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Dyes and Pigments 67 (2005) 215–221
DYES and PIGMENTS
www.elsevier.com/locate/dyepig

# Evaluation of modified leather dyeing technique using black dyestuffs from the economical view

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Received 6 July 2004; received in revised form 12 August 2004; accepted 3 December 2004
Available online 25 February 2005

**Abstract**

From the economical point of view, this work illustrates the effect of compact dyeing technique of chrome tanned leather on the final quality of wet-finished leather in which all wet-finishing steps are in one drum, using black dyestuffs (Acid Black 210 and Remazol black B), with low concentrations, this was done alternative to that used in leather industry. In case of soft leather dyed with Acid Black 210 and top dyed with Remazol Black B the colour strength of both grain and flesh sides increases when using a mixture of (chitosan/non-ionic surfactant, after storing for five days), while the dye penetration decreases compared with heavily retanned leather impregnated with a mixture of (sodium alginate/non-ionic surfactant, after storing for five days) under the same condition without affecting the exhaustion percentage of the dye bath. Using Remazol Black B, the colour strength and the dye bath exhaustion were not affected, while the total dye penetration through cross-section of the dyed leather was improved under the action of either sodium alginate mixture or the natural tanning materials. In addition, the effect of this new technology on the final properties of wet-finished chrome tanned leather such as light and rubbing fastness, mechanical properties and thermal stability were studied.


## 1. Introduction

It has been estimated that 50–60% of all leather made is dyed black. In our industry, the colour black is as popular as ever and it is set to continue to be in great demand in the future. Black leather that is expected to fulfil the highest standards of quality and appearance poses a particular challenge to Dyers, this is especially true today, because the degrees of fastness are continuously increasing and customers demand heavily retanned leathers dyed to deep, brilliant shades, while demand is increasing for leathers that are dyed through the cross-section. The technique of leather dyeing has remained unchanged for a long time [1]. The vinyl sulphone dyestuffs possess high affinity for hydrogen active compounds, they react very easily with primary or secondary amines and alcoholic OH groups, so these vinyl sulphone dyes through covalent linkage with amino groups of collagen produce water and solvent fast leather [2].

High quality leathers necessitate careful monitoring and control at various stages of manufacturing, such as retanning, dyeing and fatliquoring. This is easily achieved when the new technology is applied. Reactive dyestuffs are very useful for the manufacture of special leather products like garments, clothing, cars and airplanes [3]. Various approaches have been developed to improve the uptake of anionic dyes on leather, the most common being to increase the cationicity of the surface and thereby enhance the electrostatic attraction operating between the dye and the substrate [4]. Cationic auxiliaries are mostly used for shade deepening for brilliant and intense dyeing. The amount of product and the timing of the addition should be in accordance with the affinity of the substrate [5]. The chitosan pretreatment of pickled hide powder increased the shrinkage temperature and also increased both the rate and

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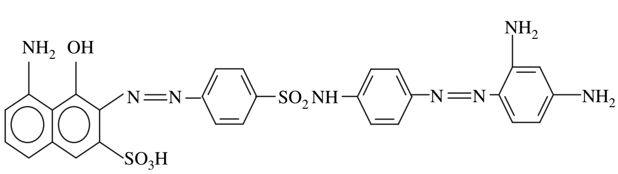
doi:10.1016/j.dyepig.2004.12.007

extent of adsorption of acid yellow 83 onto chrome tanned hide powder [6]. The achievement of better dye fastness is one of the great requirements of Tanners. A new reactive dye system is based on activation of the leather by increasing the number of reactive sites for the subsequent dyeing, which is followed by retanning, fatliquoring and acid fixation [7]. The sequence of leather dyeing technique can be broken, fatliquors can be added both with and before retannage, sometimes neutralization and dyeing are performed together [8]. Neutralization, retanning, dyeing and fatliquoring when applied in compact formulations require a special selection of products and/or product combinations that are compatible [9]. The used pretreatments of chrome tanned leather such as ammonia, urea, chitosan in combination with non-ionic surfactant, influenced the charge of leather, and consequently improved the dyeability of the leather with both metal complex and mono- and di-chloro triazin reactive dyes [10].

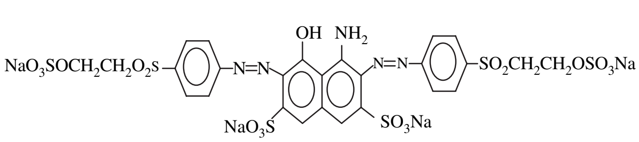
## 2. Materials

(1) Chrome tanned leather of thickness 1.5–1.9 mm and 3.6% chromium oxide was supplied by a Tannery located in Cairo.
(2) Auxiliary chemicals such as Coripol MB (fatliquor), Dolatan F1 (retanning agent), mimosa and quebracho extracts (vegetable tanning agents) were obtained from commercial sources.
(3) The two following commercial dyes were supplied by Dystar L.P. with different chemical structures:

- Acid Black 210 (anionic dye).



- Remazol Black B C.I. Reactive Black 5.



## 3. Methods

### 3.1. Dyeing

Compact dyeing recipes of both soft and heavy retanned leather with the used dyestuffs are illustrated in Appendices 1 and 2, respectively.

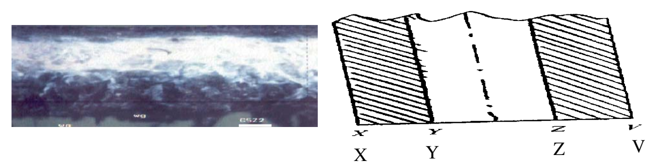
### 3.2. Dyeing properties

#### 3.2.1. Colour measurements [11]

The dyed leather samples were evaluated for colour strength, expressed as $K/S$ (where $K$ is the absorption coefficient and $S$ is the scattering coefficient), using Ultra Scan XE. On the other hand, the dye uptake of all the used dyestuffs were calculated by sampling the dye bath before and after the dyeing process using UV–Vis Shimadzu Spectrophotometer.

#### 3.2.2. Degree of the dye penetration

According to Haroun [10] and Przepiorkowska [12], a novel method to calculate the degree of dye penetration is by using Askania GSZ 2T Light Microscope compact with Video Camera "CF11/2 Kappa" and photography by "Mitsubishi Printer CP700". The degree of penetration was calculated along cross-section of dyed samples with 2.5× magnification and 2000 mm scale as follows:




$$\%\ \text{Penetration from the grain side} = \frac{VZ}{VX} \times 100\%$$

$$\%\ \text{Penetration from the flesh side} = \frac{YX}{VX} \times 100\%$$

where VX is skin thickness, VZ is thickness of coloured layer from grain side and YX is the thickness of coloured layer from flesh side.

#### 3.2.3. Light and rubbing fastness

*3.2.3.1. Fastness to artificial light.* Leather specimen of not less than 1 cm × 6 cm is exposed to artificial light using "Atlas Xenotest Alphalm" under IUF402 and DIN [13] 54004 condition with eight fastness standards which consist of pieces of wool cloth dyed with standard blue dyes of different degrees of fastness.

*3.2.3.2. Rubbing fastness [14].* The rubbing fastness (dry, wet and pH 8) of the dyed samples were measured according to DIN standards [15] using "Otto Specht Bally Finish Tester".

### *3.3. Mechanical properties [16,17]*

#### *3.3.1. Tensile strength, elongation at break and tear resistance*

According to DIN standards, the dyed samples were measured using "Tira Test Apparatus".

### *3.4. Shrinkage temperature [18,19]*

According to DIN 53336, the shrinkage temperature of all dyed samples were measured Using "DTM 1010 Digital Thermometer, Type NiCr-NiAl, measuring range −30–1100 °C.

## 4. Results and discussions

Fig. 1 shows the colour strength ($K/S$) and dye bath exhaustion of the grain and flesh sides of treated leather by 2% chitosan + 2% supralan UF mixture (after storing for five days) and 2% sodium bicarbonate + 2% supralan UF mixture achieved using 2% owf of Acid Black 210, followed by top dyeing using 1% owf of Remazol Black B, and 1% owf of Remazol Black B in the case of soft and heavy retanned leather, respectively, relative to the untreated counterparts (without the treatment addition during the compact dyeing technique).

Using the chitosan, which is cationic polymer (poly electrolyte), in aqueous solution of non-ionic surfactant as treatment during compact dyeing technique leads to intensification of shade at both the grain and the flesh sides of dyed chrome tanned leather samples and also leads to the enhancement of the dye uptake compared with the untreated ones. The additional amino groups within the treated samples, which under the acidic conditions (pH 5.5) prevalent during the exhaustion stage of dyeing, would be protonated and thereby result in increased ion–ion interaction operating between the anionic dye and the treated leather. The addition of chitosan to an aqueous solution of supralan UF (poly glycol ether) leads to alteration of the nature of the aggregated species present, however, chitosan molecules and supralan micelles interactions might occur [20–22]. After storing for five days, the viscosity of the mixture was sharply decreased (nearly like water) due to the electrostatic attraction between the formed micelles and the chitosan chains. At pH 4–5, the protonated amino groups in chitosan molecules were increased, consequently the electrostatic repelling between the same sign charged groups took place which led to the swelling of the macromolecule; so this treatment mixture can be easily adsorbed and penetrated to a greater extent through leather fibre, especially on the flesh side than that of the grain one owing to the well-known structural differences between the two sides because the flesh side has more open structure fibre.

The strongly anionic sulpho groups present in the anionic dye interact electrovalently with the cationic amino groups of chitosan treatment mixture and collagen fibre forming salt-like linkage, in slightly acidic medium (pH 4–5) as follows [23]:

$$\mathrm{Dye-SO_3^-} + \mathrm{{}^+H_3N-Protein} \rightarrow \mathrm{Dye-SO_3^- \cdots {}^+H_3N-Protein}$$

$$\mathrm{Dye-SO_3^-} + \mathrm{{}^+H_3N-Chitosan} \rightarrow \mathrm{Dye-SO_3^- \cdots {}^+H_3N-Chitosan}$$

Another type of interaction between the anionic dye and the leather may occur, which is called secondary valence interaction (dipole–dipole and hydrogen bonding interactions), involving the hydroxyl, amino and sulphonic function groups present in the dye molecule and the amide groups of the collagen fibre as follows [3]:

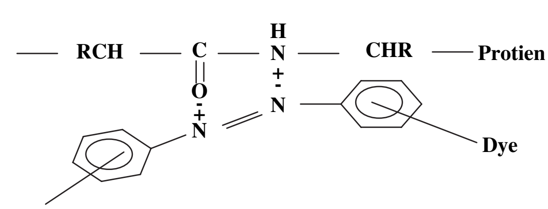



Fig. 2 shows the colour strength and dye bath exhaustion of the grain and the flesh sides of treated leather by 2% sodium alginate + 2% supralan UF mixture (after storing for five days) achieved using 1% and 0.25% owf of Remazol Black B, in the case of soft and heavy retanned leather, compared with 2% and 0.5% owf of the used reactive dye in case of the untreated ones, respectively.

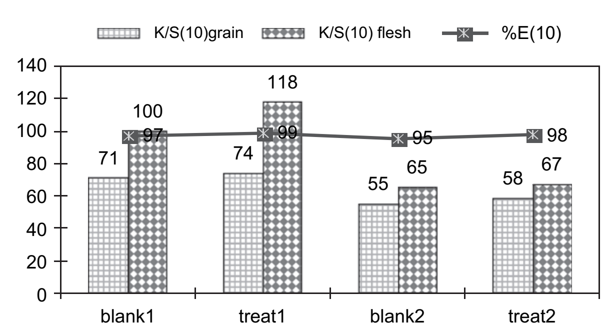



Fig. 1. Effect of compact dyeing technique on the dyeing measurements of dyed leather with Acid Black 210 [Blank1: soft leather without addition of the treatment mixture, Treat1: soft leather treated with the mixture (2% chitosan/2% supralan UF, after storing for five days), Blank2: heavy retanned leather without addition of the treatment mixture, Treat2: heavy retanned leather treated with the mixture (2% sodium bicarbonate/2% supralan UF, at PH 8)].

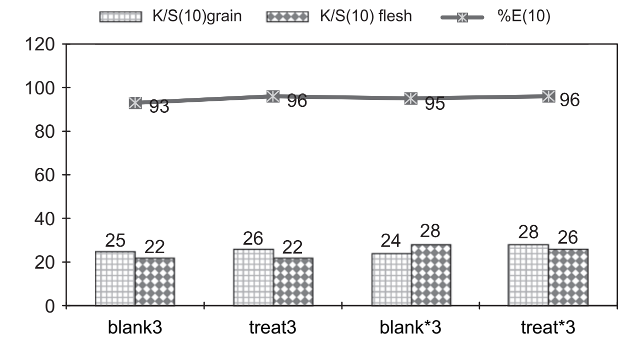



Fig. 2. Effect of compact dyeing technique on the dyeing measurements of dyed leather with Remazol Black B [Blank3: soft leather without addition of the treatment mixture, Treat3: soft leather treated with the mixture (2% sodium alginate/2% supralan UF, after storing for five days), Blank*3: heavy retanned leather without addition of the treatment mixture, Treat*3: heavy retanned leather treated with the mixture (2% sodium alginate/2% supralan UF, after storing for five days)].

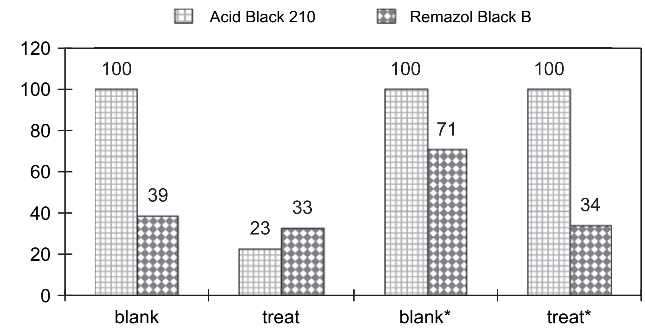
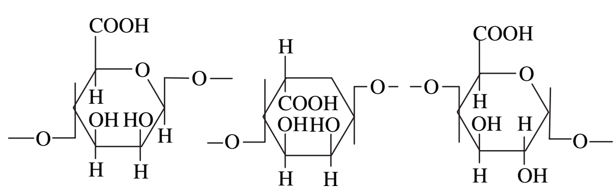



Fig. 3. Effect of compact dyeing technique on the penetration degree of the used black dyes [Blank: including (blank1 and blank3), Treat: including (treatment1 and treatment3), Blank*: including (blank2 and blank*3), Treat*: including (treatment2 and treatment*3).

Sodium alginate (highly viscous) is a hydrophilic polysaccharide and a polymer of a hydro-D-mannuronic acid as follows [3]:




Some of chemical variables that affect algin solutions are pH and the presence of sequestrates [24]. The lower molecular weight sodium alginate was stable at pH as low as 3.0, however, some residual calcium content increase in viscosity at pH 5.0, so it is unstable at this pH, 2.5% medium viscosity sodium alginate solution is pseudoplastic over a wide range of shear rates (10–10 000 $s^{-1}$) while at low concentrations (1%), the solutions have almost constant viscosity, to overcome this problem, 2% sodium alginate was mixed with 2% aqueous solution of non-ionic surfactant (supralan UF) followed by storing for five days, and then used as treatment during compact dyeing technique to enhance the dye uptake and improve the colour shade of dyed leather.

In the case of vinyl sulphone reactive dye in the presence of alkali, nucleophilic addition reaction between collagen fibre of leather and the dye takes place but another mechanism (like the anionic dye) might occur, i.e. some of the electrostatic attractions between sulphonic, amino and amide groups of collagen fibre and also hydroxyl and carboxyl groups of alginic acid treatment (pH 4–5) and amino group of the dye, in addition to hydrogen bonding and dipole–dipole interactions.

Fig. 3 shows the effect of the compact dyeing technique on the penetration degree of the black dyes. Despite the high colour shade at the surface, in the case of using a mixture of chitosan/supralan UF during compact dyeing technique, Acid Black 210 completely penetrated through the dyed leather; this may be due to the adsorbed chitosan at the surface of the treated leather, so most of the dye molecules reacted with the chitosan at the surface, and consequently, the dye penetration decreased. Also, some of the chitosan molecules in aqueous solution of non-ionic surfactant were trapped by the dye molecules in the dyeing bath, so the concentration of the dye also decreased.

It can be concluded that if we need penetrated dyed leather the compact dyeing technique with 2% Acid Black dye, which contains disazo, amino, hydroxy and sulpho groups, was more preferred in the case of soft or heavy retanned chrome tanned leather, while if we need surface dyed leather only, the addition of the chitosan mixture was strongly recommended especially in the case of soft leather. On the other hand, the bifunctional vinyl sulphone reactive dye (Remazol Black B) was attached at the surface of the dyed leather. It can be concluded that if we need black dyed leather with strong shade at the grain surface, the compact dyeing technique with natural tanning materials and low dye concentration (0.25%) was strongly recommended.

Table 1 shows the rubbing (dry, wet and pH 8) and the light fastness of the wet-finished leather. Both rubbing and light fastness of soft and heavy retanned leather dyed with (2%) Acid Black 210 followed by top dyeing with (1%) Remazol Black B improved, in the case of using both the mixtures of 2% chitosan and 2% sodium bicarbonate mixed with 2% supralan UF relative to the untreated ones, respectively. In the case of the chitosan mixture, the chitosan was adsorbed at the surface of the treated leather very well, so the dye molecules were not aggregated and the light fading was decreased, while the alkali addition (sodium bicarbonate) led to an increase in the positive charges at the surface of the treated leather and the dye rapidly attached at the surface very well.

On the other hand, the decrease in the concentration of the bifunctional vinyl sulphone reactive dye (from

Table 1
Effect of the dye concentration on the fastness properties of dyed leather using compact dyeing technique

| Dye types | Dye concentration (%) | Leather types | Fastness with respect to colour | | | |
|---|---|---|---|---|---|---|
| | | | Rubbing | | | Light |
| | | | Dry | Wet | pH 8 | |
| Acid Black[a] 210 | 2 | Blank1 | 3–4 | 3 | 3 | 2 |
| | 2 | Treat1 | 4 | 3–4 | 3–4 | 3 |
| | 2 | Blank2 | 4 | 3–4 | 3–4 | 2 |
| | 2 | Treat2 | 4–5 | 3–4 | 3–4 | 3 |
| Remazol Black B | 2 | Blank1 | 4 | 3–4 | 3 | 2 |
| | 1 | Treat1 | 4 | 3–4 | 3 | 3 |
| | 0.5 | Blank*3 | 3–4 | 3 | 3 | 3 |
| | 0.25 | Treat*3 | 3–4 | 4 | 4 | 3 |

[a] In case of Acid Black 210, all dyed leather samples top dyed with 1% Remazol Black B.

2% to 0.25%) has no remarkable effect on both rubbing and light fastness of the soft and heavy retanned leather in the case of using 2% sodium alginate + 2% supralan UF mixture after storing for five days, this may be due to the formation of large complex molecules between the reactive dye and sodium alginate; consequently, the dye was precipitated inside the fibre structure of the treated leather.

Table 2 shows the effect of the compact dyeing technique on the mechanical properties (tensile strength, elongation at break and split tear strength) of wet-finished leather relative to the ultra compact (according to TFL Leather Technology Ltd. formulation of standard wet-blue) and the German specification standards (according to Deutsche guetegemeinschaft Moebel e.v., DIN 53328 and DIN 53329). All the mechanical properties were improved in comparison to the ultra compact technique. The produced wet-finished leather recommended all requirements of both auto&gloves and clothing leathers.

Table 2
Effect of the compact dyeing technique on the mechanical properties of wet-finished leather relative to the international standards

| Mechanical tests | Compact technique | Ultra compact[c] | International standards[d] | |
|---|---|---|---|---|
| | | | Auto | Gloves&Clothes |
| Tensile strength ($N/mm^2$) | 17[a]–15[b] | 11 | ≥9 | ≈10 |
| Elongation at break (%) | 62[a]–59[b] | 55 | ≤60 | ≥50 |
| Split tear strength ($N/mm^2$) | 44[a]–36[b] | 34 | ≥20 | ≥15 |

[a] Acid Black 210, 2% chitosan + 2% supralan UF after storing for five days.
[b] Remazol Black B, 2% sodium alginate + 2% supralan UF after storing for five days.
[c] According to TFL Leather Technology Ltd. formulation of standard wet-blue.
[d] According to Deutscheguetegemeinschaft Moebel e.v., DIN 53328 and DIN 53329.

Table 3
Effect of the compact dyeing technique on the thermal stability of dyed leather

| Dye types | Leather types | Shrinkage temperature (°C) |
|---|---|---|
| Acid Black* 210 | Blank1 | 97 |
| | Treat1 | 100 |
| | Blank2 | 103 |
| | Treat2 | 101 |
| Remazol Black B | Blank1 | 96 |
| | Treat1 | 100 |
| | Blank*3 | 109 |
| | Treat*3 | 115 |

Table 3 shows the effect of compact dyeing technique on the thermal stability of wet-finished leather. Shrinkage is a phenomenon, associated with dimensional changes of leather when subjected to heating, so the shrinkage temperature value is taken as a measure of hydrothermal stability of leather. It was observed that the thermal stability of the wet-finished soft and heavy retanned leathers was improved in the case of both chitosan and sodium alginate mixture when compared with the untreated ones, however, the shrinkage temperature increased from 96 °C and from 109 °C to 115 °C relative to the untreated/treated soft and heavy retanned wet-finished leather. This may be due to the increase in leather fibre stabilization by the introduction of the high molecular weight (polymers) compounds such as natural tanning materials, chitosan and sodium alginate between the leather fibrils and the dye cross-linking, especially in the case of Remazol Black B.

From the economical point of view, in the case of using Acid Black dye, if the complete penetrated dyed leather was demanded (soft or heavy retanned) with minimum dye concentration (2%), the mixture of 2% chitosan and 2% sodium bicarbonate mixed with non-ionic surfactant after storing for five days was strongly recommended. However, in the case of bifunctional vinyl sulphone reactive black dye, if the surface dyed leather (soft or heavy retanned) was demanded with a dye concentration of 0.25% only, the mixture of 2% sodium alginate + 2% supralan UF after storing for five days was strongly recommended.

## 5. Conclusion

The affinity of the dye to leather depends mutually on the structure and state of both the dye and the leather, for the leather, it depends on the type of tannage, the presence of chemical active substances in the float and surface active agents on the fibre surface; for the dye, it depends on the structure of the dyestuffs and their sensitivity to any of the dyeing conditions.

We can use the compact dyeing technique, in which the black dyes (bifunctional vinyl sulphone reactive), retanning and fatliquoring materials add in one drum, consequently, the consumption of the used dye concentration was decreased and the total time and temperature of the processes were saved relative to the classical one. So, the wet-finishing processes of leather improved economically.

## Appendix 1. Compact dyeing recipe of soft leather

| All steps compact in the same drum | Used chemicals | Concentration (%) | Temperature (°C) | Time (min) | |
|---|---|---|---|---|---|
| Neutralization | Water | 200 | 40 | | |
| | Sodium formate | 0.5 | | 15 | |
| | Sodium bicarbonate | 0.5 | | 15 | pH 5.5 |
| Retanning | Dolatan F1 | 2 | 40 | 15 | |
| | EDTA | 1 | | 15 | |
| Treatment mixture[a] | Chitosan/SUF | 2/2 | 30 | 30 | pH 5.5 |
| | Sodium bicarbonate/SUF | 2/2 | 30 | 30 | |
| | Sodium alginate/SUF | 2/2 | 30 | 30 | |
| Dyeing[b] | Acid Black 210 | 2 | 40 | 30 | |
| | Remazol Black B | 1 | 40 | 30 | pH 4.5 |
| Fatliquoring | Coripol MB | 3 | 40 | 30 | |
| Dye-fatliquor fixation | Lactic acid (30%) | 3 | 55 | 30 | pH 3.5 |
| Drain washing | Water | 100 | 55 | 5 | |
| Top dyeing[c] drain/washing/horse-up/dry | Remazol Black B | 1 | 55 | 30 | Final pH 3.5 |

[a] Another sample processed without this stage.
[b] In case of untreated samples (blank), the concentration of the reactive dye increased to 2%.
[c] Using top dyeing only in case of starting the compact dyeing technique with Acid Black 210.

## Appendix 2. Compact dyeing recipe of heavy retanned leather

| All steps compact in the same drum | Used chemicals | Concentration (%) | Temperature (°C) | Time (min) | |
|---|---|---|---|---|---|
| Neutralization | Water | 200 | 40 | | |
| | Sodium formate | 0.5 | | 15 | |
| | Sodium bicarbonate | 0.5 | | 15 | pH 5.5 |
| Retanning | Dolatan F1 | 2 | 40 | 15 | |
| | Mimosa extract | 5 | | 15 | |
| | Quebracho extract | 5 | | 15 | |
| | EDTA | 1 | | 20 | |
| Treatment mixture[a] | Sodium bicarbonate/SUF | 2/2 | 30 | 30 | pH 5.5 |
| | Sodium alginate/SUF | 2/2 | 30 | 30 | |
| Dyeing[b] | Acid Black 210 | 2 | 40 | 30 | pH 4.5 |
| | Remazol Black B[d] | 0.25 | 40 | 30 | |
| Dye-fixation | Acetic acid (30%) | 2 | 55 | 5 | pH 3.5 |
| Drain washing | Water | 100 | 55 | 5 | |
| Top dyeing[c] drain/washing/horse-up/dry | Remazol Black B | 55 | 30 | 1 | Final pH 3.5 |

[a] Another sample processed without this stage.
[b] In case of untreated samples (blank), the concentration of the reactive dye increased to 2%.
[c] Using top dyeing only in case of starting the compact dyeing technique with Acid Black 210.
[d] Another sample processed with 0.5% Remazol Black B, in case of the untreated samples (blank).

## Appendix 3. Some of the terminology used in leather industry

- Chrome tanning: Process which converts the raw hides into leather using soluble chromium salts, primarily basic chromium sulphate.
- Fatliquoring: Process which makes leather more soft and flexible using some of sulphated and sulphonated fatty acids and waxes in addition to emulsifier.
- Heavy retanning: Process which makes leather more fuller and compact for the production of shoe (sole) leather and bookbinding, i.e. using vegetable tanning materials.
- Soft leather: It is more flexible, softer and suitable for clothing, cars and garments, etc.
- Neutralization: Process which nuetralizes the high negative charges of leather after tanning process to make it suitable for the dyeing, fatliquoring and retanning (wet-finishing), i.e. increasing pH from 2 to 6.

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