# Establishing Optimum Bleaching Conditions for Whitening Canary Wool

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ABSTRACT *It's a genuine problem to bleach canary wool—an Indian variety of unevenly yellowish pigmented wool. Had the wool been evenly pigmented the problem would not have been so severe. While the lower half of the fibre is heavily pigmented, the upper half is lightly pigmented or not pigmented at all. However, whether pigmented or not, both the portions of the fibre require bleaching action. Through this work, it has been established how to whiten canary wool by resorting to optimum scouring with a non-ionic detergent, optimum mordanting and optimum activated peroxide bleaching. The statistical techniques that have been used for optimization are orthogonal array experimentation with appropriate transformation of the data in the form of $\bar{X}$, $-\ln(S)$, desirability function, loss function and distance function. For testing the equality of variances, Bartlett's Test has been used. This work has potentially tremendous economic impact from the standpoint of import substitution of raw wool and export promotion of worsted yarn and worsted suits and shirts.*

KEY WORDS: Mordanting, bleaching, Berger index, alkali solubility, OA, ANOVA, desirability function, loss function, distance function

## Introduction

Canary wool is generally obtained from sheep of hot and humid areas such as Arizona, North Mexico, South Africa, Australia, Spain, Portugal, the Middle East and India. It differs from ordinary wool by its permanent yellow colouration. The autumn clip is found to be canary stained while the spring clip is white. These canary-stained fleeces are unevenly coloured. Only the lower half is deeply stained while the upper half is either lightly stained or not stained at all. This uneven yellow staining is a very serious fault of the canary wool, since it cannot be dyed uniformly in pastel or bright shades. Consequently, its use in worsted suits and shirts is impossible.

## Objective

A lot of work has been done on the bleaching of pigmented animal fibres. However, these works were not specifically aimed at canary wool. And since canary wool discolouration does not respond to ordinary scouring treatments or ordinary bleaching treatments, it was decided that the optimum bleaching

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condition for canary wool should be determined by using an activated bleaching process or catalysed bleaching process.

## The Cause of Canary Colouration

A literature survey reveals that pigments are not uncommon in animal fibres. In animal (and human) hair two types of pigments occur (Bereck, 1994).

(a) Eumelanin: responsible for black, dark brown and grey colours, and commonly referred to as melanin.
(b) Pheomelanin: present in yellow, reddish brown and red hair.

The structure and biosynthesis of melanin is not exactly known. Eumelanin and Pheomelanin are thought to be formed by different mechanisms, and differences in their chemical structures are indicated by their visible absorption spectra. Melanin is inherently attached to a protein matrix called melanoprotein. Its chemical composition differs greatly from that of keratin (fibre). Both Eumelanin and Pheomelanin occur in the form of discrete granules, which due to their high chemical resistance can be easily isolated from the fibre, e.g. by acid hydrolytic, reductive, or enzymatic degradation of the fibre keratin. The isolated granules show a rod-like structure, having a length of 0.5–1.3 $\mu$m and a width of 0.2–0.45 $\mu$m; the width/length ratio varies between 0.3 and 0.5. The melanin content can be as high as 10%.

## The Bleaching of Pigmented Fibres

Melanin pigments have a highly cross-linked structure and are extremely stable against chemical attack. As such, they are practically unaffected by reducing bleaching agents (Bereck, 1994). The only efficient way, known at present, to decompose and decolourize melanin is a treatment with oxidizing agents in an alkaline medium. Such treatments, however, cause substantial damage to the more sensitive fibre keratin. Since the pigmentation in canary wool is non-uniform, the bleaching action should be restricted primarily to the heavily pigmented portions. Laxer & Whewell (1955) were the first to realize that pigmented fibres absorb iron from ferrous sulphate ($FeSO_4$) solution more rapidly, and to a greater extent, than white fibres, probably due to the formation of a metal complex with the melanin of the pigment granules. The union between iron and the fibre is reasonably firm, and this bound iron is a useful catalyst for promoting bleaching when the iron-containing fibres are immersed in solutions of hydrogen peroxide ($H_2O_2$).

This technique of first allowing the pigmented fibres to absorb iron and then using this as a catalyst in a subsequent bleaching step was extensively studied and developed further by A. Bereck (1985, 1994). His process consists of three steps—mordanting, rinsing and bleaching. The first step consists of treating the fibre material with a solution of iron ($Fe^{++}$) sulphate. The fibres are then rinsed and finally bleached with hydrogen peroxide ($H_2O_2$). The presence of $Fe^{++}$ sulphate causes $H_2O_2$ to undergo radical decomposition, leading to oxidizing species that are far more aggressive than the perhydroxyl ion, which is generally

regarded as the bleaching agent under usual alkaline bleaching conditions. These radicals bring about not only a more complete disruption of the melanin polymer but are also very efficient in decolourizing the dye formed (Bereck, 1994). The three steps of the process are discussed in detail below.

## Mordanting

As mentioned earlier, mordanting consists of treating the wool fibres with a solution of a metal salt, which acts as a catalyst in the subsequent bleaching step. A comparison of different metal salts showed that only $Fe^{++}$ and $Fe^{+++}$ had significant catalytic activity (Bereck, 1985, 1994). The use of $Fe^{+++}$ led to the same bleaching effect as $Fe^{++}$, but the absorption of the former was not as selective; that is, both the pigmented and the unpigmented parts of the fibre absorbed $Fe^{+++}$ to more or less the same extent. This resulted in heavily damaged fibre keratin during bleaching. The use of $Fe^{++}$, on the other hand, offers several advantages: its absorption is highly selective under a wide range of conditions, its salts are relatively cheap and the iron content obtained in effluent water is comparatively high.

Bereck, (1985, 1994) made a detailed study of the conditions under which the iron salt can be applied to the fibre. It was found that increasing the concentration of $Fe^{+}$ ions above 0.035 mol per litre did not significantly enhance the iron uptake by the pigmented fibres. The appropriate conditions were found to be:

pH: 3.0–3.5
Treatment Time: 60 minutes
Temperature: 80°C

The presence of chelating agents such as EDTA, citric acid, oxalic acid or tartaric acid impaired the selectivity of absorption.

One of the most important findings of Bereck's study was that it was necessary to add a suitable reducing agent to the bleaching bath in order to prevent the oxidation of $Fe^{++}$ to $Fe^{+++}$. This is of great importance since the oxidized product lacks the ability of being selectively absorbed by the fibres. It transpired that hypophosphorus acid $(H_3PO_2)$ is an excellent stabilizer for $Fe^{++}$ ions under mordanting condition (Bereck, 1994). More importantly, cystine hardly suffered any attack under conditions that favour disulphide scission induced by sulphur-containing reducing agents. Formic acid (HCOOH) is usually used to bring down the pH to the range 3.0–3.5.

## Rinsing

The rinsing operation immediately follows the mordanting step, and is of immense importance, especially with regard to the selectivity of absorption and consequently to fibre damage. The iron–melanin interaction is, as Laxer & Whewell (1955) postulated, apparently much stronger than the iron–keratin interaction. This fact can be utilized in the selective desorption of iron so that the absorbed metal is completely removed from the fibre keratin and is localized exclusively at the pigment granules. The activated bleaching will therefore be

restricted to the pigment granules and the fibre keratin will undergo a simple peroxide bleaching. It has been recommended (Bereck, 1994) that rinsing be carried out in two stages each of a 10 minutes duration and at a temperature of 80°C. The recommended concentration of hypophosphorus acid ($H_3PO_2$) is 1 gram per litre.

## Bleaching with Hydrogen Peroxide

The amount of hydrogen peroxide (30% w/w) used ranges from 35–55 ml per litre. The pH is adjusted to 8.0–8.5 with ammonium hydroxide and the temperature is maintained in the range of 50°–60°C. The bleaching time varies from 1–2 hours. Tetrasodium pyrophosphate was found to be the most suitable stabilizing agent (Bereck, 1985). When used at a concentration of 10 grams per litre it renders a substantial degree of fibre protection. It may be noted that $HO_2$ and $HO_2$ free radicals, formed on the surface of, or within the fibre, bring about the enhanced bleaching action.

## Role of the Reducing Agent

In the activated hydrogen peroxide bleaching process, every precaution is taken to ensure that the retention of the ferrous ions is selective. In spite of this there is some non-selective retention of ferrous ions on wool which, upon subsequent treatment with hydrogen peroxide, leads to an undesirable light brown discolouration from the ferric species. Giving the wool a second bleaching treatment with a reducing agent has solved this problem.

In a bleaching process, as much as 60–90% of the original $H_2O_2$ can remain unutilized at the end of the process (Bereck, 1994). Arifoglu & Marmer (1990, 1992) developed an ingenious method for utilizing this spare peroxide. They converted it into the strong reducing agent, thiourea dioxide, by adding thiourea to the liquor. They also showed that, in order to effect complete conversion of thiourea to thiourea dioxide in the bleaching bath, the pH of the bath has to be lowered to 4.0–5.0 and maintained in this range for at least 10 minutes. The thiourea dioxide thus formed is a powerful reductive bleaching agent. At the end of the period, the pH of the medium should be adjusted to about 7.0 (Arifoglu et al., 1992).

Thus, at the end of the peroxide bleaching stage, the bleaching bath is made slightly acidic (with acetic acid) and then the required amount of thiourea is added; after about 10 minutes the pH is raised to about 7.0 with ammonium hydroxide, and bleaching is carried out for a further 10–20 minutes. This treatment enhances the whiteness of the fibres considerably.

## The Present Work

The literature on pigment removal from animal fibres mentions mordanting, rinsing and bleaching processes. Since canary wool does not respond to ordinary scouring treatment, the literature does not mention much about scouring—the purpose of which is the removal of wastes such as suint, wax, dirt and foreign

particles from wool fibre. However, through this current work it has been observed that prior to carrying out mordanting, rinsing and bleaching processes of canary wool, scouring can be done by non-ionic detergent—Alfox 200. The recipe of this scouring process is similar to the recipe for the scouring of merino wool used for worsted shirts and suits. The recipe for the scouring of merino wool is:

Detergent (Alfox 200): 1.5% on the weight of material (o.w.m.)
Alkali ($Na_2CO_3$): 0.15% o.w.m.
Temperature: 40°–45°C
Material:Liquor Ratio: 1:30
Time: 20 minutes

It should be noted that, in order to make worsted yarn out of the wool fibre through the spinning process, the grease recovery after scouring should be between 0.3 to 0.4%.

However, the above recipe as such was not found to be very effective for canary wool since the post-scouring grease content was found to be much less than 0.3%. Hence, a small experiment was carried out with two technologically important factors—the concentration of Alfox 200 and the concentration of alkali ($Na_2CO_3$). Two levels were considered for each factor. The levels of other factors remained unaltered. The purpose of this experiment is to find the optimum levels of the concentration of Alfox 200 and the concentration of alkali ($Na_2CO_3$) so that post-scouring loss is the minimum and the corresponding grease content is between 0.3 to 0.4% on the weight of the material. The experimental results are given in Table 1.

It has been observed that, corresponding to trial number 2, the residual grease content is 0.34%. For trials 1 and 3 it is found to be less than 0.3% and for trial 4 it is found to be 0.399%. Hence, trial 2 is considered as the optimum scouring recipe, which is given in the following.

Detergent (Alfox 200): 2.5% on the weight of material (o.w.m.)
Alkali ($Na_2CO_3$): 0.15% o.w.m.
Temperature: 45°C
Material:Liquor Ratio: 1:30
Time: 20 minutes

Note that the grease content percentage of raw wool was found to be 0.499.

Table 1. Result of the scouring experiment

| Trial | Initial sample weight (g) | Alfox 200 (%) | $Na_2CO_3$ (%) | Temp. (°C) | Time (min.) | Water (c.c.) | Final sample weight (g) | Weight loss (g) | Loss (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 2.01 | 1.5 | 0.15 | 45 | 20 | 60 | 1.85 | 0.16 | 7.96 |
| 2 | 2.00 | 2.5 | 0.15 | 45 | 20 | 60 | 1.84 | 0.17 | 8.50 |
| 3 | 2.01 | 1.5 | 0.20 | 45 | 20 | 60 | 1.83 | 0.18 | 8.96 |
| 4 | 2.00 | 2.5 | 0.20 | 45 | 20 | 60 | 1.86 | 0.14 | 7.00 |

## The Method of Residual Grease Test

One gram of scoured wool is treated with 5–6 ml of dichloromethane (DCM). It acts as an organic solvent and dissolves the residual grease. Subsequently, the mixture of residual grease and DCM is gradually heated to about 70°C to evaporate the DCM. The residual grease is weighed by an electronic balance. The residual grease percentage is calculated based on scoured wool weight.

## Experiments Carried out at the Mordanting Stage

### *Objective*

The objective is to achieve a unique optimal factor level combination of a response in an orthogonal array experimentation under a heteroscedastic situation. In this problem the response is iron retention by wool fibres, which is to be maximized with minimum variation.

### *Background Information*

In this problem, the response is the extent of iron retention in wool in grams. In order to escalate the bleaching action, it is necessary to maximize the iron retention after scouring with the optimum recipe.

Based on the literature survey and technical discussions, it has been found that the amount of iron retained by wool is influenced by a number of factors (see Table 2), such as:

(1) $FeSO_4$ concentration (factor A)
(2) Initial pH (factor B)
(3) Hypo 1 (Hypo phosphorous acid $H_3PO_2$ concentration at mordanting bath) (factor C)
(4) Mordanting temperature (factor D)
(5) Mordanting time (factor E)
(6) Rinsing temperature (factor F)
(7) Rinsing time (factor G)
(8) Hypo 2 (Hypo phosphorous acid $H_3PO_2$ concentration at rinsing bath) (factor H)

**Table 2.** Selection of the factors and their levels

| Factors | Level 1 | Level 2 | Level 3 |
|---|---|---|---|
| $FeSO_4$ concentration (g/l) [A] | 4.0 | 8.0 | 12.0 |
| Initial pH [B] | 2.0 | 3.5 | 5.0 |
| Hypo 1 (ml/l) [C] | 1.0 | 3.0 | 5.0 |
| Mordanting temperature (°C) [D] | 50 | 70 | 90 |
| Mordanting time (min) [E] | 30 | 60 | 90 |
| Rinsing temperature (°C) [F] | 50 | 70 | 90 |
| Rinsing time (min) [G] | 10 | 20 | 30 |
| Hypo 2 (ml/l) [H] | 0.0 | 1.0 | 2.0 |

It may be worth mentioning here that an interaction between factors is nothing but a change in the effect of one factor due to a change in the levels of other factors. From a technological standpoint, the following interactions were considered for carrying out the experiment.

(1) FeSO$_4$ concentration and mordanting temperature (A × D).
(2) Initial pH and mordanting temperature (B × D).

Since the nature of the effect (linear or nonlinear) of various factors was not known beforehand, it was decided to consider three equidistant levels for each factor. Again, based on the literature survey and technological considerations, the above levels were chosen. The material:liquor ratio should be 1:30.

### Apparatus and Instruments

All treatments are done in an Open Bath Beaker Dyeing Machine, with temperature control and a timer facility. A digital pH meter sets the pH of the bath. Iron is estimated by the titration method using burettes and pipettes.

### Selection of the Orthogonal Array (Ross, 1989)

There were eight factors, which we believe have an influence on mordanting, each of which can assume three levels. If a full factorial experiment were attempted then $3^8$ (=6561) tests would have been performed. Since it was infeasible to carry out such a huge number of experiments, the orthogonal array technique was used to facilitate the estimation of the effects of relevant factors and two-factor interactions. The minimum required degrees of freedom in an experiment are nothing but the sum total of all factors and interaction degrees of freedom. While selecting a particular OA, the following inequality must be satisfied.

$$v_{LN} \geqslant v_{\text{required for factors and interactions}} \tag{1}$$

where $v_{LN}$ = total degrees of freedom available in an orthogonal array = number of trails minus one = $N - 1$.

For this experiment, there are eight factors with $8 \times 2 = 16$ degrees of freedom and two interactions with $2 \times 4 = 8$ degrees of freedom. Hence,

$$v_{\text{required for factors and interactions}} = 16 + 8 = 24$$

Since the $L_{27}$ orthogonal array consists of 26 total degrees of freedom, $v_{LN} = 26$ and it can be found that through the selection of the $L_{27}$ orthogonal array, condition (1) gets satisfied.

### Assignment of Factors and Interactions to the $L_{27}$ Array

Linear graphs are used to aid in this process. Each Orthogonal Array has a particular set of linear graphs associated with it. They indicate the various columns to which factors may be assigned and also the columns of the array,
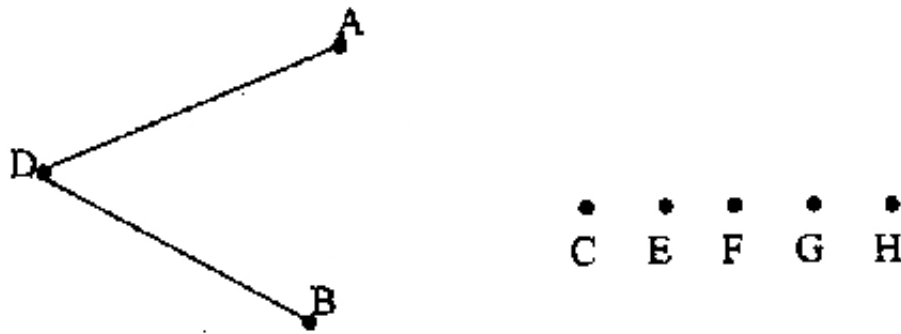
**Figure 1.** Required linear graph



**Figure 2.** Standard linear graph



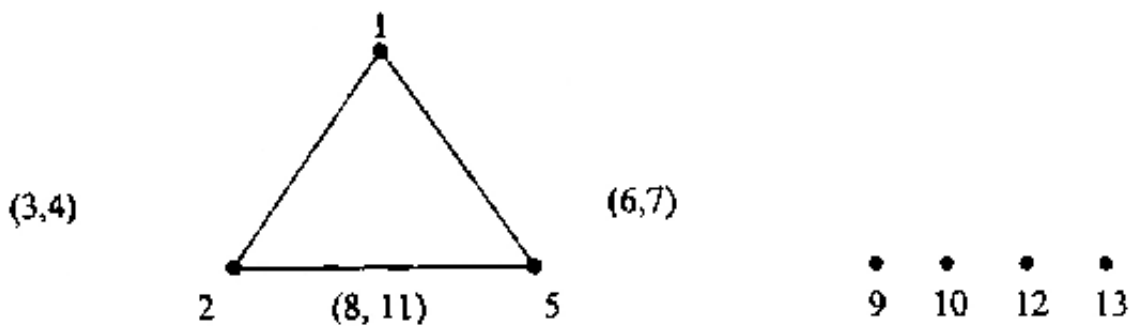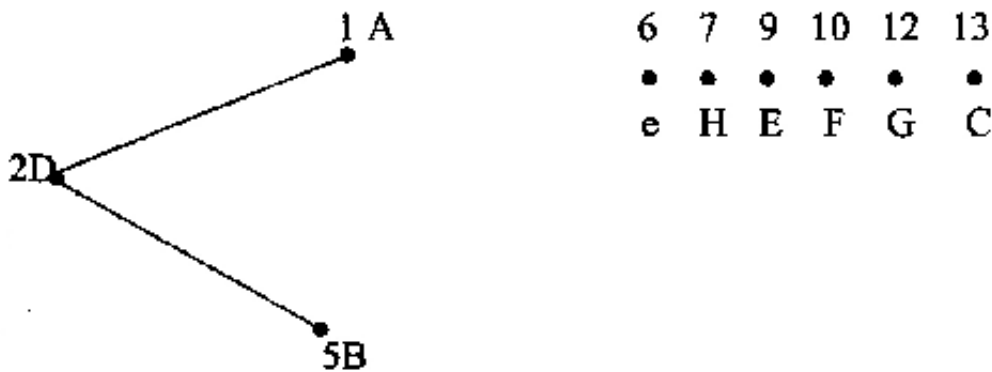**Figure 3.** Modified linear graph

which can subsequently be used to evaluate the interaction of the factors. The nodes used in Figures 1–3 represent the columns available for a three-level factor and the lines joining two nodes represent the two columns, which together evaluate the relevant interaction.

Hence, the assignment of factors and interactions took place in the following manner.

| Factors | Column no. |
|---------|------------|
| A | 1 |
| B | 5 |
| C | 13 |
| D | 2 |
| E | 9 |
| F | 10 |
| G | 12 |
| H | 7 |

| Interactions | Column no. |
|--------------|------------|
| $A \times D$ | 3,4 |
| $B \times D$ | 8,11 |

### About the Response--Iron Retention

For determining the $Fe^{++}$ ions absorbed by wool during the mordanting and rinsing stage, the amount of $Fe^{++}$ ions in the initial mordanting liquor before introducing the wool sample is determined. Le it be $A$ grams. Then the post-mordanting amount of $Fe^{++}$ ions present in the mordanting liquor is determined. Let it be $B$ grams. Then the amount of $Fe^{++}$ ions is determined in the rinsed solution. It represents the amount of $Fe^{++}$ being selectively given up by wool. Let it be $C$ grams. Hence, the net amount of $F^{++}$ ions absorbed by the canary wool sample is given by $X = (A-B-C)$.

The amount of $Fe^{++}$ present in the solution is determined by titrating the acidified ferrous solution with standard potassium dichromate ($K_2Cr_2O_7$) solution using sodium diphenylamine sulphonate (0.2% aqueous solution) as the indicator along with $H_3PO_4$ acid (Vogel, 1978).

### Titration

25 ml iron solutions are taken out with the help of a pipette and poured into a 500 ml beaker. Then, six to eight drops of the indicator [Na-Diphenylamine Sulphonate (0.2% aqueous solution)] are added along with 5 cc of syrupy $H_3PO_4$ acid, and titration is carried out with the $K_2Cr_2O_7$ solution from the burette with constant stirring. The volume of the $K_2Cr_2O_7$ solution is noted when the colour changes to green and then to violet.

### Statistical Approach

As the iron retention has two replicates, it is necessary to check the homoscedasticity of these two replicates (Montgomery, 1991). Therefore, first, Bartlett's Test is conducted (see Appendix 1). Based on the test we have rejected the null

Table 3. The $L_{27}$ orthogonal array

| Trial no | | | | | | | Columns | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 |
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| 2 | 1 | 1 | 1 | 1 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| 3 | 1 | 1 | 1 | 1 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| 4 | 1 | 2 | 2 | 2 | 1 | 1 | 1 | 2 | 2 | 2 | 3 | 3 | 3 |
| 5 | 1 | 2 | 2 | 2 | 2 | 2 | 2 | 3 | 3 | 3 | 1 | 1 | 1 |
| 6 | 1 | 2 | 2 | 2 | 3 | 3 | 3 | 1 | 1 | 1 | 2 | 2 | 2 |
| 7 | 1 | 3 | 3 | 3 | 1 | 1 | 1 | 3 | 3 | 3 | 2 | 2 | 2 |
| 8 | 1 | 3 | 3 | 3 | 2 | 2 | 2 | 1 | 1 | 1 | 3 | 3 | 3 |
| 9 | 1 | 3 | 3 | 3 | 3 | 3 | 3 | 2 | 2 | 2 | 1 | 1 | 1 |
| 10 | 2 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| 11 | 2 | 1 | 2 | 3 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 |
| 12 | 2 | 1 | 2 | 3 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 |
| 13 | 2 | 2 | 3 | 1 | 1 | 2 | 3 | 2 | 3 | 1 | 3 | 1 | 2 |
| 14 | 2 | 2 | 3 | 1 | 2 | 3 | 1 | 3 | 1 | 2 | 1 | 2 | 3 |
| 15 | 2 | 2 | 3 | 1 | 3 | 1 | 2 | 1 | 2 | 3 | 2 | 3 | 1 |
| 16 | 2 | 3 | 1 | 2 | 1 | 2 | 3 | 3 | 1 | 2 | 2 | 3 | 1 |
| 17 | 2 | 3 | 1 | 2 | 2 | 3 | 1 | 1 | 2 | 3 | 3 | 1 | 2 |
| 18 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 2 | 3 | 1 | 1 | 2 | 3 |
| 19 | 3 | 1 | 3 | 2 | 1 | 3 | 2 | 1 | 3 | 2 | 1 | 3 | 2 |
| 20 | 3 | 1 | 3 | 2 | 2 | 1 | 3 | 2 | 1 | 3 | 2 | 1 | 3 |
| 21 | 3 | 1 | 3 | 2 | 3 | 2 | 1 | 3 | 2 | 1 | 3 | 2 | 1 |
| 22 | 3 | 2 | 1 | 3 | 1 | 3 | 2 | 2 | 1 | 3 | 3 | 2 | 1 |
| 23 | 3 | 2 | 1 | 3 | 2 | 1 | 3 | 3 | 2 | 1 | 1 | 3 | 2 |
| 24 | 3 | 2 | 1 | 3 | 3 | 2 | 1 | 1 | 3 | 2 | 2 | 1 | 3 |
| 25 | 3 | 3 | 2 | 1 | 1 | 3 | 2 | 1 | 2 | 1 | 2 | 1 | 3 |
| 26 | 3 | 3 | 2 | 1 | 3 | 1 | 3 | 1 | 3 | 2 | 3 | 2 | 1 |
| 27 | 3 | 3 | 2 | 1 | 3 | 2 | 1 | 2 | 1 | 3 | 1 | 3 | 2 |

hypothesis of equality of variances for iron retention corresponding to the 27-treatment combinations (Table 3). Hence, carrying out ANOVA with the individual 54 observations, or with 27 higher, the better type signal-to-noise ratios are dispensed with. Instead, a better approach is adopted to isolate the location parameter and the dispersion parameter by separate analysis of $Y$ and $-\ln(S)$ (Montgomery, 1991). Since all the standard deviations are found to be less than unity, analysis of $-\ln(S)$ has been done instead of log $S$. The analyses are performed as:

(1) univariate analysis of variance for $Y$,
(2) univariate analysis of variance for $-\ln(S)$.

*Data*

Refer to Appendix 2 for $Y$ and $-\ln(S)$ transformation of iron retention data (Table 4).

**Table 4.** Experimental data

| Trial no | A | D | B | H | E | F | G | C | Fe retention replicate (1) | Fe retention replicate (2) |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 4 | 50 | 2.0 | 0.0 | 30 | 50 | 10 | 1.0 | 0.01368 | 0.01354 |
| 2 | 4 | 50 | 3.5 | 1.0 | 60 | 70 | 20 | 3.0 | 0.04104 | 0.03204 |
| 3 | 4 | 50 | 5.0 | 2.0 | 90 | 90 | 30 | 5.0 | 0.06840 | 0.04650 |
| 4 | 4 | 70 | 2.0 | 0.0 | 60 | 70 | 30 | 5.0 | 0.02736 | 0.01635 |
| 5 | 4 | 70 | 3.5 | 1.0 | 90 | 90 | 10 | 1.0 | 0.01368 | 0.01283 |
| 6 | 4 | 70 | 5.0 | 2.0 | 30 | 50 | 20 | 3.0 | 0.05472 | 0.03492 |
| 7 | 4 | 90 | 2.0 | 0.0 | 90 | 90 | 20 | 3.0 | 0.05472 | 0.03456 |
| 8 | 4 | 90 | 3.5 | 1.0 | 30 | 50 | 30 | 5.0 | 0.05472 | 0.04572 |
| 9 | 4 | 90 | 5.0 | 2.0 | 60 | 70 | 10 | 1.0 | 0.02736 | 0.02536 |
| 10 | 8 | 50 | 2.0 | 2.0 | 60 | 90 | 20 | 5.0 | 0.08208 | 0.08109 |
| 11 | 8 | 50 | 3.5 | 0.0 | 90 | 50 | 30 | 1.0 | 0.06840 | 0.05430 |
| 12 | 8 | 50 | 5.0 | 1.0 | 30 | 70 | 10 | 3.0 | 0.04104 | 0.03105 |
| 13 | 8 | 70 | 2.0 | 2.0 | 90 | 50 | 10 | 3.0 | 0.02736 | 0.02167 |
| 14 | 8 | 70 | 3.5 | 0.0 | 30 | 70 | 20 | 5.0 | 0.05472 | 0.05321 |
| 15 | 8 | 70 | 5.0 | 1.0 | 60 | 90 | 30 | 1.0 | 0.06840 | 0.03670 |
| 16 | 8 | 90 | 2.0 | 2.0 | 30 | 70 | 30 | 1.0 | 0.04104 | 0.01107 |
| 17 | 8 | 90 | 3.5 | 0.0 | 60 | 90 | 10 | 3.0 | 0.08208 | 0.07201 |
| 18 | 8 | 90 | 5.0 | 1.0 | 90 | 50 | 20 | 5.0 | 0.06840 | 0.05811 |
| 19 | 12 | 50 | 2.0 | 1.0 | 90 | 70 | 30 | 3.0 | 0.05472 | 0.05472 |
| 20 | 12 | 50 | 3.5 | 2.0 | 30 | 90 | 10 | 5.0 | 0.08208 | 0.08006 |
| 21 | 12 | 50 | 5.0 | 0.0 | 60 | 50 | 20 | 1.0 | 0.06840 | 0.04311 |
| 22 | 12 | 70 | 2.0 | 1.0 | 30 | 90 | 20 | 1.0 | 0.02736 | 0.02603 |
| 23 | 12 | 70 | 3.5 | 2.0 | 60 | 50 | 30 | 3.0 | 0.01368 | 0.01283 |
| 24 | 12 | 70 | 5.0 | 0.0 | 90 | 70 | 10 | 5.0 | 0.02736 | 0.02603 |
| 25 | 12 | 90 | 2.0 | 1.0 | 60 | 50 | 10 | 5.0 | 0.06840 | 0.04311 |
| 26 | 12 | 90 | 3.5 | 2.0 | 90 | 70 | 20 | 1.0 | 0.01368 | 0.01283 |
| 27 | 12 | 90 | 5.0 | 0.0 | 30 | 90 | 30 | 3.0 | 0.04104 | 0.01107 |

## Analaysis and Results at the Mordanting Stage

*Analysis of Variance for Response $Y$*

In order to carry out the analysis of variance for response $Y$ (Table 5, Table 6) the following calculations have been performed.

(1) The sum of squares for each factor is calculated by using the following equation

$$SS_{FACTOR} = \Sigma i[(A_i)^2/n_{Ai}] - CF$$

where $A_i$ = the sum of observations under the $i$th level of factor $A$,
$n_{Ai}$ = number of observations under the $i$th level of factor $A$.

(2) The formula for

$$SS_{INTERACTION} = \Sigma i \Sigma j[(A_i \times B_j)^2/n_{ij}] - SS_A - SS_B - CF$$

where $n_{ij}$ = number of observations under the $ij$th level combination of factors $A$ and $B$ or any other concerned factors.

**Table 5.** ANOVA for average iron retention

| Sources of variation (SOV) | Sum of squares (SS) | Degrees of freedom (DF) | Mean square (MS) | F ratio | Contribution % |
|---|---|---|---|---|---|
| A | 0.00168 | 2 | 0.00084 | 1.62 | 5.57 |
| B | 0.00016 | 2 | 0.00008 | 0.15 | 0.00 |
| C | 0.00237 | 2 | 0.00118 | 2.29 | 11.57 |
| D | 0.00224 | 2 | 0.00112 | 2.17 | 10.42 |
| E | 0.00028 | 2 | 0.00014 | 0.27 | 0.00 |
| F | 0.00151 | 2 | 0.00076 | 1.46 | 4.15 |
| G | 0.00029 | 2 | 0.00014 | 0.28 | 0.00 |
| H | 0.00002 | 2 | 0.00001 | 0.02 | 0.00 |
| A × D | 0.00133 | 4 | 0.00033 | 0.64 | 0.00 |
| B × D | 0.00064 | 4 | 0.00016 | 0.31 | 0.00 |
| e (empty column) | 0.00103 | 2 | 0.00052 | | |
| Total | 0.01155 | 26 | | | |

$F_{0.05,2,2} = 19$    $F_{0.01,2,2} = 99$
$F_{0.05,4,2} = 19.25$    $F_{0.01,4,2} = 99.2$

**Table 6.** ANOVA for average iron retention (after pooling)

| SOV | SS | DF | MS | F | Cont. % |
|---|---|---|---|---|---|
| A | 0.00168 | 2 | 0.00084 | 4.01 | 14.31 |
| C | 0.00237 | 2 | 0.00118 | 5.67 | 20.31 |
| D | 0.00224 | 2 | 0.00112 | 5.36 | 19.16 |
| F | 0.00151 | 2 | 0.00076 | 3.62 | 12.89 |
| Pooled error | 0.00021 | 18 | 0.00001 | | |
| Total | 0.00801 | 26 | | | |

$F_{0.05,2,18} = 3.55$ and $F_{0.01,2,18} = 6.01$

(3) The formula for contribution

$$\text{contribution} \% = \frac{SS - DF \times MS_E}{SS_T} \times 100$$

where $SS$ = sum of squares of the concerned factor
$DF$ = degrees of freedom of the concerned factor
$MS_E$ = mean square error

*Analysis of Variance for Response* $-\ln(S)$

See Tables 7 and 8.

## Optimum Iron Retention Values for Significant Factor Levels

In order to select the optimum levels of the various factors, the sum of the response tables is used (Tables 9 to 13). It can be recalled that both $\bar{Y}$ and $-\ln(S)$ are to be maximized (see Figures 4 to 8).

Table 7. ANOVA for $-\ln(S)$ of iron retention

| Sources of variation (SOV) | Sum of squares (SS) | Degrees of freedom (DF) | Mean square (MS) | F ratio | Contribution % |
|---|---|---|---|---|---|
| A | 13.71418 | 2 | 6.85709 | 3.81 | 7.43 |
| B | 17.03560 | 2 | 8.51780 | 4.73 | 9.87 |
| C | 1.10893 | 2 | 0.55446 | 0.31 | 0.00 |
| D | 13.70487 | 2 | 6.85244 | 3.80 | 7.42 |
| E | 8.40873 | 2 | 4.20437 | 2.33 | 3.53 |
| F | 10.74647 | 2 | 5.37323 | 2.98 | 5.25 |
| G | 1.59195 | 2 | 0.79597 | 0.44 | 0.00 |
| H | 2.96799 | 2 | 1.48400 | 0.82 | 0.00 |
| A×D | 6.02797 | 4 | 1.50699 | 0.84 | 0.00 |
| B×D | 57.19097 | 4 | 14.29774 | 7.94 | 36.73 |
| e (empty column) | 3.60354 | 2 | 1.80177 | | |
| Total | 136.10119 | 26 | | | |

$F_{0.05,2,2}=19$  $F_{0.01,2,2}=99$
$F_{0.05,4,2}=19.25$  $F_{0.01,4,2}=99.25$

Table 8. ANOVA for $-\ln(S)$ of iron retention (after pooling)

| SOV | SS | DF | MS | F | Cont. % |
|---|---|---|---|---|---|
| A | 13.71418 | 2 | 6.85709 | 64.54 | 9.92 |
| B | 17.03560 | 2 | 8.51780 | 80.17 | 12.36 |
| D | 13.70487 | 2 | 6.85243 | 64.49 | 9.91 |
| E | 8.40873 | 2 | 4.20437 | 39.57 | 6.02 |
| F | 10.74647 | 2 | 5.37323 | 50.57 | 7.74 |
| B×D | 57.19097 | 4 | 14.29774 | 134.56 | 41.71 |
| Pooled error | 1.27503 | 12 | 0.10625 | | |
| Total | 136.10119 | 26 | | | |

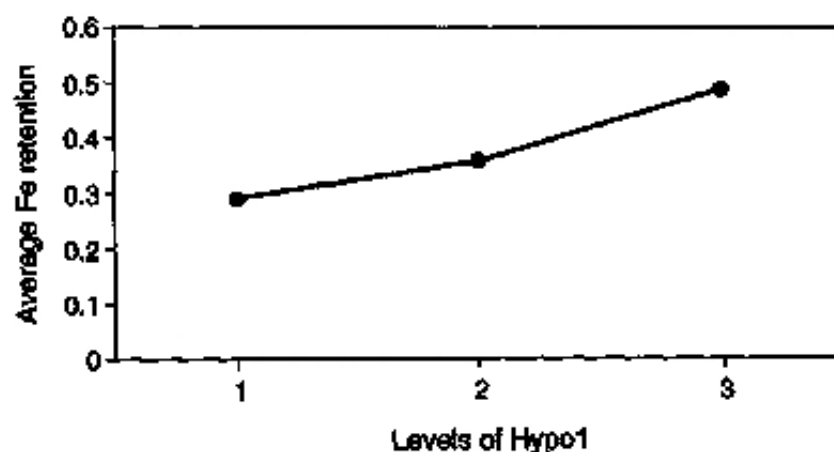$F_{0.05,2,12}=3.89$  $F_{0.01,2,12}=6.93$  $F_{0.05,4,12}=3.26$  $F_{0.01,4,12}=5.41$



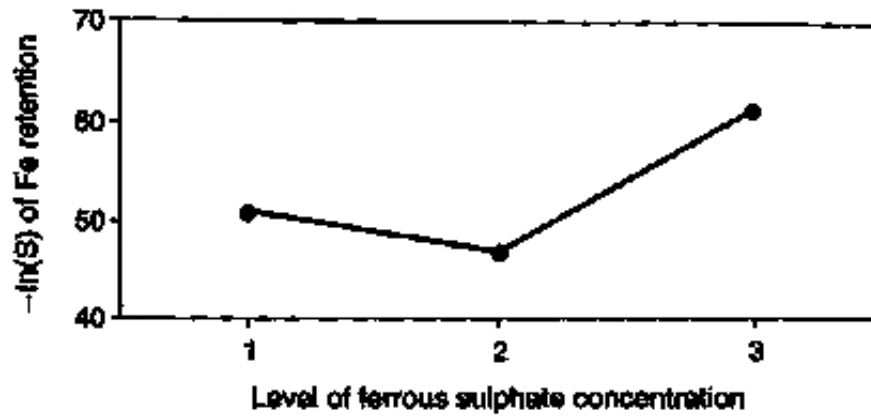Figure 4. Effect of Hypo 1 concentration on average iron retention

**Figure 5.** Effect of ferrous sulphate concentration on $-\ln(S)$ of Fe retention
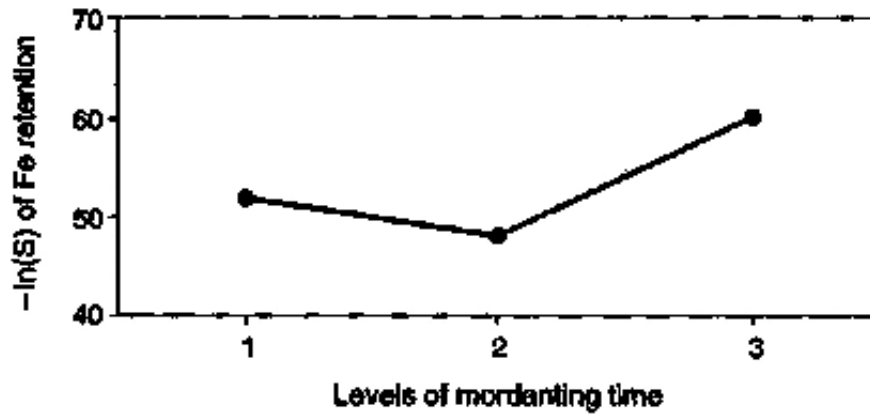


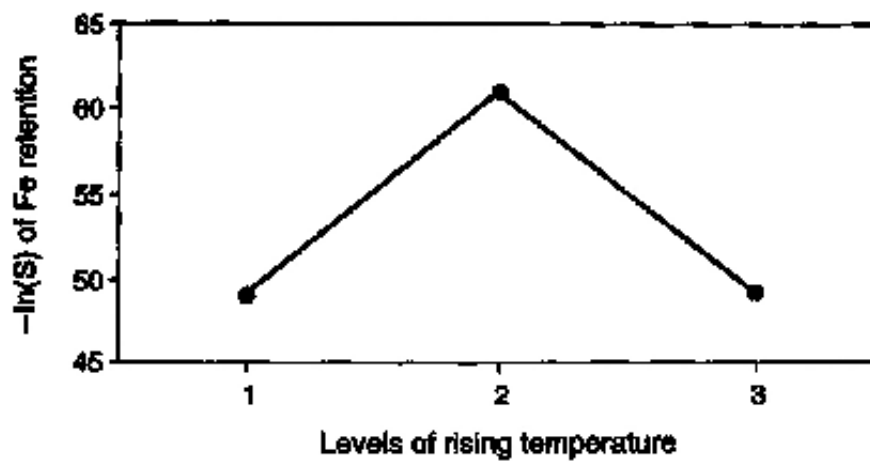**Figure 6.** Effect of mordanting time on $-\ln(S)$ of iron retention



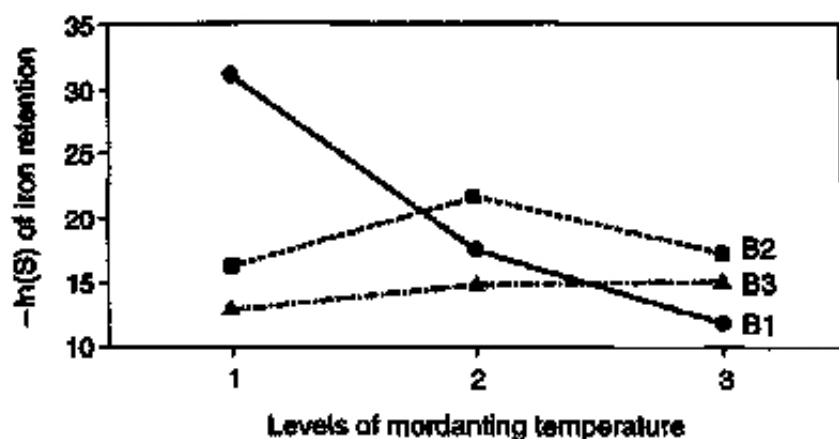**Figure 7.** Effect of rinsing temperature on $-\ln(S)$ of iron retention

**Figure 8.** Interaction between initial pH (*B*) and mordanting temperature (*D*) on $-\ln(S)$ of iron retention

**Table 9.** The sum of iron retention values for different levels of factor $C$ for $Y$

| Levels of factor $C$-Hypol (ml/l) | Sum of Fe retention (g) |
|---|---|
| $C_1$ (1.0) | 0.289 |
| $C_2$ (3.0) | 0.358 |
| $C_3$ (5.0) | 0.492 |

**Table 10.** The sum of $-\ln(S)$ of iron retention for factor A

| Levels of factor $A$ (FeSO$_4$ Conc.) | Sum of $-\ln(S)$ values of Fe retention (g) |
|---|---|
| $A_1$ (gm/l) | 50.856 |
| $A_2$ (gm/l) | 46.701 |
| $A_3$ (gm/l) | 61.090 |

**Table 11.** The sum of $-\ln(S)$ of iron retention for factor $E$

| Levels of factor $E$ (mordanting time) | Sum of $-\ln(S)$ of Fe retention (g) |
|---|---|
| $E_1$ (30 min) | 51.566 |
| $E_2$ (60 min) | 47.950 |
| $E_3$ (90 min) | 59.942 |

**Table 12.** The sum of $-\ln(S)$ of iron retention for factor $F$

| Levels of factor $F$ (rinsing temperature) | Sum of $-\ln(S)$ of Fe retention (g) |
|---|---|
| $F_1$ (50°) | 49.063 |
| $F_2$ (70°) | 61.182 |
| $F_3$ (90°) | 49.213 |

**Table 13.** The sum of $-\ln(S)$ of iron retention for $B \times D$ interaction

| Levels of initial pH ($B$) | Levels of mordanting temperature ($D$) | | |
|---|---|---|---|
| | 50°C | 70°C | 90°C |
| 2.0 | 31.156 | 17.341 | 12.129 |
| 3.5 | 16.214 | 21.681 | 17.419 |
| 5.0 | 13.144 | 15.036 | 15.339 |

## Conclusion and Recommendations on Mordanting

Since the contribution of factor $C$ (Hypol concentration in ml/l) is about 20.31% towards average iron retention ($\bar{Y}$) variation and it does not contribute to the variation of $-\ln(S)$, this factor is to be considered as a signal factor for adjusting the mean. This is acceptable from a technological standpoint also.

The other factors, namely $A$ (FeSO$_4$ concentration), $E$ (mordanting time), $F$ (rinsing temperature) and $B \times D$ (interaction between initial pH and mordanting temperature) contribute about 87.66% towards the variability of the response $-\ln(S)$. These factors are to be considered as control factors.

The optimum factor level combination for mordanting of canary wool is given in the following table.

**Table 14.** Optimum levels of factors for mordanting

| Factors | Optimum levels | Purpose |
|---|---|---|
| FeSO$_4$ concentration ($A$) | 3rd level (12.0 g/l) | Reducing noise |
| Initial pH ($B$) | 1st level (2.0) | Reducing noise |
| Hypo 1 ($C$) | 3rd level (5.0 ml/l) | Adjusting the signal |
| Mordanting temperature ($D$) | 1st level (50°C) | Reducing noise |
| Mordanting time ($E$) | 3rd level (90 minutes) | Reducing noise |
| Rinsing temperature ($F$) | 2nd level (70°C) | Reducing noise |

One can keep factors $G$ (rinsing time) and $H$ (Hypo2 concentration) at any of the three levels chosen for experimentation based on economic and other considerations. And the material:liquor ratio should be 1:30.

## Estimated Mean and Confidence Interval

The estimated mean for ($\bar{Y}$) is given by

$$\hat{\mu} = \bar{C}_3 + \bar{T} - \bar{T} = 0.0547$$

$$CI = \sqrt{\frac{F_{\alpha,1,V_e} \overline{V_e}}{\eta_{eff}}}$$

where $\eta_{eff} = N/[1 + [\text{Total degrees of freedom associated with items used in } \hat{\mu}$ estimate]]

$V_e =$ error variance
$\alpha = 0.05$
$v_e =$ error degrees of freedom

$$\eta_{eff} = \frac{27}{1+2} = 9$$

$$CI = \sqrt{\frac{4.41 \times 0.000011592}{9}} = 0.00238$$

The estimated mean for $-\ln(S)$ is given by

$$\hat{\mu}_{A3B1D1E3F2} = \bar{A}_3 + \bar{B}_1\bar{D}_1 + \bar{E}_3 + \bar{F}_2 - 3\bar{T}$$

$$\hat{\mu} = 6.788 + 10.385 + 6.660 + 6.798 - 3 \times 5.906$$

$$= 12.913$$

$$\eta_{eff} = \frac{27}{1+10} = 2.45$$

$$CI = \sqrt{\frac{4.75 \times 0.10625}{2.45}} = 0.454$$

## Experiments carried out at the Bleaching Stage after Optimal Mordanting

After going through the available literature regarding this matter, it is found that there are seven potential factors for removal of canary wool colouration. This removal of colouration whitens the wool and the whiteness is measured by an index called the Berger index. Simultaneously, the tensile strength is measured by the alkali solubility of the fibres.

### Factors and Levels

Three levels are considered for each of these potential factors, based on technological considerations from the available literature on the subject (Bereck, 1985, 1994; Arifoglu & Marmer, 1990, 1992; Earland & Little, 1985; Trollip *et al.*, 1985; Ceggara *et al.*, 1998; Leven, 1997). The material:liquor ratio should be 1: 30. The factors and levels are identified as in Table 15.

In addition to the seven main factors, two interactions are thought to be of influence. These are the interactions between:

(1) $H_2O_2$ concentration (*A*) and bleaching time (*C*);
(2) $H_2O_2$ concentration (*A*) and bleaching bath temperature (*D*).

There are seven main factors and two 2-factor interactions, giving a total of 22

Table 15. The selected factors and corresponding levels for bleaching

| Factors | Levels | | |
| --- | --- | --- | --- |
| | 1 | 2 | 3 |
| Hydrogen peroxide concentration ($A$) | 35 ml $l^{-1}$ | 45 ml $l^{-1}$ | 55 ml $l^{-1}$ |
| Bleaching bath pH ($B$) | 9.5 | 10.0 | 10.5 |
| Bleaching time ($C$) | 1 hr | 1.5 hr | 2 hr |
| Bleaching bath temperature ($D$) | 50°C | 55°C | 60°C |
| Thiourea concentration ($E$) | 20% on weight of material (o.w.m.) | 30% (o.w.m.) | 40% (o.w.m.) |
| Post-bleaching time 1 ($F$) | 10 min | 20 min | 30 min |
| Post-bleaching time 2 ($G$) | 10 min | 20 min | 30 min |

degrees of freedom. The Orthogonal Array (OA) $L_{27}$ has 26 degrees of freedom. Hence, $L_{27}$ is the chosen OA. The Linear Graph used in this case is as follows.
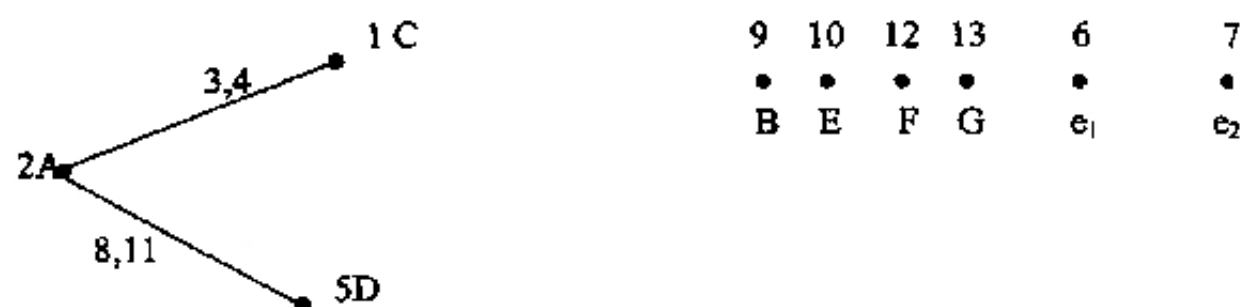


The two responses considered in this experiment are (a) the Berger index and (b) the alkali solubility.

(a) The Berger index indicates the extent of whiteness of the wool. As the Berger index increases, the whiteness of the canary wool increases.
(b) The alkali solubility, on the other hand, measures the tensile strength of fibres. The greater is the alkali solubility, the less is the tensile strength of fibres. It is to be noted that the bleaching process may affect the tensile strength of fibres. Therefore, an increase in Berger index may lead to an unwanted increase in alkali solubility.

## Objective of the Bleaching Process

The objective is to achieve a unique optimal factor level combination in the orthogonal array experimentation under a multi-response situation, keeping in view two responses—the Berger index, which is to be maximized, and the alkali solubility, which is to be minimized.

**Table 16.** Experimental data at the peroxide bleaching stage

| | | | | | | | | Berger index | | | Berger index | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Trial no | C 1 | A 2 | D 5 | B 9 | E 10 | F 12 | G 13 | 1 | 2 | Alkali solubility (%) | Mean | Standard Deviation |
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 14.05 | 14.06 | 48 | 14.055 | 0.00707 |
| 2 | 1 | 1 | 2 | 2 | 2 | 2 | 2 | 14.39 | 14.52 | 22 | 14.455 | 0.09192 |
| 3 | 1 | 1 | 3 | 3 | 3 | 3 | 3 | 3.28 | 3.66 | 14 | 3.470 | 0.26870 |
| 4 | 1 | 2 | 1 | 2 | 2 | 3 | 3 | 12.28 | 11.36 | 16 | 11.820 | 0.65054 |
| 5 | 1 | 2 | 2 | 3 | 3 | 1 | 1 | 3.40 | 3.28 | 12 | 3.340 | 0.08485 |
| 6 | 1 | 2 | 3 | 1 | 1 | 2 | 2 | 12.13 | 12.64 | 20 | 12.385 | 0.36062 |
| 7 | 1 | 3 | 1 | 3 | 3 | 2 | 2 | 4.51 | 7.16 | 14 | 5.835 | 1.87383 |
| 8 | 1 | 3 | 2 | 1 | 1 | 3 | 3 | 6.79 | 5.46 | 22 | 6.125 | 0.94045 |
| 9 | 1 | 3 | 3 | 2 | 2 | 1 | 1 | −0.81 | −0.90 | 18 | −0.855 | 0.06364 |
| 10 | 2 | 1 | 1 | 2 | 3 | 2 | 3 | 1.36 | 1.86 | 22 | 1.610 | 0.35355 |
| 11 | 2 | 1 | 2 | 3 | 1 | 3 | 1 | 12.79 | 12.79 | 40 | 12.880 | 0.12728 |
| 12 | 2 | 1 | 3 | 1 | 2 | 1 | 2 | 16.12 | 15.69 | 14 | 15.905 | 0.30406 |
| 13 | 2 | 2 | 1 | 3 | 1 | 1 | 2 | 14.90 | 14.27 | 26 | 14.585 | 0.44548 |
| 14 | 2 | 2 | 2 | 1 | 2 | 2 | 3 | 18.22 | 18.63 | 34 | 18.425 | 0.28991 |
| 15 | 2 | 2 | 3 | 2 | 3 | 3 | 1 | 11.62 | 11.41 | 28 | 11.515 | 0.14849 |
| 16 | 2 | 3 | 1 | 1 | 2 | 3 | 1 | 10.05 | 11.76 | 18 | 10.905 | 1.20915 |
| 17 | 2 | 3 | 2 | 2 | 3 | 1 | 2 | 4.88 | 4.97 | 22 | 4.925 | 0.06364 |
| 18 | 2 | 3 | 3 | 3 | 1 | 2 | 3 | 4.38 | 3.97 | 24 | 4.175 | 0.28991 |
| 19 | 3 | 1 | 1 | 3 | 2 | 3 | 2 | 0.03 | 0.44 | 14 | 0.235 | 0.28991 |
| 20 | 3 | 1 | 2 | 1 | 3 | 1 | 3 | 17.66 | 18.09 | 14 | 17.875 | 0.30406 |
| 21 | 3 | 1 | 3 | 2 | 1 | 2 | 1 | 16.57 | 16.33 | 28 | 16.450 | 0.16971 |
| 22 | 3 | 2 | 1 | 1 | 3 | 2 | 1 | 17.91 | 16.59 | 18 | 17.250 | 0.93338 |
| 23 | 3 | 2 | 2 | 2 | 1 | 3 | 2 | 18.85 | 17.80 | 22 | 18.325 | 0.74246 |
| 24 | 3 | 2 | 3 | 3 | 2 | 1 | 3 | −1.22 | −1.60 | 32 | −1.410 | 0.26870 |
| 25 | 3 | 3 | 1 | 2 | 1 | 1 | 3 | 18.29 | 19.19 | 30 | 18.740 | 0.63640 |
| 26 | 3 | 3 | 2 | 3 | 2 | 2 | 1 | 5.96 | 4.94 | 44 | 5.450 | 0.72125 |
| 27 | 3 | 3 | 3 | 1 | 3 | 3 | 2 | 21.74 | 23.28 | 34 | 22.510 | 1.08894 |

*Statistial Approach*

Since the Berger index has two replicates, it is necessary to check the homoscedasticity of these two replicates under different treatment conditions. So, first Bartlett's test is conducted (see Appendix 3) and we fail to reject the null hypothesis of equality of variances corresponding to the 27 treatment combinations.

Subsequently the following analyses are carried out:

- univariate analysis of variance for each of the response,
- univariate analysis of variance considering the desirability function of the responses,
- univariate analysis of variance considering the loss function of the responses,

- univariate analysis of variance considering the distance function of the responses.

## Various Functions Used for Analysis

### *The Desirability Function.*

This is a function approach developed initially by Harrington (1965) to optimize the multi-response situation. Later, Derringer & Suich (1980), generalized this approach to handle a large number of responses.

For the average Berger index (the higher the index the better type response) the Desirability Function used is

$$
di = \begin{cases} 0 & \hat{Y}_i \leqslant Y_{i*} \\ \left[\dfrac{\hat{Y}_i - Y_{i*}}{Y_i^* - Y_{i*}}\right]^r & Y_{i*} < \hat{Y}_i < Y_i^* \\ 1 & \hat{Y}_i \geqslant Y_i^* \end{cases}
$$

whereas for alkali solubility (the lower the better type response) the Desirability Function used is

$$
di = \begin{cases} 0 & \hat{Y}_i \geqslant Y_i^* \\ \left[\dfrac{Y_i^* - \hat{Y}_i}{Y_i^* - Y_{i*}}\right]^r & Y_i^* \geqslant \hat{Y}_i \geqslant Y_{i*} \\ 1 & Y_{i*} \geqslant \hat{Y}_i \end{cases}
$$

where

$\hat{Y}_i$ is the response,
$Y_{i*}$ is the minimum acceptable value,
$Y_i^*$ is the maximum acceptable value.

In this problem for the Berger index, the value of $Y_{i*}$ is considered as 0 and the value of $Y_i^*$ is considered as 25.

For alkali solubility, the value of $Y_{i*}$ is considered as 10 and that of $Y_i^*$ is considered as 50.

We can obtain the overall Desirability Function ($D$) for a treatment combination for the responses as $D = (\prod_{i=1}^{n} d_i)^{1/n}$

The overall desirability varies from 0 to 1 and the most desirable value of the desirability function is 1. The value of the exponent $r$ in the desirability function is taken between 0 to 1 and the value is chosen according to the importance of that response. In this problem, both the responses are considered to be of same importance. Accordingly, three values of $r$ are taken into consideration: $r = 0.5$, $r = 0.75$ and $r = 1$ for both responses. The desirability values are given in Appendix 4.

## The Loss Function

The loss function is used to quantify losses associated with a deviation from the desired target values for all the responses.

$$\text{Loss Function } (L) \text{ is defined as } L = \sum_{i=1}^{n} \frac{W_i |Y_i - T_i|}{S_i}$$

Where

$n$ = number of responses,
$Y_i$ = $i$th response value. For the Berger index, the average is considered as the response,
$T_i$ = the target value of the $i$th response. It is average for the Berger index and an individual value for alkali solubility,
$S_i$ = standard error of the $i$th response,
$W_i$ = weight for the $i$th response.

An estimate of the standard error is obtained by dividing the square root of the error mean square of a response by the square root of the number of replicates. According to the loss function, the optimum set-up will be selected where the loss is minimum.

It is found that both responses are equally important. It is therefore decided to choose the weight of both responses as 0.5. The values of the loss function are given in Appendix 4.

## The Distance Function

Khuri & Conlon (1981) introduced an approach using a distance function considering the variance and correlation among the responses.

$$\text{The Distance Function is defined as } d = \left[ \frac{\{(Y-T)' \Sigma^{-1} (Y-T)\}}{\{Z'(x) (X'X)^{-1} Z(X)\}} \right]^{0.5}$$

where

$Y$ = vector of response,
$T$ = vector of target response,
$Z(x)$ = vector of factor level for a given combination,
$X$ = design matrix,
$\Sigma$ = sample variance covariance matrix of responses.

According to the distance function, the optimum combination will be selected where the distance is a minimum. The values of the distance function are given in Appendix 4.
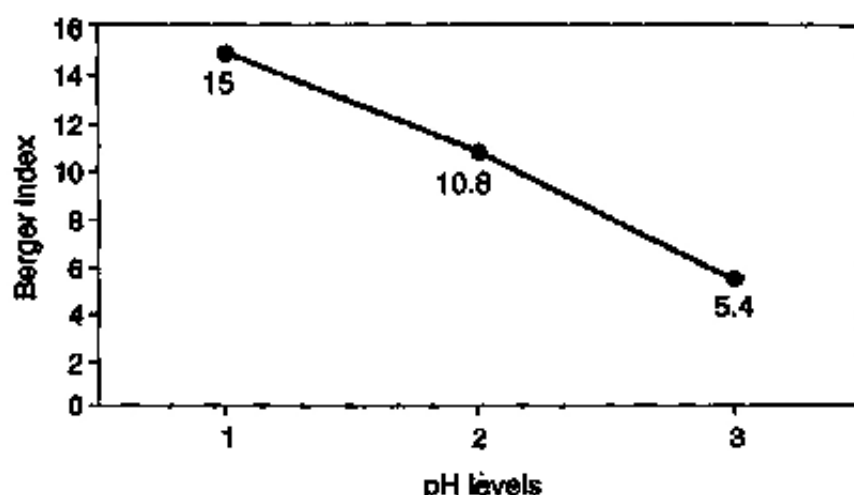
**Figure 9.** Effect of the initial bleaching bath pH on Berger index

## Results After Analysis at the Bleaching Stage

Hence, through separate univariate analysis of variance, it is found that factor $B$ (pH of the bleaching bath) does effect the Berger index and its preferred level is level one, that is, 9.5. Even though the contribution percentage of the $A \times D$ ($H_2O_2$ concentration × bleaching bath temperature) interaction is found to be 18.24, the computed $F$ value (5.75) is found to be less than the critical $F$ value (6.39) at 5% level of significance.

No factor is found to be significant for alkali solubility.

It is to be noted that for $r = 0.5$ the contributions of $B$, $E$ and $A \times D$ are found to be on the higher side. However, the corresponding $F$ ratios are found to be insignificant.

**Table 17.** ANOVA for the Berger index

| SOV | SS | DF | MS | $F_0$ | Cont. % | $F_{critical}$ $\alpha = 0.05$ |
|---|---|---|---|---|---|---|
| $A$ | 93.351 | 2 | 46.675 | 1.94 | 1.80 | 6.94 |
| $B$ | 842.270 | 2 | 421.135 | 17.47* | 31.60 | 6.94 |
| $C$ | 223.488 | 2 | 111.744 | 4.64 | 6.98 | 6.94 |
| $D$ | 35.264 | 2 | 17.632 | 0.73 | 0.00 | 6.94 |
| $E$ | 212.913 | 2 | 106.456 | 4.42 | 6.55 | 6.94 |
| $F$ | 14.420 | 2 | 7.210 | 0.30 | 0.00 | 6.94 |
| $G$ | 91.553 | 2 | 45.777 | 1.90 | 1.73 | 6.94 |
| $A \times C$ | 337.585 | 4 | 84.396 | 3.50 | 9.60 | 6.39 |
| $A \times D$ | 554.735 | 4 | 138.684 | 5.75 | 18.24 | 6.39 |
| $e_1$ | 42.663 | 2 | 21.331 | | | |
| $e_2$ | 53.744 | 2 | 26.872 | | | |
| $e_1 + e_2$ | 96.407 | 4 | 24.102 | | | |
| Replication error | 10.943 | 27 | 0.405 | | | |
| Total | 2512.929 | 53 | | | | |

**Table 18.** ANOVA for akali solubility

| SOV | SS | DF | MS | $F_0$ | $F_{critical}$ at $\alpha = 0.05$ |
|---|---|---|---|---|---|
| A | 18.072 | 2 | 9.036 | <1 | 6.94 |
| B | 12.739 | 2 | 6.369 | <1 | 6.94 |
| C | 160.294 | 2 | 80.147 | <1 | 6.94 |
| D | 41.183 | 2 | 20.592 | <1 | 6.94 |
| E | 377.183 | 2 | 188.592 | 1.64 | 6.94 |
| F | 18.072 | 2 | 9.036 | <1 | 6.94 |
| G | 254.517 | 2 | 127.258 | 1.10 | 6.94 |
| $A \times C$ | 798.817 | 4 | 199.704 | 1.73 | 6.39 |
| $A \times D$ | 277.928 | 4 | 69.482 | <1 | 6.39 |
| $e_1$ | 10.961 | 2 | 5.481 | | |
| $e_2$ | 450.072 | 2 | 225.036 | | |
| $e_1 + e_2$ | 461.033 | 4 | 115.258 | | |
| Total | 241.838 | 26 | | | |

Now let us see the results of these various functions.

**Table 19.** ANOVA for desirability function with $r = 1$

| SOV | SS | DF | MS | $F_0$ | $F_{0.05}$ | Cont. % |
|---|---|---|---|---|---|---|
| A | 0.0734 | 2 | 0.0369 | 2.092 | 6.94 | 2.76 |
| B | 0.3562 | 2 | 0.1781 | 10.100 | 6.94 | 23.23 |
| C | 0.0340 | 2 | 0.0170 | 0.965 | 6.94 | 0.00 |
| D | 0.0218 | 2 | 0.0109 | 0.618 | 6.94 | 0.00 |
| E | 0.0693 | 2 | 0.0347 | 1.967 | 6.94 | 2.47 |
| F | 0.0296 | 2 | 0.0148 | 0.840 | 6.94 | 0.00 |
| G | 0.1035 | 2 | 0.0517 | 2.936 | 6.94 | 4.94 |
| $A \times C$ | 0.0417 | 4 | 0.0104 | 0.592 | 6.39 | 0.00 |
| $A \times D$ | 0.5815 | 4 | 0.1453 | 8.248 | 6.39 | 36.98 |
| $e_1$ | 0.0425 | 2 | 0.0213 | | | |
| $e_2$ | 0.0279 | 2 | 0.0139 | | | |
| $e_1 + e_2$ | 0.0704 | 4 | 0.0176 | | | |
| Total | 1.3814 | 26 | | | | |

It is to be noted that, for the distance function, the contributions of $B$, $G$ and $A \times D$ are found to be on the higher side. However, the corresponding $F$ ratios are found to be insignificant.

Based on the desirability functions ($r = 1$, $r = 0.75$) and the loss function, the optimal factor level is found as $A_2 B_1 D_1$.

## Conclusions

From the analysis of variance of the desirability function (for $r = 1$, $r = 0.75$ and $r = 0.5$), loss function and distance function, it is found that for maximizing the Berger index (whiteness) and minimizing alkali solubility (fibre tendering) while

Table 20. ANOVA for desirability function with $r = 0.75$

| SOV | SS | DF | MS | $F_0$ | $F_{0.05}$ | Cont. % |
|---|---|---|---|---|---|---|
| A | 0.0605 | 2 | 0.0302 | 1.593 | 6.94 | 1.56 |
| B | 0.3307 | 2 | 0.1653 | 8.703 | 6.94 | 20.34 |
| C | 0.0310 | 2 | 0.0155 | 0.818 | 6.94 | 0.00 |
| D | 0.0369 | 2 | 0.0184 | 0.971 | 6.94 | 0.00 |
| E | 0.1083 | 2 | 0.0541 | 2.849 | 6.94 | 4.88 |
| F | 0.0536 | 2 | 0.0268 | 1.410 | 6.94 | 1.08 |
| G | 0.1039 | 2 | 0.0519 | 2.736 | 6.94 | 4.58 |
| $A \times C$ | 0.0682 | 4 | 0.0170 | 0.898 | 6.39 | 0.00 |
| $A \times D$ | 0.5699 | 4 | 0.1425 | 7.500 | 6.39 | 34.32 |
| $e_1$ | 0.0456 | 2 | 0.0228 | | | |
| $e_2$ | 0.0303 | 2 | 0.0152 | | | |
| $e_1 + e_2$ | 0.0759 | 4 | 0.0189 | | | |
| Total | 1.4389 | 26 | | | | |

Table 21. ANOVA for desirability function with $r = 0.5$

| SOV | SS | DF | MS | $F_0$ | $F_{0.05}$ | Cont. % |
|---|---|---|---|---|---|---|
| A | 0.0395 | 2 | 0.0198 | 0.920 | 6.94 | 0.00 |
| B | 0.2708 | 2 | 0.1354 | 6.308 | 6.94 | 15.63 |
| C | 0.0357 | 2 | 0.0179 | 0.832 | 6.94 | 0.00 |
| D | 0.0672 | 2 | 0.0336 | 1.565 | 6.94 | 1.67 |
| E | 0.1628 | 2 | 0.0814 | 3.793 | 6.94 | 8.23 |
| F | 0.0947 | 2 | 0.0474 | 2.206 | 6.94 | 3.55 |
| G | 0.0981 | 2 | 0.0490 | 2.285 | 6.94 | 3.78 |
| $A \times C$ | 0.1156 | 4 | 0.0289 | 1.346 | 6.39 | 2.04 |
| $A \times D$ | 0.4874 | 4 | 0.1219 | 5.676 | 6.39 | 27.54 |
| $e_1$ | 0.0502 | 2 | 0.0251 | | | |
| $e_2$ | 0.0357 | 2 | 0.0179 | | | |
| $e_1 + e_2$ | 0.0859 | 4 | 0.0215 | | | |
| Total | 1.4577 | 26 | | | | |

bleaching canary wool, the optimum levels of the common contributory factors are as shown in Table 24.

The other factors responsible for bleaching can be kept at any of the three levels based on economic or other considerations. The material:liquor ratio should be 1:30.

The following formulae suggested by Taguchi (Ross, 1989) are used to estimate the Predicted Value and Confidence Interval (CI) for different functions at the optimum treatment combination.

$$\text{Predicted Value} = \bar{A}_2 \bar{D}_1 + \bar{B}_1 - T$$
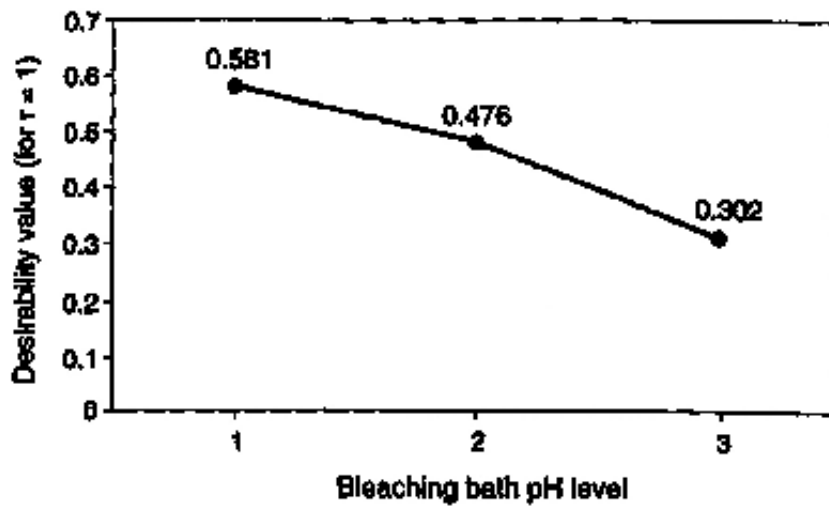
$$CI = \sqrt{\frac{F_{0.05, 1, f_e} \times V_e}{n}}$$
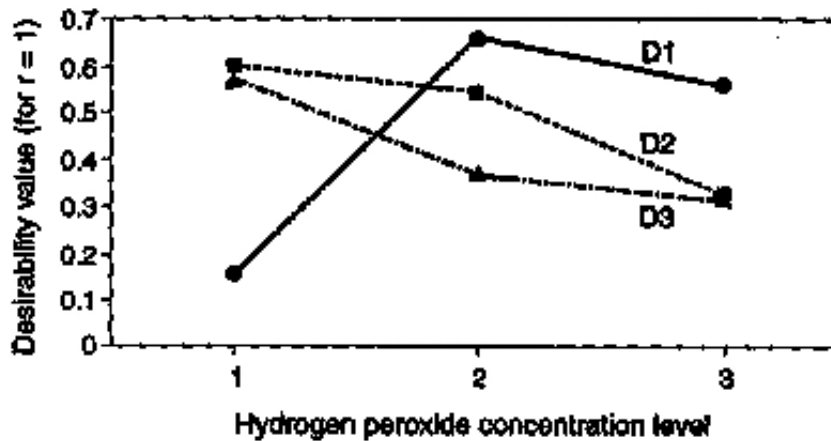
**Figure 10.** Effect of bleaching bath pH (factor B)



**Figure 11.** Interaction between hydrogen peroxide concentration (*A*) and bleaching bath temperature (*D*)



**Figure 12.** Effect of bleaching bath pH (factor B)

**Figure 13.** Interaction between hydrogen peroxide concentration (*A*) and bleaching bath temperature (*D*)
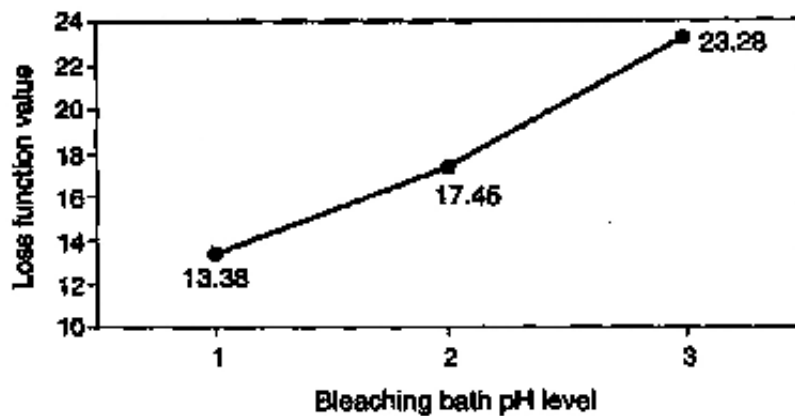


**Figure 14.** Effect of bleaching bath pH (factor B)



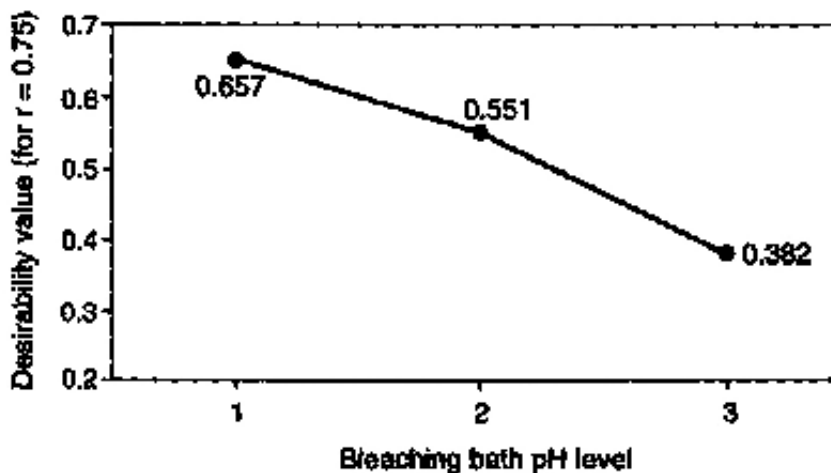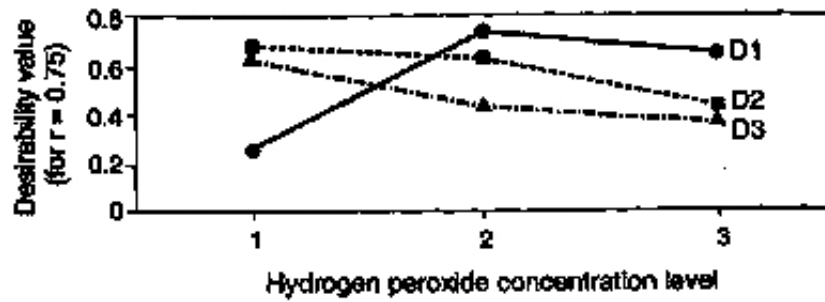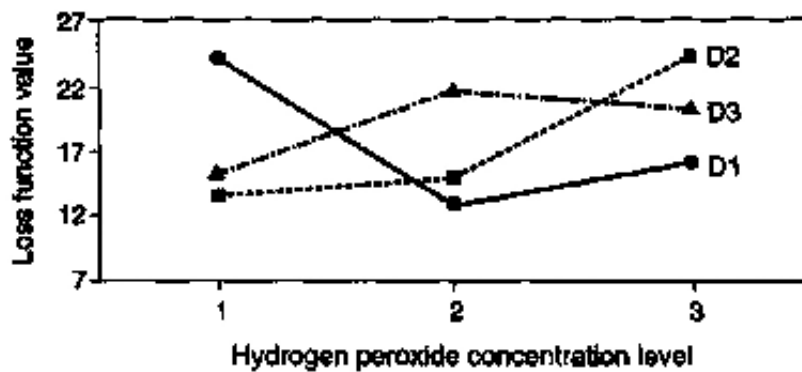**Figure 15.** Interaction between hydrogen peroxide concentration (*A*) and bleaching bath temperature (*D*)

where

$F_{0.05,1,v} = F$ ratio required
$V_e$ = error variance
and $n$ = number of tests under that condition.

Since the Desirability function has a narrower confidence interval, it is preferable compared to the Loss function or Distance function. Subsequent to carrying out

Table 22. ANOVA for loss function

| SOV | SS | DF | MS | $F_0$ | $F_{0.05}$ | Cont. % |
|---|---|---|---|---|---|---|
| A | 62.9961 | 2 | 31.498 | 5.22 | 6.94 | 4.05 |
| B | 445.1340 | 2 | 222.567 | 36.90 | 6.94 | 34.47 |
| C | 70.0463 | 2 | 35.023 | 5.80 | 6.94 | 4.61 |
| D | 13.0259 | 2 | 6.513 | 1.08 | 6.94 | 0.07 |
| E | 64.8127 | 2 | 32.406 | 5.37 | 6.94 | 4.20 |
| F | 8.9259 | 2 | 4.463 | 0.74 | 6.94 | 0.00 |
| G | 81.3185 | 2 | 40.660 | 6.74 | 6.94 | 5.51 |
| $A \times C$ | 73.4617 | 4 | 18.365 | 3.04 | 6.39 | 3.93 |
| $A \times D$ | 412.5874 | 4 | 103.147 | 17.09 | 6.39 | 30.91 |
| $e_1$ | 16.8898 | 2 | 8.445 | | | |
| $e_2$ | 7.2405 | 2 | 3.620 | | | |
| $e_1 + e_2$ | 24.1303 | 4 | 6.033 | | | |
| Total | 1256.439 | 26 | | | | |

Table 23. ANOVA for distance function

| SOV | SS | DF | MS | $F_0$ | $F_{0.05}$ | Cont. % |
|---|---|---|---|---|---|---|
| A | 3.158 | 2 | 1.579 | 0.338 | 6.94 | 0.00 |
| B | 37.395 | 2 | 18.697 | 4.004 | 6.94 | 15.77 |
| C | 2.238 | 2 | 1.119 | 0.240 | 6.94 | 0.00 |
| D | 2.865 | 2 | 1.433 | 0.307 | 6.94 | 0.00 |
| E | 5.443 | 2 | 2.722 | 0.583 | 6.94 | 0.00 |
| F | 4.628 | 2 | 2.314 | 0.496 | 6.94 | 0.00 |
| G | 20.510 | 2 | 10.255 | 2.196 | 6.94 | 6.28 |
| $A \times C$ | 23.744 | 4 | 5.936 | 1.271 | 6.39 | 2.85 |
| $A \times D$ | 59.226 | 4 | 14.806 | 3.171 | 6.39 | 22.80 |
| $e_1$ | 2.182 | 2 | 1.091 | | | |
| $e_2$ | 16.494 | 2 | 8.247 | | | |
| $e_1 + e_2$ | 18.676 | 4 | 4.669 | | | |
| Total | 177.883 | 26 | | | | |

Table 24. Optimum factor level combination for bleaching

| Serial number | Contributing factors | Optimum levels |
|---|---|---|
| 1 | $H_2O_2$ concentration (A) | 45 ml per litre (2nd level) |
| 2 | Initial pH of the bleaching bath (B) | 9.5 (1st level) |
| 3 | Bleaching bath temperature (D) | 50°C (1st level) |

ANOVA of the desirability values, one can conclude about the significance or otherwise of any factor by computing Taguchi's contribution percentage.

## Technical Impact of the Study

Through this experiment, the Berger index increased from 10 to about 22.5 at the cost of a slight increase in alkali solubility from 12% to about 14%. The confirmatory trial is given in Table 26.

**Table 25.** Predicted values and confidence intervals of various functions at the optimal condition

| Function | Predicted value | Confidence interval |
|---|---|---|
| Desirability value for $r = 1$ | 0.783810 | $\pm 0.368369$ |
| Desirability value for $r = 0.75$ | 0.853408 | $\pm 0.381732$ |
| Desirability value for $r = 0.5$ | 0.926361 | $\pm 0.407142$ |
| Loss function value | 8.242484 | $\pm 6.819923$ |
| Distance function value | 2.974662 | $\pm 5.999832$ |

**Table 26.** Results of confirmatory trial at the optimum bleaching condition

| Sample no. | Berger index | Mean & S.D. | Alkali solubility | Mean & S.D. |
|---|---|---|---|---|
| 1 | 22.2 | | 14.10 | |
| 2 | 22.7 | | 14.40 | |
| 3 | 23.1 | 22.46 & 1.045 | 14.27 | 14.11 & 0.688 |
| 4 | 20.8 | | 12.98 | |
| 5 | 23.5 | | 14.82 | |

*Likely Economic Impact of these Results*

Since canary wool has an uneven yellow colouration, it cannot be dyed in pastel or bright shades. This is a serious hurdle for the use of canary wool in worsted suits or shirts in blended form or otherwise and, as such, canary wool is primarily used for low-grade blankets or knitted fabrics in some under-developed countries.

In order to dye canary wool, the prerequisite is a uniform whiteness of fibre. However, canary wool is such a kind of fibre where only the lower half of the fibre is deeply yellow stained while the upper half is either lightly stained or not stained at all.

In this work it has been established that, by adopting an activated peroxide bleaching method, a uniform whiteness of canary wool fibre can be obtained in order to make the fibre suitable for dyeing and, in turn, suitable for suits and shirts, either on its own or by appropriate blending with other fibres, say, polyester. Some varieties of the canary wool can be used singly for suits and shirts, subject to satisfying other physical properties of the fibre, say, fineness, comfort etc. Incidentally, the fineness of the wool used in this experiment is around 29.15 $\mu$m.

It is to be noted that Merino wool has the lion's share of the contribution towards worsted suits and shirts, which are in great demand in cold countries. The price of Merino wool is about \$12 per kg. Considering that about 30–40% is the usage of wool fibres for the medium- and high-grade garment sector, compared with the usage of other fibres, namely cotton, synthetic, silk, flax etc, one can easily anticipate the tremendous impact of this work from the perspective of Business Management.

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## Appendix 1

*Bartlett's Test for Equality of Variance of Iron Retention*

This test may be viewed as a formal test of the hypothesis:

$$H_0 = \sigma_1^2 = \sigma_2^2 = \sigma_3^2 \ldots = \sigma_a^2$$

$$H_1 = \text{the above not true for at least one } \sigma_i^2$$

In Bartlett's test, the procedure involves computing a statistic whose sampling distribution is closely approximated by the chi-square distribution with $a-1$ degree of freedom, where the random samples are from an independent normal population.

The test statistic

$$\chi_0^2 = 2.3026 \frac{q}{c}$$

where

$$q = (N-a)\log S_p^2 - \sum_{i=1}^{a} (n-1)\log S_i^2$$

$$C = 1 + \frac{1}{3(a-1)} \left( \sum (n_i-1)^{-1} - (N-a)^{-1} \right)$$

and

$$S_p^a = \frac{\sum_{i=1}^{a} (n_i-1) S_i^2}{(N-a)}$$

and $S_i^2$ is the sample variance of the $i$th population, $a$ is the number of treatment combinations.

$N = an$
$n = $ no. of replications

*Decision criteria*

Reject $H_0$ when $\chi_o^2 > \chi_{a,a-1}^2$ where $\chi_{a,a-1}^2$ is the upper $\alpha$ percentage point of the chi-square distribution with $a-1$ degrees of freedom.

In this problem, the value of

$$S_p^2 = 0.000114771$$

$$\sum_{i=1}^{a} (n-1)\log S_i^2 = -138.502677$$

where $a = 27$

$$q = (N-a)\log S_p^2 - \sum_{i=1}^{a} (n-1)\log S_i^2$$

$$= 27\log 0.000114771 + 138.502677$$

$$= -106.3845449 + 138.502677$$

$$= 32.1181321$$

$$c = 1 + 1/(3 \times 26) [27 - 1/27]$$

$$= 1.345679012$$

Test statistic:

$$\chi_o^2 = 2.3026 \times 32.1181321/1.345679012$$

$$\approx 54.96$$

$$\chi_{0.05,26}^2 = 41.92$$

Conclusion: since $\chi_o^2 > \chi_{0.05,26}^2 = 41.92$, $H_0$ is rejected and it is concluded that there is a significant difference in the variances.

## Appendix 2

### $Y$ and $-\ln(S)$ transformation of iron retention

| $Y$ | $-\ln(S)$ |
|---|---|
| 0.01361 | 9.22044 |
| 0.03654 | 5.05722 |
| 0.05745 | 4.16792 |
| 0.02186 | 4.85548 |
| 0.01326 | 7.41702 |
| 0.04482 | 4.26860 |
| 0.04464 | 4.25066 |
| 0.05022 | 5.05706 |
| 0.02636 | 6.56168 |
| 0.08159 | 7.26256 |
| 0.06135 | 4.60815 |
| 0.03605 | 4.95264 |
| 0.02452 | 5.51597 |
| 0.05397 | 6.84156 |
| 0.05255 | 3.79801 |
| 0.02606 | 3.85411 |
| 0.07705 | 4.94482 |
| 0.06325 | 4.92274 |
| 0.05472 | 14.67291 |
| 0.08107 | 6.54827 |
| 0.05575 | 4.02375 |
| 0.02670 | 6.96915 |
| 0.01326 | 7.42233 |
| 0.02670 | 6.96915 |
| 0.05575 | 4.02375 |
| 0.01326 | 7.41702 |
| 0.02606 | 3.85411 |

## Appendix 3

*Statistical Tests for Equality of Variance--Bartlett's Test (Bleaching)*

This test may be viewed as a formal test of the hypothesis:

$$H_0 = \sigma_1^2 = \sigma_2^2 = \sigma_3^2 = \ldots = \sigma_n^2$$

$$H_1 = \text{the above not true for at least one } \sigma_i^2$$

In Bartlett's test the procedure involves computing a statistic whose sampling distribution is closely approximated by the chi-square distribution with $a-1$ degrees of freedom where the random samples are from an independent normal population.

The test statistic is

$$\chi_o^2 = 2.3026 \frac{q}{c}$$

where

$$q = (N-a)\log S_p^2 - \sum_{i=1}^{a} (n-1)\log S_i^2$$

$$C = 1 + \frac{1}{3(a-1)}\sum (n_i-1)^{-1} - (N-a)^{-1}$$

and

$$S_p^2 = \frac{\sum_{i=1}^{a} (n_i-1)S_i^2}{(N-a)}$$

where $S_i^2$ is the variance of each treatment combination.

If $\chi_o^2 > \chi_{a,a-1}^2$ then the hypothesis is rejected.

In this problem the value of

$$S_p^2 = \frac{12.7279}{27} = 0.4714$$

$$q = 27 \times \log(0.4714) - [\log(0.00707) + \log(0.09192) \ldots + \log(1.08894)]$$

$$= -8.81837 - [-14.4978]$$

$$= 5.679479$$

$$c = 1 + \frac{1}{3.26}\left(27 - \frac{1}{27}\right)$$

$$= 1 + \frac{1}{3.26} \times 26.963$$

$$= 1.3456$$

So the

$$\chi_o^2 = 2.3026 \times \frac{5.679479}{1.3456}$$

$$= 9.718763$$

Here at 95% *CI* the value of $\chi_o^2$ is 41.92. So we cannot reject the Null Hypothesis.

**Appendix 4. Transformed data**

| Trial no. | C | A | D | B | E | F | G | Berger index 1 | Berger index 2 | Alkali solubility % | r=1 | r=0.75 | r=0.5 | Loss function | Distance function |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 14.05 | 14.06 | 48 | 0.16766 | 0.26201 | 0.40946 | 19.3878 | 10.1000 |
| 2 | 1 | 1 | 2 | 2 | 2 | 2 | 2 | 14.39 | 14.32 | 22 | 0.63619 | 0.71235 | 0.79762 | 13.4230 | 5.9612 |
| 3 | 1 | 1 | 3 | 3 | 3 | 3 | 3 | 3.28 | 3.66 | 14 | 0.35344 | 0.40573 | 0.54806 | 23.0278 | 8.0083 |
| 4 | 1 | 2 | 1 | 1 | 2 | 2 | 3 | 12.28 | 11.36 | 16 | 0.63394 | 0.71045 | 0.79620 | 14.8554 | 4.4096 |
| 5 | 1 | 2 | 2 | 2 | 3 | 3 | 1 | 3.4 | 3.28 | 12 | 0.35626 | 0.46113 | 0.59687 | 22.7345 | 6.0544 |
| 6 | 1 | 2 | 3 | 3 | 1 | 1 | 2 | 12.13 | 12.64 | 20 | 0.60955 | 0.68985 | 0.78074 | 15.1278 | 4.8722 |
| 7 | 1 | 3 | 1 | 3 | 3 | 1 | 2 | 4.51 | 7.16 | 14 | 0.45832 | 0.55703 | 0.67700 | 20.5921 | 5.4402 |
| 8 | 1 | 3 | 2 | 1 | 1 | 2 | 3 | 6.79 | 5.46 | 22 | 0.41413 | 0.51624 | 0.64353 | 22.0020 | 5.7492 |
| 9 | 1 | 3 | 3 | 2 | 2 | 3 | 1 | -0.81 | -0.9 | 18 | 0.00000 | 0.00000 | 0.00000 | 28.3364 | 7.4173 |
| 10 | 2 | 1 | 1 | 2 | 3 | 2 | 3 | 1.36 | 1.86 | 22 | 0.21232 | 0.31278 | 0.46078 | 26.6520 | 7.9920 |
| 11 | 2 | 1 | 2 | 3 | 1 | 3 | 1 | 12.79 | 12.97 | 40 | 0.35889 | 0.46368 | 0.59907 | 18.8894 | 7.3335 |
| 12 | 2 | 1 | 3 | 1 | 2 | 1 | 2 | 16.12 | 15.69 | 14 | 0.75669 | 0.81131 | 0.86988 | 10.2211 | 0.0190 |
| 13 | 2 | 2 | 1 | 2 | 1 | 1 | 2 | 14.9 | 14.27 | 26 | 0.59164 | 0.67460 | 0.76918 | 14.1434 | 5.0358 |
| 14 | 2 | 2 | 2 | 3 | 2 | 2 | 3 | 18.22 | 18.63 | 34 | 0.54295 | 0.63252 | 0.73685 | 11.8972 | 8.7267 |
| 15 | 2 | 2 | 3 | 1 | 3 | 3 | 1 | 11.62 | 11.41 | 28 | 0.50332 | 0.59756 | 0.70945 | 17.7323 | 6.2689 |
| 16 | 2 | 3 | 1 | 3 | 2 | 3 | 2 | 10.05 | 11.76 | 18 | 0.59073 | 0.67382 | 0.76859 | 16.2249 | 4.9500 |
| 17 | 2 | 3 | 2 | 1 | 3 | 1 | 2 | 4.88 | 4.97 | 22 | 0.37135 | 0.47570 | 0.60938 | 23.2379 | 7.8181 |
| 18 | 2 | 3 | 3 | 2 | 1 | 2 | 3 | 4.38 | 3.97 | 24 | 0.32947 | 0.43487 | 0.57399 | 24.4374 | 8.2951 |
| 19 | 3 | 1 | 1 | 3 | 1 | 2 | 3 | 0.03 | 0.44 | 14 | 0.09198 | 0.16702 | 0.30328 | 26.3595 | 6.8522 |
| 20 | 3 | 1 | 2 | 1 | 2 | 3 | 1 | 17.66 | 18.09 | 14 | 0.80218 | 0.84763 | 0.89565 | 8.1922 | 2.1268 |
| 21 | 3 | 1 | 3 | 2 | 3 | 1 | 2 | 16.57 | 16.33 | 28 | 0.60158 | 0.68308 | 0.77562 | 12.6498 | 4.5357 |
| 22 | 3 | 2 | 1 | 2 | 2 | 3 | 2 | 17.91 | 16.59 | 18 | 0.74297 | 0.80025 | 0.86196 | 9.6902 | 3.2370 |
| 23 | 3 | 2 | 2 | 3 | 3 | 4 | 2 | 18.85 | 17.8 | 22 | 0.71631 | 0.77862 | 0.84635 | 9.4374 | 3.9766 |
| 24 | 3 | 2 | 3 | 1 | 1 | 1 | 3 | -1.22 | -1.6 | 32 | 0.00000 | 0.00000 | 0.00000 | 31.8979 | 11.0680 |
| 25 | 3 | 3 | 1 | 2 | 2 | 2 | 1 | 18.29 | 19.19 | 30 | 0.61221 | 0.69211 | 0.78244 | 10.7185 | 4.1693 |
| 26 | 3 | 3 | 2 | 3 | 3 | 2 | 1 | 5.96 | 4.94 | 44 | 0.18083 | 0.27730 | 0.42524 | 27.3957 | 11.5500 |
| 27 | 3 | 3 | 3 | 1 | 3 | 3 | 2 | 21.74 | 23.28 | 34 | 0.60013 | 0.68185 | 0.77468 | 7.6901 | 4.3947 |