(REFEREED RESEARCH)

AN INVESTIGATION ON COLOUR ANALYSIS AND FASTNESS PROPERTIES OF THE DENIM FABRIC DYED WITH A DIFFERENT METHOD

FARKLI BİR METOTLA BOYANMIŞ DENİM KUMAŞIN RENK ANALİZİ VE HASLIK ÖZELLİKLERİ ÜZERİNE BİR ARAŞTIRMA

Şehpal TÖLEK, Füsun DOBA KADEM

University of Çukurova, Department of Textile Engineering, Adana, Turkey

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ABSTRACT

Denim is a fabric which is produced by weaving dyed warp and weft yarns. The most important feature of denim fabric is the colour which is created by indigo dyeing. The mere reason for this special look is magical ''Indigo'' which the warp yarns are dyed with. In this study, an innovative dyeing method was applied by adding an extra step to indigo-sulphur dyeing. The main target was achieving the unique colour transitions and appearances. At the scheduled research, colour analysis and fastness properties were evaluated.

Keywords: Denim, Indigo Dyeing, Sulphur Dyeing, Colour Fastness.

ÖZET

Denim, boyalı çözgü ve atkı ipliklerinin dokunması ile üretilen bir kumaş türüdür. Denim kumaşın en önemli özelliği indigo boyama ile elde edilen rengidir. Bu özel görünümün temel sebebi çözgü ipliklerinin boyandığı indigo adı verilen boyadır. Bu çalışmada indigo+sülfür boyamaya ilave bir boyama adımı eklenerek yenilikçi bir uygulama yapılmıştır. Temel amaç özgün renk geçişleri ve görünümler elde etmektir. Yapılan çalışmada renk analizi ve haslık özellikleri değerlendirilmiştir.

Anahtar Kelimeler: Denim, İndigo Boyama, Kükürt Boyama, Renk Haslığı

Corresponding Author: Füsun DOBA KADEM e-mail: fusundobakadem@gmail.com

1. INTRODUCTION

Denim has been an indispensable part of the textile industry for years. In a globalizing world, the evaluation and the supply curve of denim fabric in the market moves to the side of visual variety. Considering these developments, designing and production of value-added products which cater to fast-growing current fashion and its demands are unavoidable.

Denim clothes which constitute a considerable percentage of current fashion, maintain their superiority on the subject of casual clothes in the world's markets. According to the International data from the WTO (World Trade Organization), being the 5th biggest garment industry supplier and 8th biggest Textile supplier, Turkey also has a considerable capacity of production of denim fabric and clothes, also an important potential of exportation.

Denim clothes are one of the most important productive branches of garment industry in Turkey. Also, this product group has a share of 25-30 % of Turkey's woven garment exportation. 368,6 million dollar worth denim fabric was exported from Turkey in the first quarter of 2015 [1].

Denim is a hard and durable, warp faced 3/1 twill cotton fabric, woven with indigo dyed warp and white filling yarns, having weights of 14½ ounces per square yard. In the last decade, denim was mainly dyed with indigo (67%), sulphur black (26%) and other sulphur colours (6%). The changing fashion trends have also led to vat, reactive and direct dyestuffs, as well as pigments being used to colour denim. Sulphur dyes are of low cost and can be applied to indigo

machinery, fabric dyeing machinery, jiggers, pad-batch and garment dyeing machinery with properly designed methods. Sulphur dyes have an appearance that is more natural than reactives or directs, having a softer appearance and allowing versatile wash down effects in laundering. Therefore, among the different dye classes other than indigo, sulphur dyes are more popular and are used for bottoming or topping or over dyeing of finished indigo dyed garments to produce a variety of shades with fancy looks. Pigments are relatively easy to apply, and since they are a surface colouration, can produce a distressed look, but they present colour fastness problems in darker shades and have a harsher feel than dyed garments. Indigo is insoluble in water, and since it belongs to the vat dye class, it has to be converted into a water soluble form of reducing under alkaline conditions. In ancient times the reduction process was carried out in wooden vats; therefore, this class of dyes is known as vat dyes. The process of dyeing of cotton with indigo essentially consists of alkaline reduction of indigo into a water soluble form known as leuco indigo, dyeing by multiple dips and air oxidation to convert leuco indigo to its water insoluble form [2].

Indigo in its natural form has been used for dyeing about 5000 years. Today, mostly, indigo is used for dyeing cotton yarns for the warp of woven denim fabric on rope dyeing ranges. The indigoferra plants yield large amount of purer dyestuff. Indigo was extracted from plants later to be replaced by synthetic indigo onto the market. But actually, the principle of dyeing indigo has never changed. Basically;

I. Dissolving the dye by reduction involving vatting and loosening up,
II. Dyeing from the vat,
III. Oxidizing in the air [3].

Denim Fabrics can be categorized as dyed with indigo and non-indigo. Many studies examining the dyeing of cotton yarn with indigo dyes and influencing factors. Studies on colour results, scales, and ring effects of indigo dyes have also been conducted and their results have been analyzed statistically. Chakraborty used a different solvent (DMSO:Dimetil sulfoxido) because of poor affinity of indigo for cotton. The results of his study were shown that, DMSO was the right solvent to extract indigo with ease [4]. Etters deliberated that high dyebath pH caused fader effect to substantivity, additive effect to absorption of dye to fiber and high level of ionization. On the other hand, moderate dyebath pH promoted better ring effect on dyed yarns.[5]. Gorenšek et. al. aimed to determine the influence of temperature, pH and the addition of sodium oxide silicium oxide (SOSO) on the dyeability of cotton warp during the process of dip-dyeing. According to the results obtained, dyeing in a dye bath at 40°C can be observed lower dyeability, pH level must be between 9 and 10,5 and SOSO increases the buffer capacity of alkaline dye baths [6].

Besides, sulphur dyes can be used in dyeing of cellulose too. In addition to the single usage of indigo dyes, or sulphur dyes, it can also be used with sulphur-dyes [7,8,9].

Özgüney et. al. studied, laser was used for the fading of denim fabrics. The effects of laser beam on the physical properties of denim fabrics such as tensile strength, tear strength, abrasion resistance, and static and kinematic friction coefficient were investigated and optimum process conditions were determined [10]. Güneşoğlu investigated the effect of PU coating on important mechanical, performance and comfort-related / breathability properties of denim fabric and evaluated the contribution of fabric and PU polymer weight at the final product [11].

There has been lost of research on denim garment industry in recent years. These studies have been an essential resource for both academic fields and suppliers. In spite of that, the number of scientific researches in the literature is not sufficient when the scale and the importance of the industry considered.

It is every producing firm's wish to extend its product range. Every producing firm endeavors to meet the demands of its customers with the collections they prepare and the products they produce. At this point, colour is a very critical parameter. Firms aim to enlarge their market share by having in stock all the colours their customers need. The aim of the producer is to respond these differences by keeping their product range extended. And this method has been applied to the demand of a mill, as a result of some product development operations.As a consequence of this study, produced fabric has been presented to the firm's customers. A great deal of positive feedback has been received relating to the resulting colour [12].

2. MATERIAL AND METHOD

Denim is one of the world's oldest fabrics, and yet it remains eternally young as long as coloured with indigo dyes. So in this study, a different dyeing method was applied by adding an extra step to indigo-sulphur dyeing at a well-known denim mill in Adana (in Turkey).

Greece and USA origin cotton bales were mixed and spun in ring spinning machines. Cotton and Lycra were used for both warp and weft. For rope dyeing, ball warps are continuously fed into the rope-about 300-400 per rope yarns paralleling and move together-for application of the indigo dyeing. The ropes are kept separated from each other throughout the various parts of the dye range. The ropes are first fed into one or more scouring baths, which consist of wetting agents detergents and caustic. It is to remove naturally occurring impurities found in the cotton fiber, such as dirt, minerals, ash, pectin, and naturally occurring waxes. The purpose of this step is to guarantee uniform wetting and uniform dyeing. Then, ropes are fed into one or more water rinsing bath.The next step is to feed rope into the indigo dye baths and skied after each dip (Figure 1). After iterative numbers of dipping, ropes follow washing pads in order to remove unfixed indigo dyestuff. Because of high pH value during the dyeing process, it needs to be lowered and this step has a neutralizing effect. This step is completed reaching sufficient humidity with the help of steamed drying cylinders. Sufficient humidity ratio is important for the efficiency of re-beaming of the ropes after dyeing. After that, the ropes of yarn are rinsed in several water baths to remove any unfixed dye. In order to extract water mechanically, ropes will pass through squeeze rolls after the

dyeing process. The yarns are then dried and coiled into large tubs [12].

Figure 1. Oxidising in the Air [13]

In this research, all parameters that aforementioned were kept the same except last sulphur dye. The main phases are given in the tables below. Hereby, the differences between this two type relevant fastness, colour assessment such as lightness, colour angle etc. were examined. Denim fabric properties which are used in the experimental study and main processes applied to denim fabric are given in Table 1.

In Table 2, methods of special dyeing processes are stated sequentially. B1,B2,B3... means the baths in the rope dyeing machine. In the 13th bath; our reference fabric had been washed, but the other fabric called "SANDWICH DYEING" had a sulphur step differently (Table 2). Warp yarns in the form of ropes (each rope contains approximately 344 yarns) pass through the dyeing baths with a speed of 28 metres per minute. Baths have a pH balance of 11,90. Sulphur dyeing process in the first bath takes place at 80±5°C. Indigo dyeing takes place at 32±5°C. These fabrics that we measured were woven on looms Picanol brand. Both two versions have same weft yarn and woven specified as weft density, reed, speed etc. In addition to this, they had same finishing processes [12].

CIE Lab (L*, a*, b*, c* and h) values and colour differences (ΔE) values of the fabrics were measured by the spectrophotometer. To determine fastness performance of samples, colour fastness to washing, to rubbing, to light, to perspiration which are classified as usage fastness tests, have been applied to the denim samples and their results have been investigated [12]. Colour measurements of both reference and sandwich fabrics were performed by employing CIELab system, with 10° standard observer and under D65 daylight and L*, a*, b*, C*, h values were recorded. L*, a*, b*, C*, h values mean the lightness, the chroma index of the red-green axis, the chroma index on the yellow blue axis, the colour angle and the colour saturation respectively in the CIELAB colour system. In this study, a Minolta brand CM 3600 D model spectrophotometer was employed. Real Colour 1.3® and CHROMA CMY® software was used to calculate the colour difference values (ΔE). This value is determined with respect to the quality standards of the manufacturer and customer demands. In this experimental study, total colour difference tolerance was assumed as 1,0 [14]. After dyeing, produced fabrics were tested, these tests and standards were explained in Table 3.

Table 1.Processes of A Denim Fabric

Parameter	**Properties**
Weaving Machine	Picanol
Warp Thread	Ne 16/1 CO+78dtex EA
Weft Thread	Ne 16/1 CO+56dtex PBT
Weave Type	Twill 3/1 Z
Reed	Comber number:80/4,Comber Width:200 cm,33 warp thread/cm,18,5 weft thread/cm
Composition-Elastane Ratio	97% CO+2%EA+1%PBT
Fabric Finishing Process	Singeing+ Washing(80°)+ Mercerizing (NAOH-28° Bè)+Sanforizing
Finishing Chemicals	Wetting agent(15 g/l),softener (2 g/l), acidity regulator(3 g/l)
Washing Recipes	**Enzyme wash (EW):** 30ʹ 45°C, cellulose enzyme+ dispersing agent **Stone wash (SW):** 30ʹ 45°C, bath brick+ dispersing agent+ desize enzyme+ abrasion enzyme **Bleach wash (B):** 15ʹ 30°C, sodium hypochlorite, 10ʹ 40°C, neutralent.

Table 2. Dyeing Workflow [12]

Dyeing Baths	**Sulphur+Indigo Dye Version (reference)**	**Sulphur+Indigo+Sulphur Dye Version(sandwich)**
B1	Sulphur Dyeing	Sulphur Dyeing
B2-B3-B4	Washing	Washing
B5-B6-B7-B8-B9-B10	Indigo Dyeing	Indigo Dyeing
B11-B12	Washing	Washing
B13	Washing	Sulphur Dyeing
B14-B15-B16	Washing	Washing

Table 3.Tests and Relevant Standards

Measured Properties	Relevant Standard	Testing Device
Tensile Strength and Elongation	TS EN ISO 13934-1:2013	Titan Universal Strength Tester
Tear Strength	TS EN ISO 13937-1:2002	Elmendorf Tearing Strength Tester
Spectrophotmetric Analysis	--	Minolta Spectrophotometer
Colour Fastness to Washing	TS EN ISO 105 C06: 2001	Rotawash Washing Fastness Device
Colour Fastness to Rubbing	TS EN ISO 105-X12:2006	Crockmeter
Colour Fastness to Light	TS 1008 EN ISO 105 B02:2013	SDL Atlas Xenotest 150S+
Determination of pH	TS EN ISO 3071:2009	pH meter
Colour Fastness to Perspiration	TS EN ISO 105 E04:2012	MESDAN 257A Perspirometer

3. RESULTS AND DISCUSSION

Both for producers and consumers, colour fastness of textile materials is one of the most important features. The aim of this study was to enlarge the colour scale for denim customers. So this method has been applied to the demand of a mill, as a result of some product development operations. The fabrics are grouped in two categories. These which dyed respectively, with sulphur and indigo dyes were named as Ref (Reference). Likewise, the fabrics which dyed in 3 steps (sulphur+indigo+sulphur) were named as Sand (Sandwich).

Strength properties are the most important aspect of any material to assess its performance. In this study, tensile and tear strength values were affected more in the warp direction. Because the fabrics have twill construction and dyeing processes were applied to warp yarns, not weft yarns. So weft yarns weren't much affected by this application. As seen in Table 4, a decrease (approximately %8) was observed in warp tensile strength after sandwich dyeing. It could be said that after sandwich dyeing application, the denim fabric may have sufficient strength to any % loss in tensile and tear strength.

Colour values of all samples and comparisons of them are presented in Table 5 and Table 6. Four replicates were used to collect the data. All mean values are given in Table 5 and Table 6.

Table 4. Test Results of Tensile Strength, Tear Strength and Elongation of The Samples[12]

		Tensile Strength			Elongation(%)			Tear Strength		
		Mean(N)	S.D	CV(%)	Mean	S.D	CV(%)	Mean(cN)	S.D	CV(%)
Ref.	Warp	1320,52	46,33	3,5	36,68	1,25	3,4	5842	218,37	3,7
	Weft	618,62	15,17	2,4	51,29	0,54	1,05	5389,6	35	0,6
Sand.	Warp	1216,63	52,2	4,2	36,87	1	2,7	5908,75	124,12	2,1
	Weft	611,11	22,24	3,6	50,91	1,01	1,98	5385,75	75,66	1,4

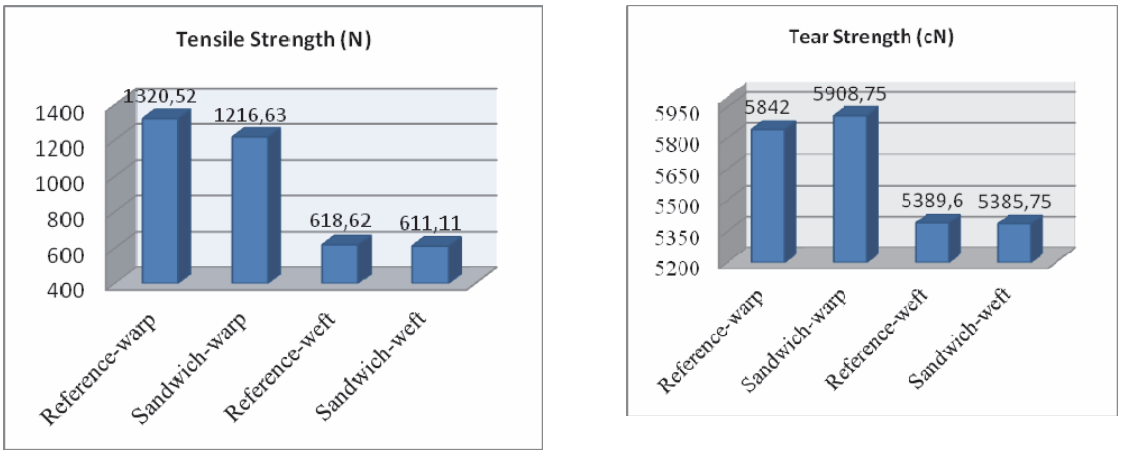


Figure 2. Strength Values of the Samples [12]

Table 5. Measured CIE-Lab Values of the Samples

Colour values/ Samples	Ref (unwashed)	Sand (unwashed)	Ref_{EW}	$Sand_{EW}$	Ref_{SW}	$Sand_{SW}$	Ref_{B}	$Sand_{B}$
L*	20,28	18,52	15,39	14,53	17,60	16,78	22,46	23,69
a*	1,077	0,87	1,00	0,42	0,34	-0,30	-0,54	-0,72
b*	-4,70	-2,69	-10,18	-6,6	-12,035	-8,73	-14,86	-1518
C	4,825	2,83	10,23	6,61	12,045	8,73	14,84	15,20
h	282,47	287,89	275,60	273,67	271,59	268,01	267,94	267,27

Table 6. Colour Assesment Results

Color Assessment		Ref to Sand	Ref_{EW} to $Sand_{EW}$	Ref_{SW} to $Sand_{SW}$	Ref_{B} to $Sand_{B}$
ΔE	Mean	2,76	3,74	3,48	0,95
	SD	0,539	0,186	0,208	0,143
ΔL		-2,546	-0,76	1,08	0,877
Δa		-0,208	-0,583	-0,643	-0,184
Δb		2,01	3,58	3,308	-0,344
Δc		-1,997	-3,527	-3,308	0,35
ΔH		0,318	0,282	0,645	0,169

When spectrophotometric results were analyzed, it was determined that, new color was;

- Darker
- Greener
- Yellower
- More weak than the reference color.

As shown, washings were progressively getting heavier. The most important result mainly obtained is that even if heavy washings were implemented, the sandwich dyed fabric protects its color, darkness and effect as against the reference dyed fabric. Only after the heaviest washing, the colors of both fabrics were getting white and these two colors were closing up to each other. The results of ΔE values of bleach washings (named B) have also proven this.

Fastness performance of reference and sandwich samples were carried out with respect to the relevant standards (in Table 3).

Table 7. Colour Fastness to Wash, Staining On to Multifibre Fabric and Shade Change [12]

	Sample	
	Reference	Sandwich
Wool	4/5	4/5
Acrylic	4/5	4/5
PES	4/5	5
Nylon	4	4/5
Cotton	3	4/5
Acetate	3/4	4/5
Shade change	4/5	4/5

Where denim fabrics are used as a garment, usually cotton products are preferred as underwear. So, it is seen obviously that this dyeing process is effective in terms of improving colour fastness to washing according to Table 7 and Figure 3. As specified in LEVI'S Standards (LS&CO. Global Fabric Performance Standards), it is known that denim has got very poor wet rubbing fastness. In the light of this information, when the fabrics' rubbing values were analyzed, sandwich dyeing didn't give the worst results than reference dyeing (Table 8). Colour fastness to light (table 9), pH values (table 10) and colour fastness to perspiration staining on to multifibre fabric and shade change (table 11) are located respectively. Many different reactions can occur between cotton, reducing chemicals and dyestuffs according to the pH values of the fabric. Pursuant to OEKO-TEX standards, fabric's pH values have to be between 5,5 and 8. In denim, the preferred range is between 6 and 7,5 [15]. It was found that, pH value of sandwich fabric was more alkaline than the reference fabric. But it is an expected result due to sulphur dye. Even so, this pH value is in the limits specified by OEKO-TEX standards.

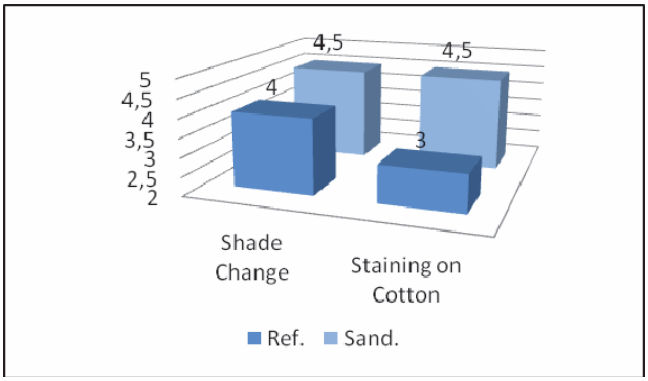


Figure 3. Colour Fastness to Washing Values [12]

As seen in Table 11, perspiration fastness rates of alkaline and acidic are generally similar to each other.

The samples were subjected to image analysis with a scanning electron microscope, and the images were shown in Figure 4. Scanning Electron Microscope (SEM) was used to observe the surface of the denim samples belonging to reference and sandwich. Also weft and warp yarn of both samples were examined with the same magnification.

Table 8. Colour Fastness to Rubbing of the Samples [12]

Sample	Colour Fastness to Rubbing -Shade Change		Colour Fastness to Rubbing -Staining On	
	Dry	Wet	Dry	Wet
Ref-Warp	5	4	4	1
Ref-Weft	5	4	3/4	1
Sand-Warp	4/5	4/5	4	1/2
Sand-Weft	5	4/5	3/4	1/2

Table 9. Colour Fastness to Light Values of the Samples [12]

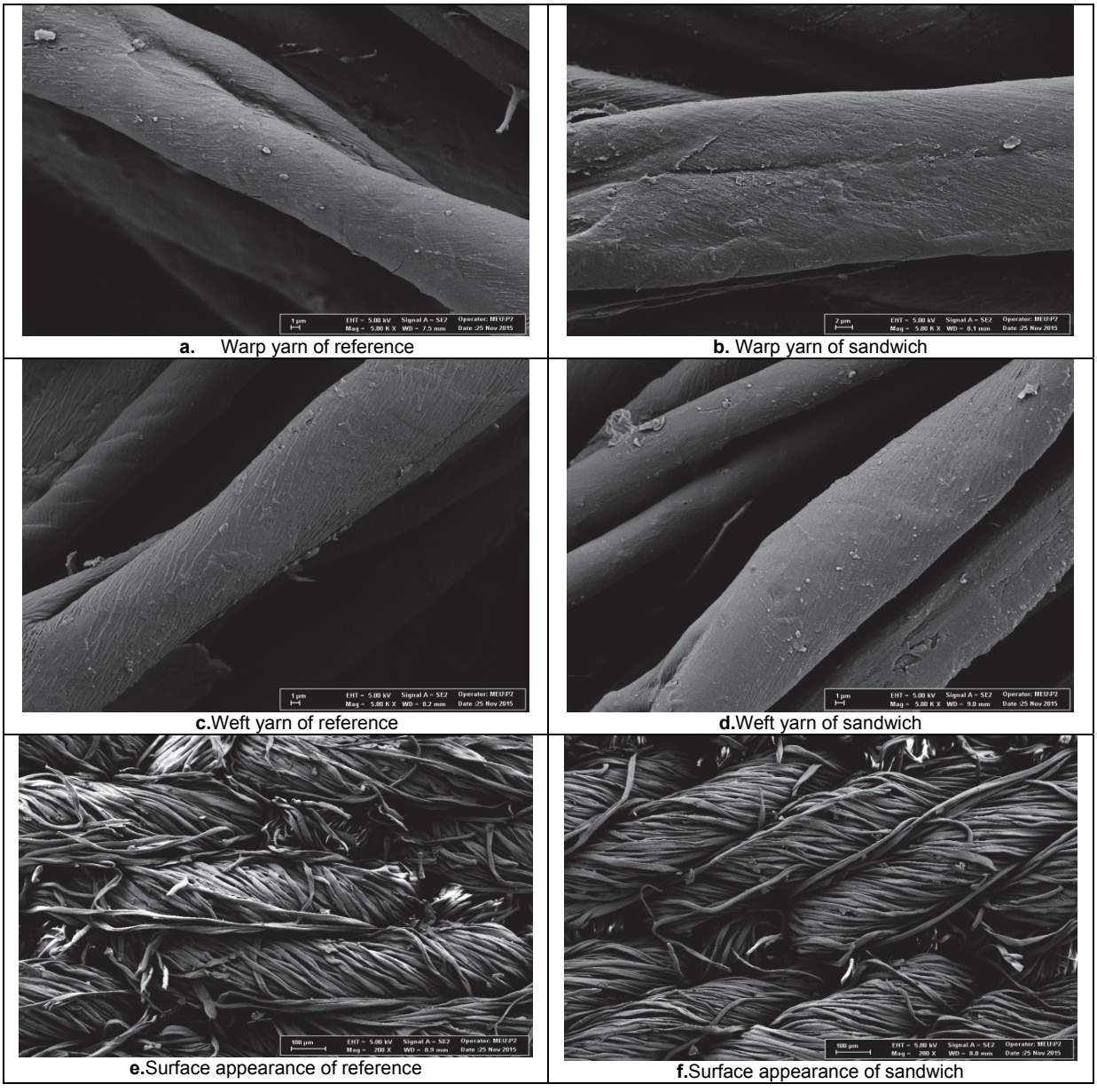
	Sample	
	Reference	Sandwich
Colour Fastness to Light	5	5

Table 10. pH values of the Samples [12]

	Sample	
	Reference	Sandwich
pH	5,5	6,5

Table 11. Colour Fastness to Perspiration Staining On to Multifibre Fabric and Shade Change of the Samples [12]

	Sample (Acidic)		Sample (Alkaline)	
	Reference	Sandwich	Reference	Sandwich
Wool	4/5	5	4	4/5
Acrylic	4	4/5	5	5
PES	4/5	5	5	5
Nylon	4/5	5	5	4/5
Cotton	4	5	4/5	4/5
Acetate	4/5	5	4/5	4/5
Shade change	4/5	4/5	4/5	4/5



a. Warp yarn of reference

b. Warp yarn of sandwich

c.Weft yarn of reference

d.Weft yarn of sandwich

e.Surface appearance of reference

f.Surface appearance of sandwich

Figure 4.SEM Analyses [12]

4. CONCLUSIONS

The meeting consumer's expectations and requests are one of the most important competition factors in denim sector. The main target to do this research is trying to get a specific color that consumer demand and want to be in firm's collections. This study was supported by a well-known denim mill. So it is easy to say that any producers could use this dyeing method to improve their colour scale.

According to the results of the research;

- ✓A different color was added to firm's collections. Thereby, color scale was developed.
- ✓In comparison to reference color, new color has not got poor fastness results. Nearly all the fastness values of reference and sandwich fabrics are approximately same.
- ✓After three washes recipes were applied to both reference and sandwich fabrics, sandwich fabric was seen darker and sulphur dyes stayed longer on this fabric.

The following points can be recommended:

- ✓Anti back-staining chemicals can be used in the finishing process. So the disadvantages may go away.
- ✓Because of the fact that the fabric's pH may be risky, during the producing pH level must be controlled.

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