

Influence of special finishes on denim properties

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This study deals with imparting special effects on denim fabric using novel finishes. Special finishes are applied on desized indigo dyed cotton fabric. Their impacts are evaluated concerning breaking force, spectral characteristics and surface appearance. The results show significant differences among modern special treatments, thus providing important data on the processing of denim. All finishes show decrease in breaking force and cause changes in spectral characteristics of the treated samples. The results indicate that some of them are acceptable only as an effective segment on a garment.

Keywords: Cotton, Denim, Finishing, Indigo dye, Tensile properties

1 Introduction

Denim is one of the older, more fashionable, and widely-used fabrics. It has become very popular within the area of fabric appearance, being modified to appeal to varied fashion trends and demands. Numerous mechanical and chemical operations of finishing can be replaced by enzyme treatments, as enzymes are ecofriendly, non-toxic, and fully biodegradable compounds. They are amylases for degradation of amylose as applied when desizing; cellulases, for the degradation of cellulose and cellulosic substrates as applied for stone-washing of denim; catalases for the degrading hydrogen peroxide as applied for bleaching, dye discolouration and effluent treatment; glucose oxidase for attacking glucose, acting as a bleaching agent. Enzyme technology, in addition to other benefits, also offers the potential of process integration¹⁻¹³. Enzymes possess an active centre on the surface which exactly matches the complementary centre of the substrate according to the so-called key-lock principle^{1,14-17}. The main advantages of enzymes used in denim finishing are efficiency, accelerated reactions, being operational under mild conditions, acting on a specific substrate exclusively, easiness of control, biodegradability, and a wide-range of industrial applications. Optimal temperature range for most enzymes is 30 - 70°C under neutral conditions^{1-2,9,14-16,18,20}. During enzymatic treatment, the removed indigo dye can be redeposited on the white weft yarn of the

denim fabric, so it is important to prevent backstaining^{19,20}.

Processing with α -amylases represents the oldest and primary enzyme application in textile finishing¹⁷. For denim fabrics, as well as for the other fabrics made from cotton and its blends, the warp threads are coated with a substance known as size or starch. It acts as a lubricant and protects the yarn during weaving. After weaving, the sizing agent has to be removed from the cotton fabric²¹. The enzymes used for the desizing of cotton are mainly of bacterial origin, such as *Bacillus subtilis*. Amylases hydrolyse starch with no harmful effects on cotton fabrics^{22,23}. Starch hydrolysing enzymes are classified according to the type of sugar produced such as α -amylases or β -amylases²⁴.

Leather finish (LF) and wax finish (WF) form a very thin layer on denim surface and thus change the surface characteristics of the treated fabric. The strato (STF) and the spider finishes (SPF) are the processes of exhaustion, while the soft resin finish (SRF) is performed by spraying²²⁻²⁴. Leather finish is a simple and fast surface treatment for specific effects, where short and dense fibres give "peach skin" effect. It can be applied either on a small part or the whole surface of denim fabric. Wax finish provides a smooth, oily and greasy touch to the treated denim. The application of special binders and chemicals provides elastic and soft, silicone-like film onto the treated areas. Special resin finishing is used for the imparting authentic three-dimensional permanent creases, resistant to wearing and washing. Resin can be applied by

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spraying directly onto the denim. Creases can be formed using binder clips or aluminium pipes before or after spraying the resin. The main disadvantage of the SRF is strength loss of the treated denim. Special finish labelled as strato is an actual and trendy one, due to the interaction with design and its discolouration ability. Primarily, different kinds of creases are formed using clips or other trimmings, e.g. a textile-tagging gun. Those parts of denim that are not accessible for chemicals (inner parts of the material) are not bleached, so the treated area resembles floating clouds. Special kind of retro-look with unique designs can be achieved using spider finish. Crackle effects can be obtained by sporadic breaks on the denim pre-treated with a special stiffening agent.

This study deals with the application of special finishes on denim for creating modern effects. Control of special effects obtained is executed through mechanical properties, spectral characteristics, and microscopic surface observation of the treated denim compared to the desized one. The impacts of special treatments on denim fabric as described in this paper and according to the available literature, have not been investigated so far. Special effects are taken in order to study the reactions of the used finishing agents with indigo dyed material, as well as their

influence on the strength and surface characteristics of the treated material.

2 Materials and Methods

2.1 Materials

Indigo dyed cotton fabric, having the specifications, twill 2/1 weave construction, mass per unit area 451.4 g/m² and warp/weft density 27/19 threads/cm, was used for the study.

Pre-treatment of the denim fabric was carried out by bio-desizing. Special finishes were applied on desized fabric using products, kindly supplied by the German-Swiss company CHT – Bezema, and the Croatian Company Kemo (Table 1).

2.2 Methods

2.2.1 Desizing

Bio-desizing (D) was carried out in a Polymat, Werner Mathis, using bacterial α -amylase with the addition of a crease prevention agent, as shown below:

α -amylase	: 1 %
Crease preventing agent	: 1.5 %
Bath ratio	: 1:5
Temperature	: 50 °C
Time	: 60 min
pH	: 6.0

Table 1—Chemical composition of agents, properties and applications

Chemical composition	Ionic character	Application field and properties
Bacterial amylase	-	Degradation of amylose in desizing
Concentrated pigment	-	For tinting an aqueous screen pastes
Acrylic polymer	Anionic	Aditive in synthetic resin applications
Hydroxylamine derivative	Anionic	Agent for the removal of manganese dioxide
Glyoxal resin	-	Crosslinking agent for synthetic resin finish
Metalic salt with organic additives	-	Catalyst for crosslinking of synthetic resin finish
Fatty acid ester	-	Crease preventing agent
Fatty acid condensation product	Cationic	Softener for protecting indigo dyed fabric from yellowing
Fatty acid condensation product with amino-functional polysiloxane	Non-ionic	Special softener for handle properties
Silicone emulsion	Non-ionic	Auxiliary for strato effect
Aqueous preparation of polysaccharides	Anionic	Special agent for spider finish effect
Modified fatty alcohol ethoxylates	Non-ionic	Detergent free of alkylphenoethoxylates
Sodium alkyl aryl sulphonate	Anionic	Washing agent
Polysiloxane emulsion	Slightly cationic	Agent for softening the coatings layer (wax finish)
Polyurethane dispersion	Anionic	Special binder for coating
Melamine resin	Non-ionic	Crosslinking agent for leather finish
Acrylic	Anionic	Special agent for leather finish

After the enzymatic desizing, enzymes were deactivated by immersing the samples for 10 min in warm water (80°C), followed by a thorough rinsing, alkali washing in a solution of anionic surfactants and soda at 80°C, and air drying.

2.2.2 Special Finishes

Leather finishing (LF) was performed using a mixture of 660 g of special agent for leather finish (Table 1), 20 g of a crosslinking agent, 283 g of water, and 37 g of concentrated black pigment. The prepared finishing bath contents were uniformly applied to the upper-side (right side) of the denim with wet pick-up of 51.5%, followed by drying at 100°C for 3 min and curing at 150°C for 5 min.

Processing of the wax finish (WF) was carried out in a finishing bath containing 200 g of slightly cationic silicone agent (Table 1), 700 g of a polyurethane binder, 20 g of a crosslinking agent, 80 g of black pigment, and 80 g of water. The prepared finishing bath content was uniformly applied to the upper-side with wet pick-up of 49.9%. The samples were dried at 100°C for 3 min and cured at 150°C for 5 min.

Soft resin finishing (SRF) was performed by spraying the content of bath prepared from 200 g/L glyoxal resin, 100 g/L acrylate polymer, and 20 g/L of a special catalyst for synthetic resin. Wrinkles were formed on a flexible aluminium tube. Drying was carried out in the Scholl apparatus at 80°C, and curing at 150°C for 10 min.

Special agent for spider finish (SPF) was uniformly applied to the upper-side of the denim with wet pick-up of 20.5%. The samples were slowly dried for 30 min at 80°C, cracked by hand, and blown using compressed air. The broken places were treated with a solution of KMnO_4 (5% solution) and left to react for 10 min. The samples were then washed and neutralized for 30 min at 40°C in a bath containing 1 g/L special detergent free of alkylphenolethoxylates (APEO) and reducing agent for releasing the manganese dioxide. Finally, the samples were treated with a non-ionic softener, centrifuged, and air dried.

The strato finish (STF) was carried out in a finishing bath containing 100 g/L non-ionic silicone emulsion and 5 g/L of KMnO_4 , in a bath ratio 1:15, over 30 min at 30°C. The samples were then washed and neutralized for 20 min at 60°C with a special agent (1 g/L) and reducing agent (4 g/L), softened, centrifuged, and air dried.

2.3 Test Methods

Various analytical techniques were used to control the concentrations of pre-treatment and treatment agents. The enzymatic activity of the bacterial amylases was tested using an AATCC test method 103-1999, while the bio-desizing effect was evaluated considering the iodine solution (KI/I_2) — dark blue reaction of the residual starch.

A tensile strength tester (Tensolab Mesdan S.P.A., Brescia, Italy) was used for the evaluation of mechanical properties of breaking force and breaking elongation of the treated samples, according to the standard EN ISO 13934-1. Tensile testing speed on the strips in warp direction was kept 100 mm/min, with the pre-load of 2 N. The results were expressed as the average number from five measurements.

The mass per unit area before and after pre-treatment were determined according to the standard ISO 3801:2003. Spectral characteristics were measured using a Daticolor Spectraflash SF 300 remission spectrophotometer, Switzerland, under the conditions: aperture size 20 mm and standard illuminate D_{65} . The change in colouration of the samples after finishing was characterized by the CIE $L^*a^*b^*$ scale and expressed as the average number obtained from five measurements on different denim areas. Morphological changes in the treated samples were observed using a Dino-Lite digital microscope, Premier, with the magnification of $\times 230$.

4 Results and Discussion

The results of mass per unit area and change in weight (Table 2) show that all treatments cause changes in the surface areas of the denim fabrics. Desizing with enzyme (dextrogenic amylase activity of 1500 BAU/g) causes maximal reduction in weight, due to the removal of starch and possible indigo dye or other sizing additives up to the level of 12.3%. Special additives in the finishes increase the weight of the denim fabric. Additionally, they form a surface layer and increase the thickness of the treated material. STF causes an increase in the surface area due to the shrinkage of the denim and the application of a high concentration of synthetic resin. The exception was SPF, where an insignificant reduction in weight can be attributed to the discolouration agent applied, potassium permanganate.

Special finishes additionally influences the mechanical properties of the treated fabric (Table 3) which include the values of breaking force (F_b) and

the ratio of breaking force as well as the associated area of denim (F_b/Q). The changes in the denim fabric mechanical properties are analysed employing breaking force and elongation (Table 3 and Fig. 1). Standard deviation (SD) measures the dispersion of a set data from its mean, while the coefficient of variation (CV) measures the unexplained or residual variability of the data.

The results obtained indicates that the special finishing processes causes changes in mechanical properties of the denim fabrics. Breaking force decreases in all the treated samples in comparison with the untreated ones. SPF and SRF has the greatest impact on the reduction of breaking force respectively (SPF 61% and SRF 52%), with equivalent values of F_b/Q (1.7 for SPF and 1.9 for SRF). The change in tensile properties of SPF and SRF is caused by resins that stiffenes the surface, despite the application of softener in the case of SPF.

Special finishes cause changes in spectral characteristics of the denim fabrics (Table 4). It is expressed considering the change in lightness (dL^*), chromaticity (dC^*), hue (dH^*), and total difference in colour (dE) of the desized fabrics, as compared to that with the untreated ones, and those that undergo

special finishing in comparison with the desized fabrics. This is expected, considering the purpose (fashion and design) of these specific treatments. However, their real practical and useful characteristics are shown through breaking force (Table 3).

The impact of special finishes on colour depends on the agent applied, as well as on the processing parameters employed. Desizing tends to remove starch from warp threads but the spectral parameters indicate that the dyestuff is also removed (Table 4). More prominent change in total colour difference of the denim fabrics treated is achieved with LF ($dE=14.147$). Black pigment added to the bath causes greying of the denim fabric. The impact of WF and SRF on colour change is found to be lower as compared to LF. SPF and STF cause strong changes in colour, due to the use of discolouring agents. Total difference in colour in the case of SRF is found not so prominent.

Special treatments show an impact on the surface and appearance of the denim. Surface characterization is performed by a digital microscope (Figs 2 and 3). Compared to the untreated denim surface (Fig. 2), there is a visible slight change in colour, as well as compaction and shrinkage of the desized fabric.

Microscopic observation ($\times 230$) of denim fabric surface presented in Fig. 3(a) shows visible spherical

Table 2—Mass per unit area and weight loss of untreated and treated samples

Samples	Label	Q g/m ²	Weight loss %
Untreated	U	451.40	-
Desized	D	395.98	12.28
Leather finish	LF	472.58	-19.34
Wax finish	WF	560.39	-41.52
Soft resin finish	SRF	424.18	-7.12
Spider finish	SPF	385.70	2.60
Strato finish	STF	408.71	-3.21

Table 3—Breaking force of denim fabric and quotient of breaking force and surface area

Label	F_b N	SD	CV %	F_b/Q N/m ² .g ⁻¹
U	2039	54	2.668	4.5
D	1675	87	5.224	4.2
LF	1890	28	1.491	4.0
WF	1487	54	6.603	2.7
SRF	803	72	8.911	1.9
SPF	660	152	22.988	1.7
STF	1552	46	2.951	3.8

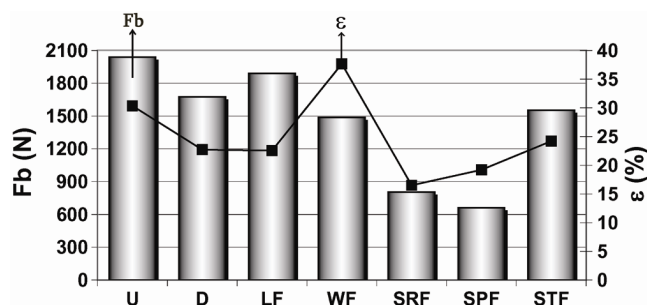


Fig. 1—Breaking force (F_b) and breaking elongation (ϵ) of untreated and treated denim

Table 4—Changes in spectral values of treated fabrics compared to untreated fabric

Label	dL^*	dC^*	dH^*	dE
D	-3.449	0.417	0.626	3.530
LF	13.285	-4.856	0.117	14.147
WF	1.477	-4.028	0.208	4.305
SRF	-3.713	-0.652	0.156	3.813
SPF	6.038	8.017	-1.393	10.259
STF	5.741	8.996	-1.681	10.987

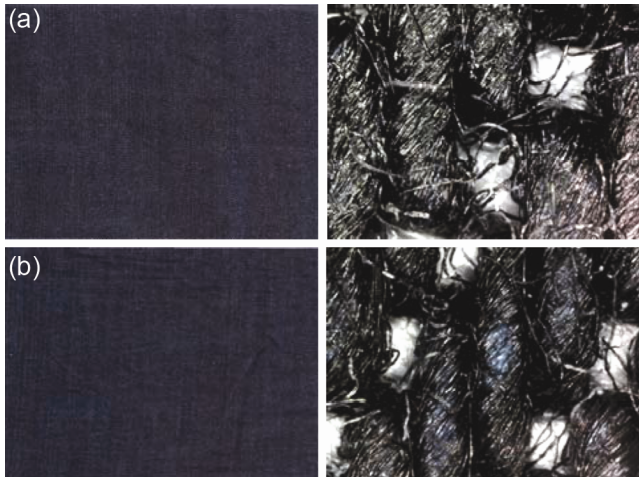


Fig. 2—Images of untreated (a) and desized (b) denim fabrics (left side – photo; right side – microscopic image $\times 230$)

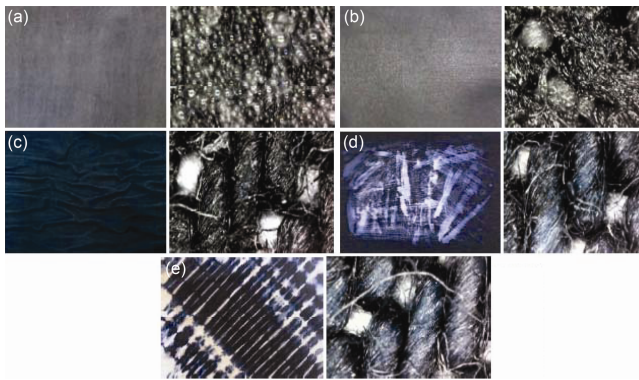


Fig. 3—Appearance of special finishes (a) LF, (b) WF, (c) SRF, (d) SPF and (e) STF (left side – photo; right side – microscopic image $\times 230$)

structures on the surface of the leather finished (LF) material. Wax finish (WF) enables various special fatty and smooth surfaces [Fig. 3(b)]. Special treatment with resin (SRF) slightly smoothens the surface of the denim fabric [Fig. 3(c)]. Spider effect [Fig. 3(d)], as a special type of retro discolouration, results in visibly light areas on the surface. Strato finish (STF) [Fig. 3(e)] as well as spider effect (SPF) offer colourless light areas with unique and aesthetic purposes. It is evident that STF results in more noticeably cloudy areas than SPF [Figs 3(d) and (e)].

4 Conclusion

Special finishing of denim results in attractive and respectable patterns as important design elements.

The results of breaking force and spectral values indicate notable differences between special treatments. Acceptable special effects, according to technological indicators, can be obtained by the strato, leather, and wax finishes. Resin and the spider finish affect fabric weight and reduce its strength and usability in garments. Therefore, these special effects could be applied only as effective parts or segments of a garment.

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