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### COMPARISON OF FELLMONGERED AND SHORN WOOL WITH SPECIAL REFERENCE TO IMPROVEMENT IN DYEING

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Despite the fact that the 'Pak Mark' wool classing system<sup>1</sup> has been well adopted for export of Pakistani wool, the classing of wool for home-use is either not practiced or is highly haphazard. One of the disadvantages of this situation is that fellmongered and shorn wools get mixed up and give rise to difficulties in processing.

Pakistani pelt is considered at home and abroad to be of a high quality for leather manufacture. This pelt is of great economic value as is thicker and denser as compared to fine wool skin which is thin and has little commercial value.<sup>2</sup> The two main methods of fellmongering that are in practice in our country are the salting and the painting methods. The former method is used extensively as it is rather inexpensive and convenient, while the later is costly and requires great care. However, the pelt produced by the painting process is superior to that by the salting method. Fellmongered wool comprises longer fibres as compared to shorn wool and is also more uniform in staple length, thus giving a higher tear value on combing.<sup>3</sup>

Previous work carried out in these laboratories<sup>4</sup> has shown that in the case of the salting method, there are no significant differences in breaking strength and dyeing behaviour of fellmongered and shorn wool. However, no work has been done so far in Pakistan, on the wool samples obtained by the painting process under practical conditions. Work done in foreign countries<sup>5</sup> indicates that in the sulphide painting process problems in scouring arise due to contamination of the wool fibre with lime. As regards dyeing, it has been shown<sup>3</sup> that the dye uptake of fellmongered wool is less than that of shorn wool with the result that 'skitteriness' occurs in the fabric. However, no method seems to have been evolved so far, for obtaining uniformity in shade of fellmongered wool.

In the present work the samples of wool obtained by the 'sulphide painting process' were employed. The damage of fellmongered wool was investigated by comparing it with that of shorn wool. Further, particular attention was given to the dyeing behaviour of fellmongered wool with a view to especially examining the dyeing ability of fellmongered wool. This was followed by investigations on improvements to the dyeing practices and on enhancement of the scouring ability of the wool so pulled.

#### Materials and Methods

*Wool Samples.* As far as is known, the only factory applying the sulphide painting process in Pakistan is

located at Lahore. At this factory, the shorn wool samples were taken from 10 skins from mid-side before fellmongering process. The pulled samples were taken after the skins had been processed, by pulling from the immediately adjacent place in the mid-side vicinity. The samples were cleaned with petroleum ether followed by ethanol and finally washed with distilled water. The samples after drying were hand-carded and vegetable matter removed. The following studies were made on them.

*Alkali Solubility.* The damage of all the samples was determined by the alkali solubility method.<sup>6</sup>

*Dyeing.* The dye-bath consists of 2% dye (acid dye), 4% H<sub>2</sub>SO<sub>4</sub> (concd) and 10% Na<sub>2</sub>SO<sub>4</sub>, based on the weight of fibre.<sup>7</sup> In order to avoid unevenness in dyeing, the temperature of the dye-bath was raised slowly to boiling point and boiling was continued for 1 hr.

*Spectrophotometric Determination of Dye Uptake.* The dye uptake of fellmongered and shorn wool was determined by using Beckman Model D.U. spectrophotometer. The dye uptake was calculated with the help of standard curve obtained from the reflection and absorption of the known concentration of the same dye using a constant frequency of 600 nm.

#### Results and Discussion

It is obvious from the results given in Table 1 that the alkali solubility of fellmongered wool is greater than that of the shorn wool. Comparing these results with the international standard,<sup>6</sup> it is clear that the fellmongered wool is slightly damaged. Statistical investigation revealed, however, that the differences in the alkali solubility of the two groups were not significant. From these observations, it can be concluded that such a difference is unlikely to affect further processing of the fellmongered wool.

From Tables 2 and 3, it is clear that the dye uptake of fellmongered and shorn wool is different. This variation in dye uptake is graphically represented in Fig. 1. This figure represents the dye-concentration of the dye-bath after exhaustion. It is obvious from the figure that the concentration of mother liquor of fellmongered wool (F) samples is greater than that of the shorn wool (S), which ultimately results in less dye uptake of fellmongered wool than that of shorn wool.

TABLE 1. COMPARISON OF THE ALKALI SOLUBILITY OF FELLMONGERED AND SHORN WOOL.

Fellmongered wool	Alkali solubility	Shorn wool	Alkali solubility
F <sub>1</sub>	17.5	S <sub>1</sub>	13.5
F <sub>2</sub>	14.7	S <sub>2</sub>	14.3
F <sub>3</sub>	14.9	S <sub>3</sub>	13.8
F <sub>4</sub>	14.7	S <sub>4</sub>	14.1
F <sub>5</sub>	14.1	S <sub>5</sub>	13.9
F <sub>6</sub>	15.5	S <sub>6</sub>	15.0
F <sub>7</sub>	15.8	S <sub>7</sub>	15.2
F <sub>8</sub>	15.4	S <sub>8</sub>	13.8
F <sub>9</sub>	16.8	S <sub>9</sub>	12.4
F <sub>10</sub>	16.7	S <sub>10</sub>	14.4

TABLE 2. SPECTROPHOTOMETRIC DETERMINATION OF UNKNOWN DYE CONCENTRATION.

Fellmongered wool	Remittance (%)	Shorn wool	Remittance (%)
F <sub>1</sub>	92.2	S <sub>1</sub>	94.5
F <sub>2</sub>	91.5	S <sub>2</sub>	93.6
F <sub>3</sub>	89.8	S <sub>3</sub>	91.6
F <sub>4</sub>	93.5	S <sub>4</sub>	95.5
F <sub>5</sub>	91.8	S <sub>5</sub>	93.5
F <sub>6</sub>	95.6	S <sub>6</sub>	96.5
F <sub>7</sub>	93.4	S <sub>7</sub>	94.6
F <sub>8</sub>	89.3	S <sub>8</sub>	90.8
F <sub>9</sub>	90.1	S <sub>9</sub>	91.9
F <sub>10</sub>	90.8	S <sub>10</sub>	92.6

TABLE 3. SPECTROPHOTOMETRIC DETERMINATION OF KNOWN DYE CONCENTRATION FOR STANDARD CURVE AT 600 nm.

Dye concn (%)	Remittance (%)
0.02	96.5
0.04	94.0
0.06	91.5
0.08	89.0
0.10	86.2
0.12	84.0
0.14	82.0
0.16	79.5
0.18	77.5
0.20	74.5

The difference in dye uptake of fellmongered and shorn wool, and also the scouring difficulties of fellmongered wool may be due to the following facts: (1) the contaminated sodium sulphide, which hydrolyses to sodium hydroxide in dyeing bath, neutralises some of the acid from the dye-bath and results in low dye uptake; (2) the contaminated lime in the sulphide painted fellmongered fibres, reduces on the one hand the acidity of the dye-bath, and, on the other hand, interferes in scouring properties of the fellmongered wool fibre by making water insoluble calcium soaps.

To show whether the colour difference in fellmongered wool was due to the root ends of the fibres, an experiment was performed in which the root ends were cut from the rest of the staple, and dyed separately under the same condition. Visually the difference in shade was apparent, which is also in confirmation with the earlier work.<sup>8</sup>

The difference in the rate of dye uptake results in differences in colour of fellmongered and shorn wool. It is obvious that there would be no difficulties in dyeing 100% fellmongered or shorn wool fabrics. As for efforts to minimise these differences, the deficiency in dye uptake of fellmongered wool alone and in blend with shorn wool employing good equalising acid dyes was improved by adding 5% H<sub>2</sub>SO<sub>4</sub> (concd) instead of 4% as usual. However by using 6% H<sub>2</sub>SO<sub>4</sub> (concd) no significant change in shade was observed resulting in wool damage.

Alternatively, dyeing of blends of the two types of wool gave good results with acid dyes, when it was pretreated with 1% H<sub>2</sub>SO<sub>4</sub> (concd). Experiments

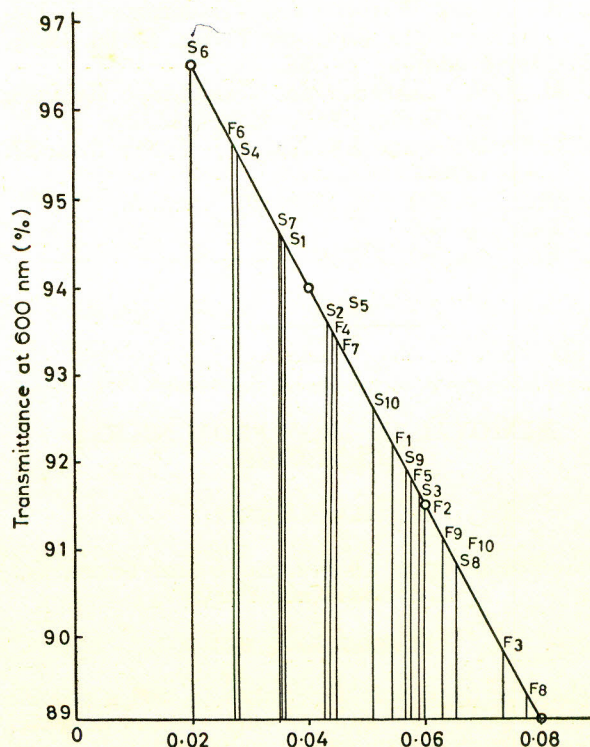


Fig. 1. Dye concentration (%).

revealed that the pretreated fellmongered wool when dyed separately gave the same shade as that of shorn wool without pretreatment.

It was observed that the scouring difficulties of sulphide painted fellmongered wool can be overcome, when it is scoured with polyethylenoxide<sup>9</sup> based nonionic detergent. The scouring can also be carried out with a hard water-resistant anionic<sup>10</sup> detergent or simply soap-soda process in the presence of a complex compound like calogen or trilon.<sup>11</sup>

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## REMOVAL OF IRON FROM NICKEL CHLORIDE

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The investigation is undertaken to find a suitable method for the removal of iron present in nickel chloride in order to make analytical grade nickel chloride. The conventional method which consists of addition of ammonium chloride and ammonium hydroxide did not work, because the amount of iron present is exceedingly small as compared with nickel ions. It is found that nickel hydroxide whose solubility product is about  $10^{-19}$  precipitates most of the iron as ferric hydroxide, whose solubility product is about  $10^{-38}$ .

### Experimental

A solution containing 500 g nickel chloride was made by heating a mixture of 226 g nickel powder and 1000 g HCl (concd). When whole nickel transferred into solution, it was cooled and 0.4 ml 20 volume  $H_2O_2$  was added and was thoroughly mixed. This solution which contained free acid was neutralized by heating with another 100 g nickel powder. Unreacted nickel was filtered off. The green filtrate (pH 3.6) was heated again with a paste of 16 g freshly prepared nickel hydroxide. After heating for 1 hr, unused hydroxide was filtered. The filtrate was crystallised after adjusting pH to 2.6 with N/1 HCl.

*Estimation of Iron in nickel Chloride.* Nickel chloride (5 g) was dissolved in 10 ml water. HCl (dil, 1 ml) and a drop of N/10  $KMnO_4$  were added to it. Ammonium thiocyanate (5 ml) was added which was followed by a mixture of 10 ml equal volumes of amyl alcohol and amyl acetate. The mixture was shaken vigorously and was allowed to separate. Lower layer was rejected. The upper layer was shaken thoroughly with 5 ml ammonium phosphate\*\* and 5 ml ammonium thiocyanate.\* The lower layer was

\*Ammonium thiocyanate (about 7.5 M) 57-g/. \*\*Ammonium phosphate (approximately M) diammonium hydrogen orthophosphate = 132 g water to produce 100 ml.

TABLE I

Experiment No.	Amount of iron (p.p.m)				
	Nickel powder	Nickel chloride		Nickel hydroxide	
		as such	Purified	before use	after use
1.	130	59	3	93	148
2.	133	60	3	95	152
3.	132	61	3	94	150
4.	135	62	3	96	155

separated and made into a total volume of 100 ml. To 20 ml of this solution, 2 ml HCl and 10 ml of a mixture of amyl alcohol and amyl acetate were added. The mixture was shaken vigorously and allowed to separate. Any colour in the upper layer should not be deeper than that obtained with a standard iron solution.

### Results and Discussion

Nickel chloride (analytical grade) could at one time be prepared direct from nickel powder. But the iron content of presently available nickel powder is such that the direct procedure cannot be used. The iron content of nickel powder and its salts before and after purification are given in Table I.

In the analytical grade nickel chloride, the amount of iron must be less than 10 p.p.m.<sup>1</sup> The amount of iron present in the nickel powder was in the range of 130–135 p.p.m. The nickel chloride prepared from this powder contained iron which varied from 59–62 p.p.m. which was well above the acceptable limits. The conventional method was applied, but it did not work. The solubility product of nickel hydroxide is  $8.7 \times 10^{-19}$  whereas the solubility product of ferric hydroxide is far lower than that of nickel hydroxide.<sup>2</sup> In other words nickel hydroxide having higher solubility product will react with ferric salt producing less soluble ferric hydroxide. This principle worked satisfactory and most of the iron was removed in this way. In order to have this process working, the following points need attention. Ferrous iron must be converted into ferric state, because the solubility product of ferric hydroxide is  $3.8 \times 10^{-38}$  whereas the solubility product of ferrous hydroxide is  $4.8 \times 10^{-16}$ , higher than that of nickel hydroxide.<sup>2</sup> The use of hydrogen peroxide for conversion of ferrous into ferric, must be added in acidic solution. Secondly the slurry of nickel hydroxide must be added in neutral nickel chloride solution, otherwise it will react with free acid and would convert into nickel chloride. Thirdly the filtrate after the removal of iron, must be adjusted to pH 2.6, before crystallisation, otherwise the solution would not be clear.

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