

## Short Communication

### Optimisation of flax rove bleaching using Box-Behnken design of experiments

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Received 26 April 2023; revised received and  
accepted 26 December 2023

This study aims to optimise the flax rove bleaching process using Box Behnken design in combination with response surface methodology. The effects of hydrogen peroxide concentration, sodium hydroxide concentration, bleaching time, and temperature on whiteness index and weight loss are evaluated for their individual and interaction effects. Statistical analysis reveals that all process variables significantly affect the whiteness index, while sodium hydroxide concentration and temperature predominantly influence weight loss. Multi-response optimisation using a desirability function identified the optimum conditions as 0.09 kg/kg H<sub>2</sub>O<sub>2</sub>, 0.02 kg/kg NaOH, 60 min, and 91.3 °C, achieving a whiteness index of 70.4 and weight loss of 8.88%. The findings demonstrate the potential for attaining high whiteness with minimal fibre damage under optimised bleaching parameters.

**Keywords:** Bleaching loss, Box-Behnken, Design flax, Whiteness index, Weight loss

Flax (*Linum usitatissimum* L.) is one of the first natural fibres utilised by mankind. Flax fibres are obtained from the stalk of a flax plant. The flax is a lignocellulosic fibre, containing cellulosic and non-cellulosic impurities such as hemicellulose, pectin, lignin and natural waxes<sup>1</sup>. The raw flax fibres are coarse, rigid and exhibit poor spinnability. To make these fibres spinnable, they are subjected to a bleaching process. The bleaching removes the non-cellulosic impurities from the fibre and improves whiteness, fibre flexibility and degree of fibre splitting. In industrial operations, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is currently the most common bleaching agent used for flax bleaching. The bleaching is carried out using a large quantity of chemicals, various auxiliaries and water at high temperature and pressure<sup>2</sup>. A few studies were

conducted to examine the effect of bleaching & alkali process factors on whiteness, fibre strength and weight loss behaviour of flax fibres<sup>3-6</sup>. Dandan *et al.*<sup>3</sup> studied the single factor effect on whiteness, weight loss and breaking strength of flax fibre. They observed that fibre whiteness and weight loss increased with an increase in H<sub>2</sub>O<sub>2</sub> concentration, temperature, time and pH. It was further noticed by Hashmi *et al.*<sup>4</sup> that tensile strength and Young's modulus decreased with an increase in the severity of alkali treatment. It was also found by Goswami *et al.*<sup>5</sup> that with an increase in alkali concentration, the weight loss & whiteness index of linen fabric increases, while yellowness & redness indices decrease. Chatterjee *et al.*<sup>6</sup> noticed an increase in weight loss of jute fibre, with an increase in H<sub>2</sub>O<sub>2</sub> concentration and bleaching time till a certain level. Further increases in peroxide concentration and bleaching time did not result in an increase in weight loss. However, the interaction effect study between the process parameters of the flax rove bleaching process, namely H<sub>2</sub>O<sub>2</sub> concentration, NaOH concentration, bleaching time and temperature, is not explicitly reported. Also, the research findings on the optimisation of the flax rove bleaching process are uncommon. The bleaching process improves the colour of fibre and fibre's spinnability but is accompanied by weight loss<sup>7-11</sup>. For industrial operations, weight loss results in value loss. Thus, it is important to optimise the bleaching process to minimise loss, maximise whiteness index, minimise fibre damage, reduce chemical consumption and save energy. Therefore, an attempt has been made to optimise the bleaching process parameters using the design of experiments approach.

In this study, bleaching experiments are performed on flax rove by varying the process variables specifically H<sub>2</sub>O<sub>2</sub> concentration, NaOH concentration, bleaching time and temperature, in accordance with a 3<sup>4</sup> Box-Behnken design of experiments. The whiteness index and bleaching loss of the bleached rove are measured as response variables. Response surface methodology is used to analyse the experimental results, and the individual as well as interaction effects of the process factors on the whiteness index and weight loss are ascertained. A simultaneous optimisation of multiple responses is carried out using

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the desirability function approach. The experimental results are then compared with the predicted results.

### Experimental

The rove bobbins, each containing approximately 1 kg of flax rove with a thread count of 3 Nm, were supplied by Jayashree Textiles, Kolkata, India. Laboratory-grade hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 50 %) and sodium hydroxide ( $\text{NaOH}$ ) (Merck), as well as hydrochloric acid ( $\text{HCl}$ ) (Loba), were used. All auxiliary chemicals, including a non-ionic wetting agent (Esteem), antioxidants (Antox), a sequestering agent (Securon 540), and a chelating agent (Contavan Gal), were of commercial grade.

Bleaching was carried out in a single-package dyeing machine (AADARSH Technologies, India) modified to accommodate the larger flax rove bobbin, with the material-to-liquor ratio maintained at 1:14. The package holding peg was designed to ensure and maintain liquor flow from inside to outside, outside to inside, and across the full package. A schematic of the modified package dyeing machine is shown in Fig. 1. Prior to bleaching, the flax rove was scoured at 60 °C for 30 min using a non-ionic wetting agent (0.7 g/L) and a chelating agent (0.9 g/L) to facilitate the bleaching process. The control bleaching process was carried out at an  $\text{H}_2\text{O}_2$  concentration of 0.08 kg/kg of

fibre, an  $\text{NaOH}$  concentration of 0.022 kg/kg of fibre, a temperature of 95 °C, and a duration of 45 min. Following bleaching, the roving bobbin was rinsed in hot water at 80 °C for 15 min, neutralised with hydrochloric acid at 35 °C for 15 min, and finally washed with cold water for 15 min.

The whiteness index of the bleached rove was determined using an X-Rite Colour Spectrophotometer with Color iQC software, employing a D65 illuminant and a 10 °C observer angle, and expressed in Hunter values. Weight loss was measured on a dried sample weight basis by drying the samples at 105 °C for 3 h, cooling them in a desiccator, and weighing them using an electronic balance. The weight loss was determined by the following equation:

$$\text{Weight loss \%} = \frac{L_0 - L_1}{L_0} \times 100 \quad \dots (1)$$

where  $L_0$  and  $L_1$  are the oven dry weights of the raw and bleached rove, respectively.

The degree of polymerisation ( $DP$ ) of the flax fibres was measured before and after bleaching based on fluidity ( $F$ ) using cupra-ammonium hydroxide under standard conditions. The  $DP$  of the raw and bleached flax fibres were calculated by using Eq. 2 (Tatjana *et al.*<sup>12</sup>):

$$DP = 2032 \left( \log_{10} \frac{74.35 + F}{F} \right) - 573 \quad \dots (2)$$

The damage factor ( $S$ ) of the bleached rove was calculated by using Eq. 3:

$$S = \frac{1}{\log_{10} 2} \log_{10} \left( \frac{2000}{DP} - \frac{2000}{DP_0} + 1 \right) \quad \dots (3)$$

where  $DP_0$  is the  $DP$  of raw flax fibre; and  $DP$ ,  $DP$  of bleached flax fibre.

The bleaching experiments were carried out according to  $3^4$  Box-Behnken design of experiments, with response surface methodology used for analysis. The process factors investigated were  $\text{H}_2\text{O}_2$  concentration,  $\text{NaOH}$  concentration, bleaching temperature, and time at three different levels (Table 1).

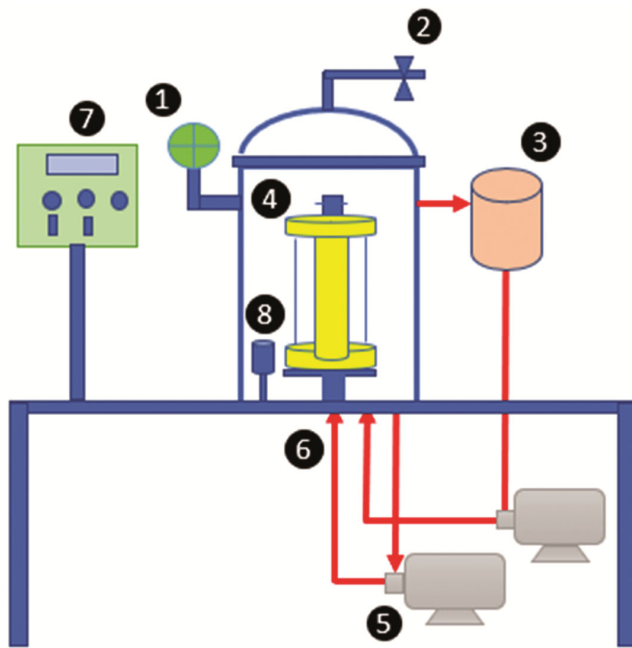


Fig. 1— Schematic diagram of modified package dyeing machine for bleaching (1- Pressure gauge, 2- Steam control valve, 3- Kitchen tank, 4-Rove bobbin, 5- Pump, 6- Liquor flow pipe, 7- Control panel and 8- Heating coil)

Table 1 — Details of process factors and their levels

Factors	Symbol	Level		
		-1	0	+1
Hydrogen peroxide, kg/kg of fibre	$Y_1$	0.06	0.08	0.1
Sodium hydroxide, kg/kg of fibre	$Y_2$	0.016	0.022	0.028
Time, min	$Y_3$	30	45	60
Temperature, °C	$Y_4$	85	95	105

The levels of variable - 1 (Low), 0 (Medium), +1 (High) were chosen based on a preliminary study and experience to ensure significant effects on both the whiteness index and weight loss. In total, 27 bleaching trials were conducted, with experimental runs 1–24 performed once and the centre point runs (25–27) performed in triplicate to estimate pure error (Table 2). The design expert software (8.0.1) was used to analyse experimental results using response surface methodology. The responses of these variables were interpreted in terms of the whiteness index and weight loss. Both individual and interaction effects of the process factors were examined, and the responses were modelled using the quadratic polynomial given in Eq. 4.

$$X_k = \beta_0 + \sum_{i=1}^{i=4} \beta_i Y_i + \sum_{i=1}^{i=4} \beta_{ii} Y_i^2 + \sum_{i<j} \beta_{ij} Y_i Y_j \dots (4)$$

where  $X_1$  and  $X_2$  denote the dependent variables — whiteness index ( $k = 1$ ) and weight loss ( $k = 2$ ), respectively;  $Y_i$  and  $Y_j$ , coded level of process factors;  $\beta_0$ , constant term; and  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  represent the regression coefficients for linear, quadratic and interaction terms, respectively. The least square method was used to estimate the regression coefficients. The coefficient of determination and residual analysis were used to evaluate model adequacy.

**Results and Discussion**

**Statistical Analysis of Effects of Process Parameters on Whiteness Index**

As described, 27 rove bleaching experiments are performed under identical environmental conditions according to 3<sup>4</sup> Box-Behnken design. The whiteness index is measured for all 27 bleached roves, with the results presented in Table 3. These experimental values are used to fit a response surface model, resulting in the following best-fitted equation for whiteness index:

$$X_1 = 72.61 + 3.06Y_1 + 2.20Y_2 + 2.08Y_3 + 2.88Y_4 + 0.6Y_1Y_2 - 1.68Y_1Y_3 + 0.11Y_1Y_4 + 0.40Y_2Y_3 - 0.23Y_2Y_4 + 0.92Y_3Y_4 - 2.54Y_1^2 - 2.01Y_2^2 - 1.01Y_3^2 - 0.93Y_4^2 \dots (5)$$

where  $X_1$  denotes the predicted whiteness index of the bleached roving sample;  $Y_1$ ,  $Y_2$ ,  $Y_3$  and  $Y_4$  represent the H<sub>2</sub>O<sub>2</sub> concentration, NaOH concentration, time and temperature, respectively.

The predicted whiteness indices based on this equation are also shown in Table 3, alongside the calculated errors, defined as the difference between experimental and predicted values. The coefficient of determination ( $R^2$ ) for the model is 0.9616, indicating a strong correlation between predicted and experimental data. Analysis of variance (ANOVA) results (Table 4) confirm that the model is statistically significant ( $P < 0.0001$ ) at the 0.05 level. However, a significant lack of fit is observed, possibly due to higher natural variation in whiteness index or deviations between predicted and actual values. The results indicate that H<sub>2</sub>O<sub>2</sub> concentration significantly affects the whiteness index, with linear, quadratic, and interactive effects with significant bleaching time ( $P < 0.05$ ). Similarly, the linear effects of NaOH concentration, bleaching time, and temperature are highly significant ( $P < 0.0001$ ).

**Response Surface Plots of Whiteness Index**

The response surface plots showing the role of bleaching process variables on the whiteness index are depicted in Fig. 2 (a)-(f). Figure 2 (a) shows that, at constant temperature (95 °C) and NaOH concentration

Table 2 — Experimental plan as per 3<sup>4</sup> Box-Behnken design

Runs	Coded level			
	H <sub>2</sub> O <sub>2</sub>	NaOH	Time	Temperature
1	-1	-1	0	0
2	+1	-1	0	0
3	-1	+1	0	0
4	+1	+1	0	0
5	-1	0	-1	0
6	+1	0	-1	0
7	-1	0	+1	0
8	+1	0	+1	0
9	0	-1	-1	0
10	0	+1	-1	0
11	0	-1	+1	0
12	0	+1	+1	0
13	0	0	-1	-1
14	0	0	+1	-1
15	0	0	-1	+1
16	0	0	+1	+1
17	-1	0	0	-1
18	+1	0	0	-1
19	-1	0	0	+1
20	+1	0	0	+1
21	0	-1	0	-1
22	0	+1	0	-1
23	0	-1	0	+1
24	0	+1	0	+1
25	0	0	0	0
26	0	0	0	0
27	0	0	0	0

Table 3 — Whiteness index of bleached rove

Run	Process factors				Whiteness index $X_I$		
	H <sub>2</sub> O <sub>2</sub> , kg/kg	NaOH, kg/kg	Time, min	Temperature, °C	Experimental	Predicted	Error
1	0.06	0.016	45	95	63.63	63.40	-0.23
2	0.10	0.016	45	95	67.48	68.32	0.84
3	0.06	0.028	45	95	66.87	66.6	-0.27
4	0.10	0.028	45	95	73.12	73.92	0.80
5	0.08	0.022	30	85	66.31	66.63	0.32
6	0.08	0.022	60	85	69.98	68.95	-0.97
7	0.08	0.022	30	105	68.95	68.55	-0.40
8	0.08	0.022	60	105	76.30	76.55	0.25
9	0.06	0.022	45	85	61.45	62.31	0.86
10	0.10	0.022	45	85	69.00	69.21	0.21
11	0.06	0.022	45	105	69.10	68.85	-0.25
12	0.10	0.022	45	105	77.07	77.19	0.12
13	0.08	0.016	30	95	65.8	65.71	-0.09
14	0.08	0.028	30	95	69.43	69.31	-0.12
15	0.08	0.016	60	95	68.98	69.07	0.09
16	0.08	0.028	60	95	74.22	74.27	0.05
17	0.06	0.022	30	95	63.35	62.24	-1.11
18	0.10	0.022	30	95	72.27	71.72	-0.55
19	0.06	0.022	60	95	69.68	69.76	0.08
20	0.10	0.022	60	95	71.89	72.52	0.63
21	0.08	0.016	45	85	65.05	64.36	-0.69
22	0.08	0.028	45	85	69.82	69.52	-0.30
23	0.08	0.016	45	105	70.46	70.58	0.12
24	0.08	0.028	45	105	74.32	74.52	0.20
25	0.08	0.022	45	95	72.72	72.61	-0.11
26	0.08	0.022	45	95	72.56	72.61	0.05
27	0.08	0.022	45	95	72.54	72.61	0.07

Table 4 — ANOVA for bleached roving whiteness index model

Source	Sum of squares	df	Mean squares	F value	P value Prob>F
Model	382.04	14	27.29	21.49	< 0.0001
$Y_1$	112.55	1	112.55	88.64	< 0.0001
$Y_2$	57.99	1	57.99	45.67	< 0.0001
$Y_3$	51.83	1	51.83	40.82	< 0.0001
$Y_4$	99.71	1	99.71	78.53	< 0.0001
$Y_1 Y_2$	1.44	1	1.44	1.13	0.3079
$Y_1 Y_3$	11.26	1	11.26	8.87	0.0115
$Y_1 Y_4$	0.044	1	0.044	0.035	0.8553
$Y_2 Y_3$	0.65	1	0.65	0.51	0.4886
$Y_2 Y_4$	0.21	1	0.21	0.16	0.6935
$Y_3 Y_4$	3.39	1	3.39	2.67	0.1284
$Y_1^2$	34.54	1	34.54	27.21	0.0002
$Y_2^2$	21.57	1	21.57	16.99	0.0014
$Y_3^2$	5.48	1	5.48	4.32	0.0599
$Y_4^2$	4.64	1	4.64	3.65	0.0802
Residual	15.24	12	1.27		
Lack of fit	15.22	10	1.52	156.34	0.0064

(0.022 kg/kg of fibre, whiteness index increases with both H<sub>2</sub>O<sub>2</sub> concentration and bleaching time, due to greater generation of per-hydroxyl (HO<sub>2</sub><sup>-</sup>) ions, enhancing lignin oxidation. The optimum whiteness index (74) of bleached rove is predicted at H<sub>2</sub>O<sub>2</sub> concentration of 0.09 kg/kg of fibre and 60 min

bleaching time. Figure 2 (b) displays the effect of temperature and time on whiteness index for a constant H<sub>2</sub>O<sub>2</sub> concentration (0.08 kg/kg of fibre) and NaOH concentration (0.022 kg/kg of fibre). The whiteness index is observed to rise with both temperature and time, with a stronger rate of increase at higher temperatures due to faster H<sub>2</sub>O<sub>2</sub> decomposition. At lower temperatures, the rate of increase in whiteness index is lower with the increase in bleaching time. Figure 2 (c) shows the effect of temperature and NaOH concentration on whiteness index for a constant H<sub>2</sub>O<sub>2</sub> concentration (0.08 kg/kg of fibre) and time (45 min). It is observed that the whiteness index increases with an increase in NaOH concentration and temperature. It is also observed that with the increase in NaOH concentration, the whiteness index increases till it reaches a maximum level, then slightly decreases. Further, it is seen that the rate of increase in whiteness index is almost linear with the increase in temperature. Figure 2 (d) indicates that, at constant temperature (95 °C) and time (45 min), whiteness index increases with both H<sub>2</sub>O<sub>2</sub> and NaOH concentration, reaching an optimum (74) at 0.095 kg/kg H<sub>2</sub>O<sub>2</sub> and 0.026 kg/kg NaOH, due to greater H<sub>2</sub>O<sub>2</sub> activation. Figure 2 (e)

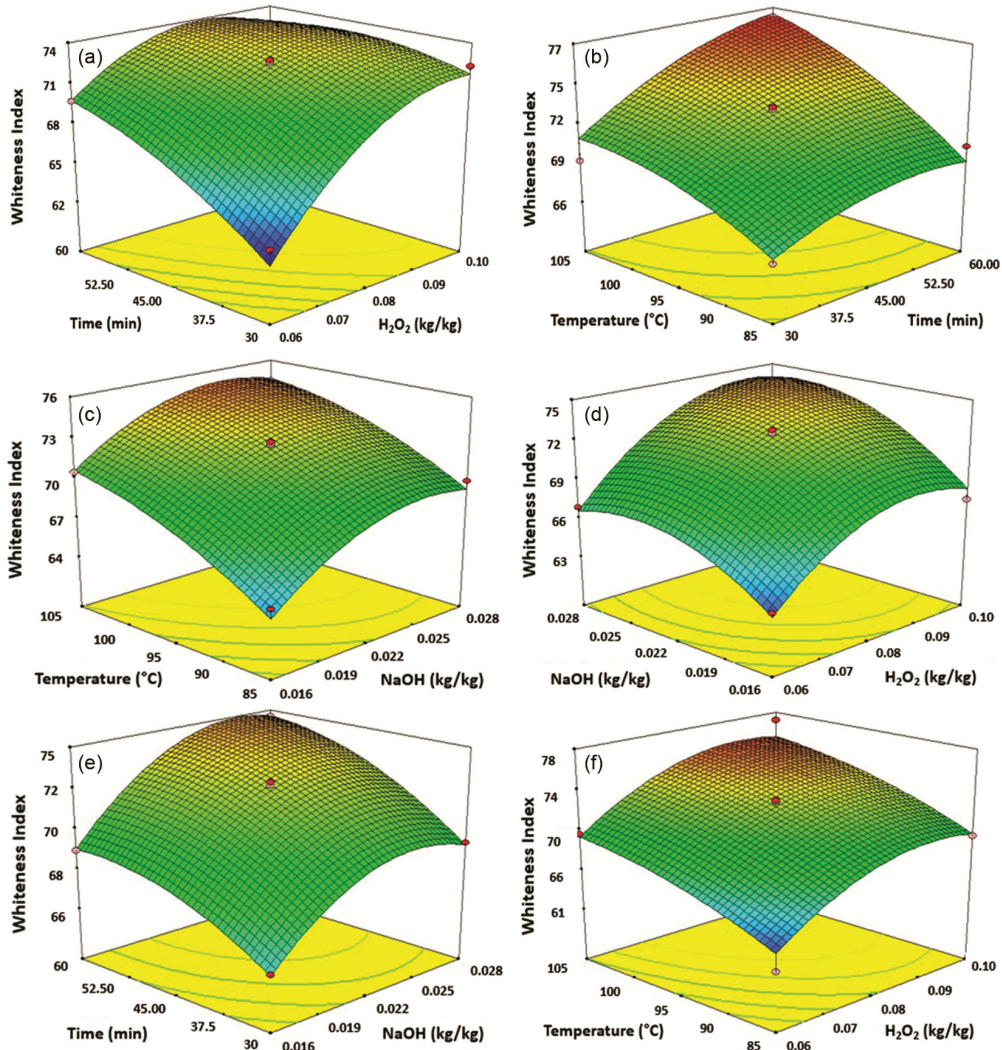


Fig. 2 — Response surface plot of whiteness index against rove bleaching process factors

shows that, at constant temperature (95 °C) and H<sub>2</sub>O<sub>2</sub> (0.08 kg/kg), higher NaOH concentration and bleaching time lead to higher whiteness, with a stronger effect at longer times. Figure 2 (f) reveals a near-linear increase in whiteness index with both temperature and H<sub>2</sub>O<sub>2</sub> concentration, reaching an optimum of 75 at 105 °C and 0.09 kg/kg H<sub>2</sub>O<sub>2</sub>.

**Statistical Analysis of Effects of Process Parameters on Weight Loss**

Weight loss for all bleached roves is presented in Table 5. The fitted response surface model for weight loss yields the following equation:

$$\begin{aligned}
 X_2 = & 10.90 + 0.027Y_1 + 1.22Y_2 + 0.26Y_3 + \\
 & 1.28Y_4 - 0.01Y_1Y_2 - 0.025Y_1Y_3 + 0.50Y_1Y_4 + \\
 & 0.15Y_2Y_3 + 0.14Y_2Y_4 + 0.24Y_3Y_4 - 0.16Y_1^2 - \\
 & 0.40Y_2^2 - 0.58Y_3^2 + 0.041Y_4^2 \dots (6)
 \end{aligned}$$

where  $Y_2$  is the predicted weight loss after bleaching.

The model shows a high correlation between predicted and experimental values ( $R^2= 0.9480$ ). ANOVA results (Table 6) confirm that the model is statistically significant ( $P < 0.0001$ ), with no significant lack of fit ( $P > 0.05$ ), indicating adequate model validity. Caustic concentration and bleaching temperature significantly affect the weight loss ( $P < 0.0001$ ). An interaction between H<sub>2</sub>O<sub>2</sub> concentration and bleaching temperature is found to be significant ( $P=0.0413$ ), while the squared effect of bleaching time approaches significance ( $P= 0.0106$ ).

**Response Surface Plot of Weight Loss**

The response surface plots depicting the role of bleaching process variables on the weight loss are

Table 5 — Weight loss of bleached rove

Run	Process factors				Weight loss $X_2$		
	H <sub>2</sub> O <sub>2</sub> , kg/kg	NaOH, kg/kg	Time, min	Temperature, °C	Experimental	Predicted	Error
1	0.06	0.016	45	95	9.13	9.08	-0.05
2	0.10	0.016	45	95	9.05	9.16	0.11
3	0.06	0.028	45	95	11.52	11.54	0.02
4	0.10	0.028	45	95	11.4	11.58	0.18
5	0.08	0.022	30	85	8.85	9.06	0.21
6	0.08	0.022	60	85	8.72	9.10	0.38
7	0.08	0.022	30	105	11.39	11.14	-0.25
8	0.08	0.022	60	105	12.23	12.14	-0.09
9	0.06	0.022	45	85	9.88	9.97	0.09
10	0.10	0.022	45	85	8.92	9.03	0.11
11	0.06	0.022	45	105	11.83	11.53	-0.30
12	0.10	0.022	45	105	12.88	12.59	-0.29
13	0.08	0.016	30	95	9.08	8.59	-0.49
14	0.08	0.028	30	95	10.68	10.73	0.05
15	0.08	0.016	60	95	9.06	8.81	-0.25
16	0.08	0.028	60	95	11.25	11.55	0.30
17	0.06	0.022	30	95	9.55	9.85	0.30
18	0.10	0.022	30	95	9.82	9.95	0.13
19	0.06	0.022	60	95	10.51	10.42	-0.09
20	0.10	0.022	60	95	10.68	10.42	-0.26
21	0.08	0.016	45	85	8.28	8.18	-0.10
22	0.08	0.028	45	85	11.07	10.34	-0.81
23	0.08	0.016	45	105	9.67	10.46	-0.89
24	0.08	0.028	45	105	13.04	13.18	0.14
25	0.08	0.022	45	95	10.89	10.90	0.01
26	0.08	0.022	45	95	11.08	10.90	-0.18
27	0.08	0.022	45	95	10.73	10.90	0.17

Table 6 — ANOVA for bleached roving weight loss model

Source	Sum of squares	df	Mean squares	F-value	p-value Prob>F
Model	42.32	14	3.02	15.62	< 0.0001
$Y_1$	0.0091	1	0.0091	0.047	0.8322
$Y_2$	17.98	1	17.98	92.93	< 0.0001
$Y_3$	0.79	1	0.79	4.09	0.0661
$Y_4$	19.56	1	19.56	101.07	< 0.0001
$Y_1 Y_2$	0.0004	1	0.0004	0.0021	0.9645
$Y_1 Y_3$	0.0025	1	0.0025	0.013	0.9114
$Y_1 Y_4$	1.01	1	1.01	5.22	0.0413
$Y_2 Y_3$	0.087	1	0.087	0.45	0.5152
$Y_2 Y_4$	0.084	1	0.084	0.43	0.5222
$Y_3 Y_4$	0.24	1	0.24	1.22	0.2918
$Y_1^2$	0.13	1	0.13	0.68	0.4245
$Y_2^2$	0.85	1	0.85	4.41	0.0575
$Y_3^2$	1.77	1	1.77	9.15	0.0106
$Y_4^2$	0.0091	1	0.0091	0.047	0.8322
Residual	2.32	12	0.19		
Lack of fit	2.26	10	0.23	7.36	0.1254

shown in Fig. 3. Figure 3 (a) displays the effect of temperature and time on weight loss for a constant H<sub>2</sub>O<sub>2</sub> (0.08 kg/kg of fibre) and NaOH (0.022 kg/kg of fibre) concentrations. The weight loss of bleached rove is observed to be higher at higher temperatures. This could be due to higher swelling of fibre at higher

temperature than at lower temperature (Eriksson *et al.* 1991). Further, it is noticed that the weight loss initially rises with time up to 50 min, then decreases, being lowest at 30 min and 85 °C. Figure 3 (b) displays the effect of temperature and NaOH concentration on weight loss for a constant H<sub>2</sub>O<sub>2</sub> and time (45 min). It is observed that the bleaching loss decreases almost linearly with a decrease in temperature and NaOH concentration. Lower NaOH concentration and lower bleaching temperature are favourable conditions for lower weight loss of bleached rove. Figure 3 (c) demonstrates that, at constant H<sub>2</sub>O<sub>2</sub> (0.08 kg/kg of fibre) and temperature (95 °C), weight loss rises with time up to 50 min, then decreases, being lowest at 30 min and NaOH concentration (0.016 kg/kg of fibre). Figure 3 (d) depicts that, at constant time (45 min) and NaOH (0.022 kg/kg of fibre), weight loss decreases linearly with temperature reduction. This could be attributed to the lower swelling of the fibre at the lower temperature than at the higher temperature. It is seen that at higher H<sub>2</sub>O<sub>2</sub> concentration, the rate of decrease in weight loss is very high with the decrease in bleaching temperature, whereas at lower H<sub>2</sub>O<sub>2</sub>

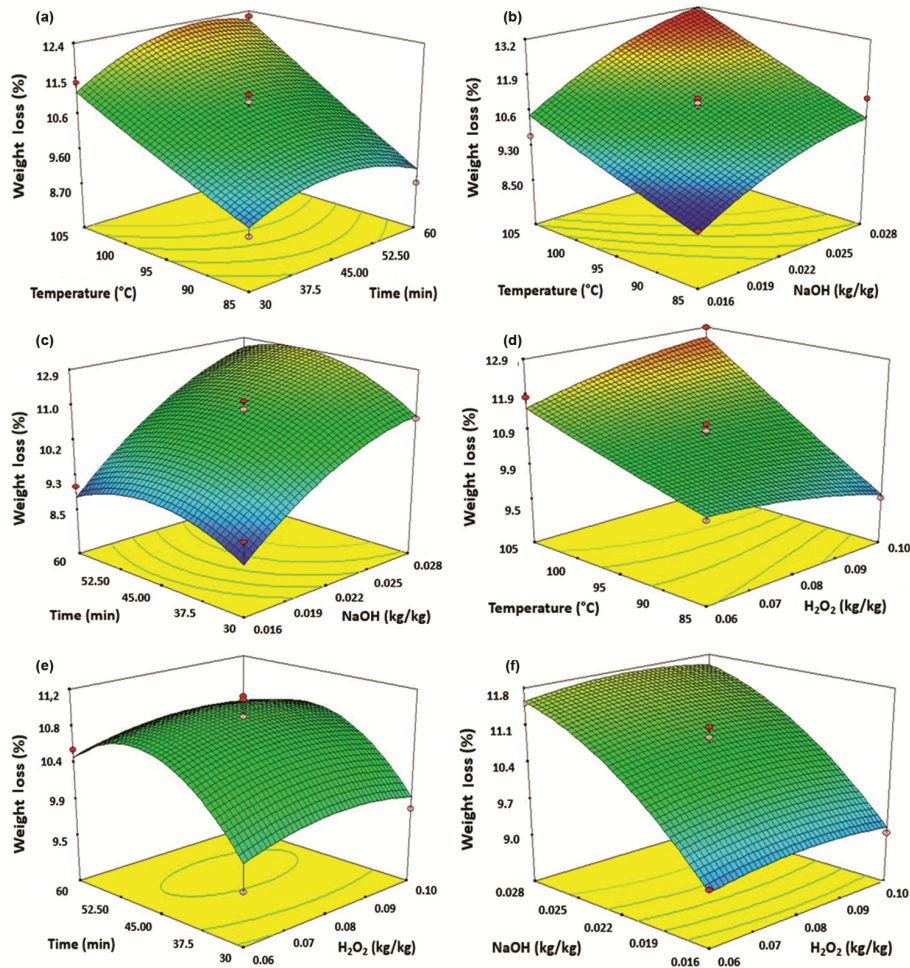


Fig. 3 — Response surface plot of weight loss against rove bleaching process factors

concentration, the rate of decrease is lower. Figure 3(e) reveals that, at constant temperature (95 °C) and NaOH (0.022 kg/kg fibre), weight loss initially rises with increases in time and H<sub>2</sub>O<sub>2</sub> concentration. Further, an increase in both factors beyond the optimum level leads to a decrease in weight loss. The bleaching loss is observed to be optimum at time 53 min and H<sub>2</sub>O<sub>2</sub> concentration (0.09 kg/kg of fibre). Figure 3 (f) illustrates that, at constant temperature (95 °C) and time (45 min), weight loss increases with NaOH concentration. This could be attributed to the dissolution of high levels of non-cellulosic impurities such as pectin, lignin and hemicellulose at higher concentrations of NaOH. H<sub>2</sub>O<sub>2</sub> concentration has no significant effect on weight loss.

### Simultaneous Optimisation of Whiteness Index and Weight Loss

The whiteness index and weight loss of bleached rove are simultaneously optimised by using the

desirability function approach. This approach was first introduced by Harrington<sup>13</sup> and developed further by Derringer and Suich<sup>14</sup>. The approach is based on the transformation of each response to a dimensionless desirability scale ( $d_i$ ) that ranges from 0 to 1, such that a completely undesirable response is represented by 0 and the most desirable response by 1. In this study, for maximisation of the whiteness index, the following desirability function is employed:

$$d_1 = \frac{X_j - X_{j,min}}{X_{j,max} - X_{j,min}} \quad \dots (7)$$

where  $d_1$  denotes individual desirability of response whiteness index;  $X_{j,min}$  &  $X_{j,max}$  denote the minimum and maximum values of whiteness index, respectively.

Whiteness index is normalised between 61.45 ( $X_{j,min}$ ) and 77.07 ( $X_{j,max}$ ). The expressions of individual desirability for whiteness index are obtained by substituting these values in Eq. 7 and substituting  $X_1$

Table 7 — DP and damage factor for raw flax fibre and bleached rove

Run	H <sub>2</sub> O <sub>2</sub>	NaOH	Time	Temp	DP	Damage factor <i>S</i>
Unbleached fibre	-	-	-	-	3013	0
6	0.08	0.022	60	85	2955	0.02
12	0.10	0.022	45	105	2663	0.12
18	0.10	0.022	30	95	2751	0.09
21	0.08	0.016	45	85	2855	0.05
25	0.08	0.022	45	95	2756	0.09

from Eq. 5. Further, for minimisation of weight loss following desirability function is chosen:

$$d_2 = \frac{X_j - X_{j,max}}{X_{j,min} - X_{j,max}} \quad \dots (8)$$

Weight loss is normalised between 8.28 ( $X_{j,min}$ ) and 13.04 ( $X_{j,max}$ ). The expression for desirability for weight loss was obtained by substituting these values and  $X_2$  from Eq. 6 in Eq. 8. Individual desirability functions are combined using the geometric mean to obtain overall desirability ( $D$ ). The expression to obtain overall desirability is shown in the Eq. 9 below:

$$D = (d_1 d_2)^{1/2} \quad \dots (9)$$

The overall desirability function also varies from 0 to 1. The optimum solution is found at H<sub>2</sub>O<sub>2</sub> concentration of 0.09 kg/kg of fibre, NaOH concentration of 0.02 kg/kg of fibre, bleaching time of 60 min and temperature of 91.3 °C, giving predicted values of whiteness index = 70.1 and weight loss = 9.09%, with  $D = 0.71$ . Experimental verification yields whiteness index = 70.4 and weight loss = 8.88%, confirming model accuracy.

### Damage Factor Analysis

Damage factors are measured for extreme bleaching conditions (Run 6, Run 12, Run 18, Run 21) and compared with a control sample (Run 25) (Table 7). All tested conditions produce damage factors within the acceptable range of 0.01–0.2, indicating no significant fibre damage.

### Conclusion

This study dealt with the optimisation of the flax rove bleaching process by using a 3<sup>4</sup> Box-Behnken design of experiment with response surface methodology of analysis. It demonstrates that hydrogen peroxide concentration, sodium hydroxide concentration, bleaching time, and temperature significantly influence both whiteness index and weight loss<sup>15-16</sup>. The whiteness index increases with higher H<sub>2</sub>O<sub>2</sub> concentration, NaOH concentration (up to an optimum), bleaching time, and temperature, while weight loss is primarily governed by NaOH concentration and temperature. Simultaneous optimisation using a desirability function identifies the optimum bleaching conditions as 0.09 kg/kg H<sub>2</sub>O<sub>2</sub>, 0.02 kg/kg NaOH, 60 min, and 91.3 °C, yielding a whiteness index of 70.4 and weight loss of 8.88% without exceeding the acceptable damage factor range. These results confirm the feasibility of achieving high whiteness with minimal fibre degradation under optimised conditions.

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