

Effect of Dyeing Process on the Strength of Individual Wool Fibers Using Black Color Reactive Dye

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Abstract

This research paper investigates the influence of the dyeing process on the strength of wool fibers, specifically focusing on the use of reactive black dye. The study involved testing and measuring the average break in extension and breaking load of undyed, blank, and dyed samples with various average diameters. To assess the strength of fibers fifty separate fibers were selected for each sample group. The findings indicate that thinner fibers exhibit reduced breaking extension and lower breaking load compared to thicker fibers. Moreover, the average breaking elongation and breaking load of both fiber types decrease following the dyeing process. The results also indicate that as fiber diameter decreases, the decrease in breaking extension becomes more pronounced, while as fiber diameter increases, the decrease in breaking load becomes more pronounced. Notably, these results align with theoretical predictions and represent a significant advancement compared to previous studies.

Key words: Dyeing Process, Strength, Wool Fibers, Black Color, Reactive Dye.



تأثير عملية صبغ الالياف الصوفية على قوتها مع تركيزها الخاص على استخدام صبغة الاسود المتفاعلة سمية العريفي ، عبد المجيد عبدالله قسم الهندسة الكيميائية كلية الهندسة صبراتة، صبراتة- ليبيا

الملخص

تدرس هذه الورقة البحثية تأثير عملية صبغ الألياف الصوفية على قوتها، مع تركيز ها الخاص على استخدام صبغة الأسود المتفاعلة .تضمنت الدراسة اختبار وقياس الكسر المتوسط في التمدد وحمولة الكسر للعينات غير المصبوغة والعينات الفارغة والعينات مصبوغة بأقطار متوسطة متنوعة .تم اختبار عدد خمسين ليف منفصلة لكل مجموعة عينة لتقييم قوة الألياف. تشير النتائج إلى أن الألياف الرفيعة تظهر تمددًا متناقصًا وحمولة كسر أقل بالمقارنة مع الألياف السميكة. علاوة على ذلك، يحدث انخفاض متوسط في التمدد وحمولة الكسر لكلاً من أنواع الألياف الرفيعة تظهر تمددًا منتاقصًا متوسط في التمدد وحمولة الكسر لكلاً من أنواع الألياف بعد عملية الصبغ. تشير النتائج أيضًا إلى أنه مع انخفاض قطر الألياف، يصبح انخفاض التمدد أكثر وضوحًا، بينما معربح انخفاض حمولة الكسر لكلاً من أنواع الألياف بعد عملية الصبغ. تشير النتائج أيضًا إلى أنه مع انخفاض قطر الألياف، يصبح انخفاض التمدد أكثر وضوحًا، بينما مع التنبؤات النظرية وتمثل تقدمًا كبيرًا مقارنةً بالدراسات السابقة. الكلمات المفتاحية: عملية الصبغ، قوة الثير، ألياف الموض، اللون الأسود، صبغة

1. Introduction

Wool belong to a group of proteins known as keratins. It has a heterogeneous composition where the protein is made up of amino acids is carboxyl, the amino-acids joined by peptide linkages [1]. The wool is dyed using varies types of dye and colors. Wool dyeing process involves four key steps. First, the fibers are wetted and swollen in the dyeing liquor. Then, dye molecules adhere to the fiber surface. Next, the dye molecules penetrate the fibers through diffusion aided by various physical and chemical forces. Finally, the dye is fixed to the fibers, and any excess dye is rinsed off. These



steps are essential for achieving successful and vibrant dyeing results on wool fibers [2].

Numerous studies have indicated that when investigating the degradation of textile materials like wool and cotton, a widely adopted approach is to study their tensile strength and elongation behavior. This is accomplished through the measurement of tensile strength and extension at the point of breakage using a tensile test method. Alterations in these properties serve as a clear indication of fiber degradation [3-4].

Tensile strength can be defined as the maximum resistance of a material to deformation during a tensile test, which is carried out until the sample is rupture. It is also known to be the breaking load or force per unit cross sectional area of the unstrained sample [5]. However Hatch [6] has defined the tensile strength by the fiber ability to resist a longitudinal pulling force without being ruptured. Naveen and coworkers have also stated that the tensile strength of any fiber declines with increasing dyeing time, probably reflecting the impact on fiber crystallinity [7]. The breaking strength is the force needed to break a textile material when it is pulled. Both as stated above can be used to investigate the effect of various treatment such as dyeing and finishing processes on textile materials [8].

The tensile strength can be determined by applying a load to the sample in its axial direction, until the sample is break. The amount of deformation is called elongation at break (breaking extension) and breaking load at break is called tensile strength. It can be measured ether in Newton or in Newton per unit area, and used as an expression for the textile material strength [9].

There are two types of tensile tests are usually used to monitor the effect of pulling forces on textile materials: the first one uses a constant rate of extension and the second type uses a constant rate of load. The first type is more common than the second type [9]. Each instrument has the same basic components, which include loading, clamping and recording mechanisms [10].

In cases where the pH of dyes used in the dyeing process is not the same, it is important to carefully manage the pH conditions to



minimize the degradation and damage to wool fibers. The tenacity of wool is often reduced after dyeing processes, leading to fiber damage, degradation, and breakage. The degradation and damage of wool fibers occur due to their optimum application pH in the isoionic region. To address this issue, reactive dyes can be utilized, as they have the ability to enhance wool quality through chemical reactions with thiol groups, H₂S, and histidine side chains present in the wool fibers. Additionally, these dyes can act as anti-setting agents, further improving the overall quality of the wool. By selecting and adjusting the pH levels appropriately during the dveing process, it is possible to minimize the negative effects on wool fibers, ensuring better preservation of their strength and integrity [11]. Currently, the dyes commonly used in the dyeing of various fiber fabrics on the market are direct dyes, reactive dyes, vat dyes, and so on, so in this research reactive dyes are used to form stable chemical bonds [12].

This paper aims to explore the impact of the dyeing process, specifically employing reactive black dye, on the strength of wool fibers. The study involves analyzing the average break in extension and breaking load of undyed, blank, and dyed samples with varying average diameters. By investigating the effects of dyeing on fiber strength, this research contributes to a deeper understanding of the dying process of wool and lays the foundation for potential improvements in this domain.

2. Experiment Work

2.1 Description of investigated sample

The investigated samples included six slivers from two different lots of wool. The average diameter of the two lots were estimated using scanning electron microscopic pictures, which was obtained using a Leo® 1430VP Scanning Electron Microscope (SEM). The first lot had an average diameter of 18.5 μ m, and from which both undyed and dyed fibers were studied. The second lot had an average diameter of 20 μ m, and from which undyed and dyed fibers were studied. The dyed samples were dyed black using reactive dye. Blank samples were also prepared, where similar dyeing conditions (T, pH and time) were used with no dye addition. Blank samples



were used in the investigation as a control group to compare against the dyed samples and establish a baseline measurement of the wool fibers properties before dying. They also helped evaluate the effectiveness of the dyeing process and ensure consistent dyeing conditions. Overall, the inclusion of blank samples enhanced the accuracy of the study and provided valuable insights into the impact of dyeing on the wool fibers.

The investigated wool fibers were undergo the same type of treatment before the dying process and the dyeing conditions for all the dyed samples were the same, where the maximum temperature was 100 °C and the standard dyeing time was about 5 hours. The tensile tester of the fibers strength used in this study is called an Instron tensile strength tester (Model 4444), which has a constant rate of extension. This machine is used to investigate tensile properties, with a load cell that can be adjusted according to the strength of the sample to improve accuracy the load used in this study was 50 N. The quantity of tested fibers for different samples was based on the statistical analyses method reported in Modern Statistics in Practice [13].

2.2 Tensile Test Procedure

In this work the British Standard (BS 3411: 1971) tensile test method for single wool fibers was used [14], while the Instron Model 4444 tensile strength tester was used to determine the tensile strength of individual fibers. This instrument is considered to be constant rate of extension testing machine. Single wool fibers were attached to a paper with adhesive tape across a rectangular gap. Then the ends of the paper frame were clamped in the two jaws of the tensile tester. The computer program allowed to run until the sample broke. For data recording the Instron software of Series IX Automated Materials Testing is used. The extension rate that was used in the fiber stretching step is 10 mm per minute with a load cell with a maximum force of 50 N. The extension rate of 10 mm per minute was chosen for the fiber stretching step in the study for several reasons. It aligns with the British Standard tensile test method for single wool fibers and the constant rate of extension testing machine used. The selected rate ensures standardized testing



conditions and accurate data recording, considering the capabilities of the equipment and load cell.

Before operating the machine, the cardboard was cut across in the middle, which left the fiber clamped in the jaws without support and ready to be tested [15]. All measurements were performed in room temperature. For each sample more than fifty separate fibers were tested. However, some results had to be discarded, when ether the sample is broken at the edge of the jaws or the sample is slipped from the jaws.

3. Results and discussions

The average diameter of the two investigated lots were estimated using scanning electron microscopic pictures, and two representative examples of such pictures are shown in Figure 1.



Figure (1): Examples of microscopic pictures for (a) first lot of wool with average of 18.5 μ m diameter fibers and (b) second lot of wool with average of 20 μ m diameter fibers. The scale bar, indicated by a line underneath the images, corresponds to a length of 30 μ m and serves as a reference for the image size.



From the obtained pictures for the first lot of wool fibers (Figure 1(a)) the average diameter was found to be approximately 18.5 μ m. On the other hand the average diameter of the second lot of wool (Figure 1 (b)) was found to has approximately 20 μ m average diameter fibers.

Figure 2 shows a typical examples of the obtained load-elongation (stress and strain) behavior of a wool fiber under standard atmospheric conditions of 65 % relative humidity and at 25 °C room temperature.



Figure 2: Tensile load-elongation diagrams for wool samples with 18.5 µm average diameter (a) and with 20 µm average diameter (b).

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The load-elongation curve is clearly divided into three regions, for all shown samples. These three regions are marked in Figure 2 (a). When the sample is stretched, the tension in the fiber increases very rapidly and almost linearly, this region (A - B) is called initial region and it is often referred to as the Hookean region.

Above this, the elongation increases rapidly for small increases in stress. This section of the curve (B - C) is known as the yield region. The third region of the curve is known as the post-yield region (C - D), and is terminated by rupture of the fiber (D). In this section of the curve, the increase in tension that accompanies a given increase in elongation is larger. Thus the three characteristic regions of the load-elongation curve are very distinct even after the dyeing process as shown in Figure 2 (a), which related to wool samples with 18.5 µm average diameter. The three characteristic regions can be observed and recognized for wool samples with 20 µm average diameter and similar general trend of load-elongation were obtained as shown in Figure 2 (b).

Figure 3 shows the relation of fiber breaking load to breaking extension or elongation for the 18.5 μ m diameter wool fibers, before dying process (Figure 3 (a)) and after blank (Figure 3 (b)) and black color (Figure 3 (c)) dying process.









Similarly Figure 4 shows the relation of fiber breaking load to breaking elongation for the 20 μ m diameter wool fibers, before (Figure 4 (a)) and after blank (Figure 4 (b)) and black color (Figure 4 (c)) dying process.

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From the wide distribution of the scattering load and elongation at the breaking point that shown in Figure 3 and Figure 4, the resulting standard deviations for both undyed and dyed fibers are slightly high. This wide distribution could be the results of variations in the fiber alignment between grips, fiber crimp, pre-tension and fibers cross-section. These and other factors can influence fiber breakage, which results in the high standard deviations.







In both Figures (3 and 4) the simple correlation coefficient (\mathbf{r}), which is dimensionless and indicates how strong the variables (in this case breaking load and elongation at break) correlate to each other. It is clear that after dying the value of the correlation coefficient \mathbf{r} goes dawn. It seems as if due to dyeing the correlation

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between the elongation and the load at break becomes weaker for both fiber types. The weakening correlation observed between elongation and the load at break for both fiber types can be attributed to surface damage resulting from the dyeing process, as well as environmental effects on the undyed samples. The results are scattered throughout the range of dyed fiber elongation-load data, indicating fibers surface damage as a main reason for a loss in strength than fiber bulk properties. The effects of surface damage for the undyed samples due to the environmental and other uncontrolled external factors such as cleaning and scouring [16] are also noticeable when the two undyed fibers scattering load and elongation are compared. Environmental effects are involved, exposure of the wool to weathering, moisture, heat, and sunlight (UV). To minimize these effects, only fibers from the same lot with close diameters were investigated.

The average of the tensile strength of individual wool fibers was also determined with tensile tests. An average of fifty fibers was measured for each sample and reported as the breaking load. Breaking extension or elongation was reported in mm per 10 mm gauge length. A summary of the results for undyed and dyed wool fibers with a diameter of 18.5 μ m are illustrated in Table 1

Parameters	Undyed samples	Dyed samples			
		Blank	Change %	Black	Change %
Breaking extension (mm)	5.740	4.894	-14.73	4.564	-21.77
Breaking load (N)	0.118	0.117	-0.85	0.107	-9.32

Table 1: Average breaking extension and breaking load of undyed, blank and dyed samples with an average diameter of 18.5 μm.

In addition to the average values Table 1 also shows the change in the Breaking extension and the Breaking load as a percentage of the undyed sample (Change %) for fibres with a diameter of 18.5 μ m, where



Change % = /(Property dyed – Property undyed) / Property undyed]*100

Table 2 shows summary of the tensile test results and the change in the measured parameter as a percentage of the undyed sample (Change %) for fibers with a diameter of 20 μ m,

Table 2: Average breaking extension and breaking load of undyed,
blank and dyed samples with an average diameter of 20 µm.

Parameters	Undyed samples	Dyed samples			
		Blank	Change %	Black	Change %
Breaking extension (mm)	6.186	5.960	-3.23	5.541	-10.42
Breaking load (N)	0.146	0.131	-10.27	0.134	-8.22

It is apparent from both tables (1 and 2) that the thinner fibers show less breaking extension as well as lower breaking load than the thicker fibers. However for both cases the average breaking elongation of the fiber as well as the average breaking load decreases after dyeing process.

The obtained results corroborate earlier findings reported by Gullbrandson regarding the negative impact of the dyeing process on the tensile strength of wool fibers [17]. This alignment with previous research provides additional support for the understanding that dyeing procedures can lead to a reduction in the mechanical properties of wool fibers, specifically in terms of tensile strength.

Tables 1 and 2 show that for both lots, 18.5µm wool fibers and 20µm fibers the breaking elongation decreases after dyeing process. The percentages of the decreasing values (Change %) are 14.73 % for blank dyed sample and 21.77 % for black color dyed sample for the 18.5µm diameter wool fibers, while for the 20µm diameter wool fibers the percentages of the decreasing values is 3.23 % for blank dyed sample and 10.42% for black color dyed sample. It is clear that elongation at break for both wool lots that blank dyed decreases, which results from the action of acid, alkali, water, heat, and mechanical stress during dyeing process. However, it is also clear



that elongation at break for both wool lots that dyed black decreases more than the blank dyed samples. This is can be explained as a result dyestuff (reactive dye) in addition to the action of acid, alkali, water, heat, and mechanical stress during dyeing.

Tables 1 and 2 also show that the breaking load for $18.5\mu m$ fibers decreases after dyeing process the percentages of the decreasing values are 0.85% for blank dyed sample and 9.32% for black color dyed sample. This is clearly illustrate the action of acid, alkali, dyestuff, water, heat, and mechanical stress during dyeing. In spite the fact that this effect for the black colored samples, in the first lot (18.5 μm fibers) is much higher than the blank dyed samples, for the second lot samples seems to be the other way around, where the percentages of the decreasing values is 10.27% for blank dyed sample and 8.22% for black color dyed sample.

Overall the results demonstrate a clear trend where decreasing fiber diameter leads to a more noticeable decrease in breaking extension or elongation. Conversely, increasing fiber diameter is associated with a more noticeable decrease in breaking load. These findings emphasize the sensitivity of breaking extension and load to changes in fiber diameter, suggesting that variations in fiber diameter can significantly influence these mechanical properties.

4. Conclusions

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Based on a thorough analysis and discussion of the results, the following points can be concluded:

1. The dying process using black color reactive dyes has an effect on the strength of individual wool fibers with different average diameters and the surface damage plays a significant role in the loss of fiber strength.

2. Although correlation coefficient (r) decreases after dying, indicating a decrease in strength, undyed samples are also subject to surface damage from environmental and external factors like cleanings and scouring.

3. As fiber diameter decreases, the decrease in breaking extension becomes more noticeable, while as fiber diameter increases, the decrease in breaking load becomes more noticeable.



4. The study's findings in terms of surface damage, and the relationship between fiber diameter and breaking extension/load provide important factors to consider in the wool dying process. These findings will serve as valuable references for future research and can guide dyeing practitioners and manufacturers in optimizing their processes.

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