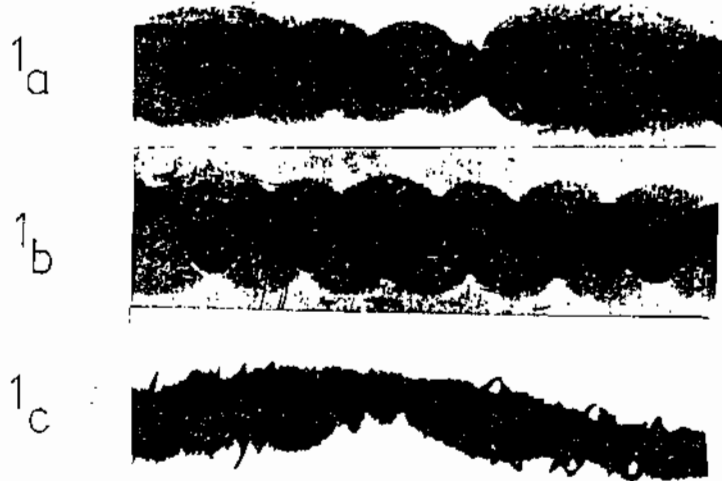


Fig. 1 Longitudinal view of fiber showing high swelling at the portion of which its suffered from mechanical and/or chemical degraded.

1<sub>a</sub>. Effect of carding on G70 cotton fiber, immersed in iodo-zinc-chloride swelling agent (200x).

1<sub>b</sub>. Effect of drawing on G70 cotton fiber, immersed in iodo-zinc-chloride swelling agent (200x).

1<sub>c</sub>. Effect of scouring, bleaching, and dyeing on G75 cotton fiber with Drimarene (200x).



Fig. 2 Longitudinal view of mature and immature cotton fibers.



Fig. 3 Forms of cotton in Dumbell test (fibres are immersed in iodo-zinc-chloride solution).

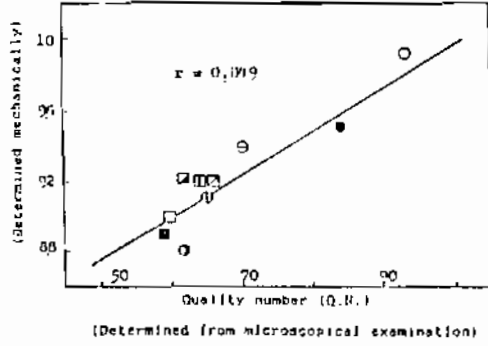


Fig. 4 Values of cotton fibre strength versus quality number for dyed Giza 75.

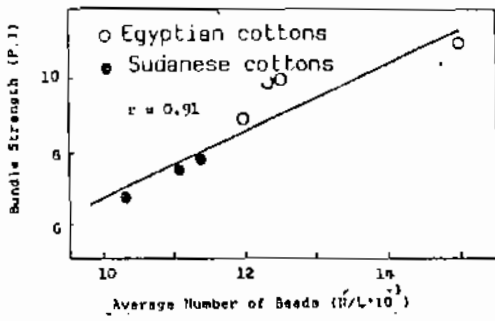


Fig. 5 Average number of beads versus fibre bundle strength.

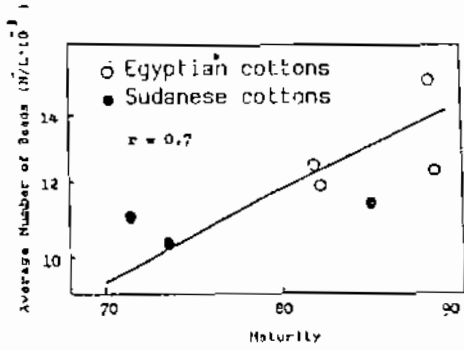


Fig. 6 Maturity of cotton fiber versus average number of beads.