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Optimization of Process Parameters for Wet Reeled Tasar Silk Yarn

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Abstract: Tasar silk get a high acceptance from the Indian consumers due to its rustic look, eco-friendly production process, unique luster and strength in the current market scenario of Vanya Silk Sector. Wet reeled Tasar Silk yarn is currently more advantageous than Traditional practice of Dry reeled Tasar Silk yarn in terms of luster, strength. But there is a limitation of Tasar fabrics in terms of diversified products in different designs, colour combinations. Bleaching of Tasar yarn, fabrics are not bleached scientific manner thus cause an uneven dyeing. In this connection to that in this article an attempt was made to optimize the process parameters to meet the requirement of the Tasar Industry. Bleaching of Wet reeled Tasar Silk Yarn was carried out using Box and Behnken design of experiment with three levels of four independent variables Hydrogen Peroxide Concentration (g/l), Sodium Silicate Concentration (g/l), Boiling Temperature (^{0}C) and duration of boiling (minutes). The effect of independent variables on dependent variables Tenacity (gm/den), Breaking Elongation (%) and Whiteness Index were studied. From the design of experiments, it was observed that 18 cc/l Hydrogen Peroxide (H_2O_2) of 30% w/v, 5 g/l Sodium Silicate (Na_2SiO_3) at 90^{0}C with 30 minutes time duration facilitates uniform degumming cum bleaching without significant deterioration of tensile properties.

Keywords: Bleaching, Degumming, Elongation, Tenacity, Whiteness Index, Wet reeled Tasar yarn

I. INTRODUCTION

All four commercial types of silk—Mulberry, Tasar, Muga, and Eri—are only produced in India; the latter three are referred to as “Vanya silk” jointly. Tasar, also known as tussah, silk is the most significant type of wild silk. Tasar silk is becoming more and more in demand because of its strength, distinct sheen, rustic appearance, and environmentally responsible manufacturing method. Two types of Tasar silk are available in India i.e. Temperate Tasar (*Antheraea proylei*) and Tropical Tasar silk (*Antheraea mylitta*). Tropical Tasar silk is primarily produced in Jharkhand, Chhattisgarh, Orissa, and Bihar in central and eastern India. A limited amount is also produced in the states of Maharashtra, West Bengal, and Andhra Pradesh.In addition to the tropical Tasar silks, India is home to other eco races. Notable ones include the Daba, Raily, Sukhinda, AP Local, and Bhandara Local. The most widely used and readily available of them is the Daba Tasar silk variant. Tasar culture is the main stay for many a tribal community in India [1].

The objective of cocoon cooking is to swell &soften the sericin to a level such that the raw silk filament is unwound smoothly in the reeling process with minimum breaks and wastage. Cooking is done either in plain water at boiling temperature in case of mulberry silk or in the presence of chemical in case of wild silks. Duration of cooking varies depending upon driage conditions, shell compactness, variety, water quality used, season of cocoon production etc. The efficiency of the reeling process is mainly dependent on the cooking quality of the cocoons. In order to soften the sericin evenly throughout the cocoon shell and unwind the cocoon thread smoothly and without breaks, a method known as "cocoon cooking" involves making water permeate the cocoon shell from the outside to the inside, from the interior to the outside, and once more from the outside to the inside. The Tasar cocoon is compact and extremely durable. It cannot be melted by boiling in ordinary water, in contrast to the mulberry cocoon. Cooking, degumming, and reeling of Tasar cocoons are challenging due to their negative solubility of sericin, which is further compounded by the tannins and metallic salts, like calcium oxalate, that are present inside the cocoon shell. [2]

Two types of reeling method are available in the field for Tasar reeling Operations i.e. Dry Reeling Method and Wet Reeling Method. It was reported that Wet reeled Tasar Silk in more advantageous than Dry reeled Tasar Silk in terms of Strength, Cohesion, Luster etc. Tasar wet reeled yarn is found as substitute warp yarn in place of imported Korean/ Chinese Raw Silk yarn. The Wet reeled yarn/fabric is highly accepted by the stakeholders/consumers. [3]

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Even though there is a traditional demand of Tasar fabrics in its natural colour but it has limitation in terms of design and fabric variety. New generation demands 100 % Tasar fabric in diversified products in different design and colour combination therefore it become necessary to dye the Tasar yarn in different colour. Recently, dyeing of Tasar yarn and fabric is practiced by some of the manufacturers in smaller quantity but quality of processing is not that much good as they are not following proper practices for time duration – concentration of chemicals – warm washing after bleaching / degumming etc., due to which it results in uneven dyeing, shade variation and poor fastness properties. At this juncture of time, it is highly requirements of the Tasar industry to optimize the process parameters the procedure for of Tasar yarn – fabric.

II. MATERIALS AND METHODS

2.1 Materials

A total of 5000 numbers of Tasar Daba "A" graded cocoons were obtained from Raw Material Bank, Central Silk Board, Chaibasa, Jharkhand. Soda/Sodium Carbonate (Na_2CO_3), 30 % w/v Hydrogen Peroxide (H_2O_2), Sodium Silicate (Na_2SiO_3) were taken from Vanya Silk Reeling Division (VSRD), Central Silk Technological Research Institute (CSTRI), Central Silk Board (CSB)

2.2 Methods

The sequence of operations involved to produce Tasar Wet reeled yarn is shown in the Fig. 1.

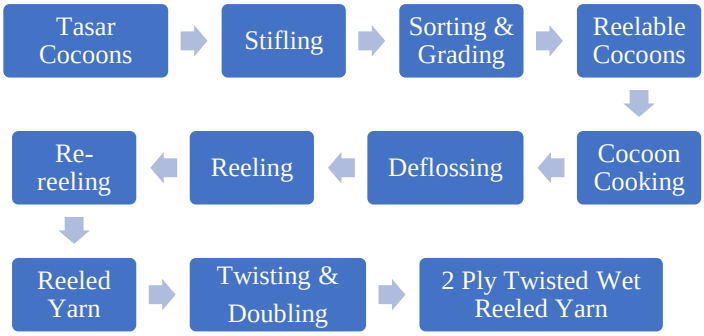

Fig 1: Sequence of Operations

2.2.1 Stifling and Sorting

The objective of stifling process is to kill the pupa to avoid the emergence as moth and also to remove moisture from the cocoon shell to store for a longer duration. Procured Tasar Daba cocoons are stifled to the optimum level in CSTRI developed Batch type electric hot air drier as per the standardized temperature profile shown in Table 1 [4]. The stifled cocoons are then sorted and separated from the defective, unreelable cocoons manually.

Table 1: Temperature & Duration of Batch Type Hot Air Drier

Temperature	Duration
115ºC	2 hours
105ºC	2 hours
90ºC	1 hour
75ºC	1 hour
60ºC	1 hour

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2.2.2 Cooking

When it comes to silk recovery and cooking efficiency, the permeation method of cooking Tasar cocoons is superior to the open pan method. [2, 5-6]. The following describes the method of cooking Tasar cocoon permeation, which takes approximately 60 minutes.

1st Step: Fifty Tasar Cocoons are placed inside the Vacuum Permeation Chamber. Tasar Cocoons receive a three-cycle permeation treatment at 300 mm Hg pressure (each cycle lasting 5-7 minutes).

2nd Step: Permeated Tasar Cocoons are then boiled in plain water for 20-25 min followed by Cooling in Two Pan Cooking Machine.

Third Step: Cooked Cocoons are then immersed in Chemical solution at 60º - 70º C and keep in soaking for overnight. The chemical solution's recipe is shown below.

Water (two ltr) for fifty Cocoons; Soda (4 g/l); Sodium Silicate (4 g/l); Hydrogen peroxide (5 cc/l).

This cooking step is repeated one after the other.

2.2.3 Deflossing, Reeling & Re- Reeling

After that, each sopped cocoon is painstakingly deflossed to determine the actual silk filament end. For continuous reeling, this genuine end makes it easier and smoother for the filament to unwind from the cocoon. The reeling has been carried out in CSTRI developed wet reeling machine [7-11]. Re-reeling is the process of moving raw silk from a tiny reel to a large reel in order to uniformly modify the skein's breadth, weight, and length.

2.2.4 Yarn Preparation

Two plies twisted wet reeled Tasar silk yarn is prepared by using Twisting and Doubling Machines for the experimental purposes. The specification of the same is given in the Table 2

Table 2: Wet Reeled Tasar Silk Yarn Specification

Particulars	Reeled Yarn	Twisted Yarn	Two Ply Twisted Yarn
Yarn Testing Details			
Denier	74	84	168
Elongation %	22	29	32
Tenacity (g/d)	2.47	2.62	2.92
Ply	Single	Single	Two
Twist		Z	S
Twist (TPM)		400	400

2.2.5 Design of Experiments

The Box and Behnken model on design of experiment for four variables and three levels was followed in order to develop the optimal bleaching recipe for Tasar yarn [12–14]. Table 3 displays the various levels as they actually occur. Table 4 provides specifics of the design matrix that was created with MINITAB 13 software. The parameters selected as independent variables are Hydrogen Peroxide (H_2O_2) Concentration (g/l), Sodium Silicate (Na_2SiO_3) Concentration (g/l), Temperature (^0C) and duration of boiling (minutes). The dependent variables are Tenacity (gm/den), Breaking Elongation (%) and Whiteness Index. A total of 27 numbers of experimental conditions are generated by Box- Behnken method. Under each condition three replicas were prepared for the testing purposes.

Table 3: Variables & their levels used in the experimental plan for bleaching of Tasar Silk

Variables	Coded Levels		
	-1	0	+1
Conc. Of H_2O_2 cc/l	6	12	18
Conc. Of Na_2SiO_3 g/l	2	4	6

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Temp in ^{0}C	75	85	95
Time Duration in Minutes	30	45	60

Table 4: Conditions of 27 experiments according to Box and Behnken response surface design along with actual values for Bleaching of Tasar Silk

Exp No	Levels				Actual Values			
	TIME	H_2O_2	Na_2SIO_3	TEMP	TIME	H_2O_2	Na_2SIO_3	TEMP
1	-1	0	0	-1	30	12	4	75
2	0	-1	+1	0	45	6	6	85
3	0	+1	-1	0	45	18	2	85
4	0	0	0	0	45	12	4	85
5	+1	0	+1	0	60	12	6	85
6	-1	0	0	+1	30	12	4	95
7	+1	0	0	+1	60	12	4	95
8	0	0	0	0	45	12	4	85
9	+1	0	-1	0	60	12	2	85
10	0	0	+1	+1	45	12	6	95
11	0	+1	0	-1	45	18	4	75
12	0	0	0	0	45	12	4	85
13	0	-1	0	+1	45	6	4	95
14	0	0	-1	-1	45	12	2	75
15	0	-1	-1	0	45	6	2	85
16	+1	+1	0	0	60	18	4	85
17	0	0	-1	+1	45	12	2	95
18	+1	-1	0	0	60	6	4	85
19	-1	0	-1	0	30	12	2	85
20	-1	-1	0	0	30	6	4	85
21	-1	+1	0	0	30	18	4	85
22	0	+1	0	+1	45	18	4	95
23	0	0	+1	-1	45	12	6	75
24	0	+1	+1	0	45	18	6	85
25	+1	0	0	-1	60	12	4	75
26	-1	0	+1	0	30	12	6	85
27	0	-1	0	-1	45	6	4	75

2.3 Testing Methods

2.3.1 Yarn Samples Conditioning

Before testing, the Tasar Wet Reeled yarn samples were stored for 24 hours at standard atmospheric conditions, which include 65 ± 2% relative humidity and 20 ± 2 °C in temperature.

2.3.2 Weight Loss %

Weight of yarns of each bleaching experiment has been measured before and after Degumming cum bleaching process. For weight measuring purpose digital electronic balance was used.
Weight loss in % = (W1-W2)/W1 X 100

Where W1 is the initial weight of conditioned Tasar Wet Reeled Yarn and W2 is the weight of bleached and degummed as well as conditioned Tasar Wet Reeled Yarn.

2.3.3 Whiteness Index (WI)

The degree to which a surface resembles the characteristics of an ideal reflecting diffuser—that is, a surface that reflects light at uniform intensities in all directions but neither transmits nor absorbs light—is known as its whiteness. The WI is measured according to AATCC Test Method 110 [15] and ASTM (American Society for Testing and Materials) E 313 standard [16]. The whiteness degree was evaluated according to the Stensby formula (Light D65, observer 10).

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WI Stensby = L – 3b + 3a (Hunter L, a, b C/2) [17]

2.3.4 Serigraph- Assessment of Yarn Tenacity & Elongation

The yarn strength testing apparatus to be utilized is a constant rate traverse pendulum type machine that is graduated in grams and has the ability to record both the breaking load and the accompanying elongation of the threads at the same time. The device must be powered in order for the movable clamp to move at a speed of 30 cm/m. All the testing done by serigraph as per the 1S 17618 (Part 5) [18]

2.3.5 Analysis of Experimental Data

Using the Box and Behnken response surface design of experiment, the experimental data were analysed for Whiteness Index (WI), Tenacity (gm/den), and Elongation (%) using MINITAB17 Statistical Software.

III. RESULTS & DISCUSSION

3.1 Observation of Weight Loss%

Sericin along with wax and colouring matter loss are the main reason for the weight deterioration of the sample after degumming cum bleaching process. Weight loss% details of 27 experimental conditions are tabulated in Table 5. Hydrogen Peroxide (H_2O_2) is the primary chemical in the bleaching approach and it plays a principal role in bleaching process. The bleaching action of hydrogen peroxide is due to the oxidation of colouring matter by per hydroxyl ion (HO_2^-). Sodium Silicate (Na_2SiO_3) acts as a stabilizing agent. Temperature and time also influence the bleaching process [19-20]. From the Fig 2 it is observed that the weight loss% is higher in Exp no 7 and lower in Exp no 14 at same concentration of H_2O_2 because of prolong bleaching action & also higher temperature provides higher rate of oxidation reaction.

Table 5: Weight loss% of 27 experimental conditions

Exp No	Avg Weight of the Yarn before bleaching (gms)	Avg Weight of the Yarn after bleaching (gms)	Weight Loss %
1	14.64	13.86	5.33
2	14.59	13.26	9.12
3	14.19	13.52	4.72
4	13.43	12.80	4.69
5	14.37	13.26	7.72
6	14.32	13.02	9.08
7	13.31	11.97	10.07
8	13.20	12.60	4.55
9	13.48	12.33	8.53
10	14.84	13.60	8.36
11	14.76	14.05	4.81
12	14.78	14.10	4.60
13	15.12	13.67	9.59
14	14.74	14.39	2.37
15	13.69	13.02	4.89
16	14.13	13.04	7.71
17	15.16	13.89	8.38
18	14.06	12.91	8.18
19	14.53	13.40	7.78
20	15.57	13.67	6.17
21	15.23	13.87	8.93
22	15.58	14.18	8.99
23	15.23	14.12	7.29
24	14.28	12.99	9.03
25	13.88	12.91	6.99
26	15.50	14.33	7.55
27	14.99	14.45	3.60

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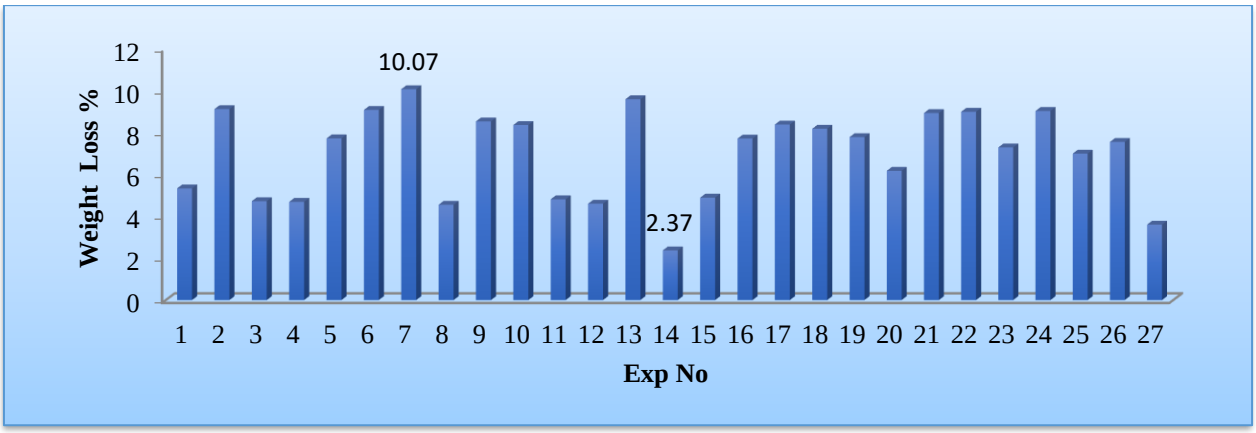

Fig 2: Weight Loss %

3.2 Effect of Independent Variables on Different Response

The degumming and bleaching of Tasar silk yarn have been done in a single bath with independent variables- Hydrogen Peroxide (H_2O_2)concentration (g/l), Sodium Silicate (Na_2SiO_3)concentration (g/l), Temperature (^0C) and duration of boiling (minutes). The effect of these independent variables on Tenacity (gm/den), Breaking Elongation (%) and Whiteness Index (WI) were studied. The values of Tenacity (gm/den), Elongation (%) and Whiteness Index of 27 experiments were tabulated and shown in the Table 6.

Table 6: Tenacity, Elongation% & Whiteness Index

Exp No	Tenacity(gm/den)	Elongation (%)	Whiteness Index WI =L-3b+3a
1	1.97	23.6	49.72
2	2.31	30.2	55.25
3	2.51	35.0	41.16
4	2.23	30.6	60.53
5	2.36	33.4	56.02
6	2.49	37.6	58.96
7	2.15	33.0	61.43
8	2.24	29.8	57.98
9	2.53	36.8	53.29
10	2.13	32.6	57.12
11	2.66	33.2	48.27
12	2.30	31.2	61.29
13	2.22	32.6	58.01
14	2.21	39.4	43.80
15	2.53	35.4	49.49
16	2.38	36.4	58.43
17	2.26	31.6	53.62
18	2.63	37.2	57.14
19	2.54	39.4	47.91
20	2.45	35.2	50.76
21	2.50	36.6	57.01
22	2.41	37.8	58.46
23	2.52	36.8	50.96
24	2.39	34.4	56.19
25	2.42	34.2	51.95
26	2.36	39.6	53.30
27	2.46	34.8	42.17

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3.2.1 Whiteness Index (WI)

The regression equation (3.1) in terms of coded factors obtained for Whiteness Index (WI) is as follows:

Whiteness Index = -313.2 + 0.739 TIME + 3.54 H_2O_2 + 15.33 Na_2SiO_3 + 6.54 TEM- 0.00449 TIME*TIME - 0.1016 H_2O_2*H_2O_2 - 1.439 NA_2SiO_3*Na_2SiO_3 - 0.03372 TEM*TEM - 0.01378 TIME*H_2O_2 - 0.0222 TIME*Na_2SiO_3 + 0.00040 TIME*TEM + 0.1967 H_2O_2*Na_2SiO_3 - 0.0129 H_2O_2*TEM - 0.0457 Na_2SiO_3*TEM (3.1)

From the regression equation (3.1) it is observed that time duration has less effect on Whiteness Index (WI) as compared to the other independent variables during degumming cum bleaching process of wet reeled Tasar yarn. This is attributed to the fact that the value of positive co-efficient of time is showing less i.e. 0.739. The equation (3.1) signifies that the value of positive co-efficient of H_2O_2 is 3.54 whereas Na_2SiO_3 and Temperature are 15.33 and 6.54 respectively. So, the concentrations of H_2O_2, concentration of Na_2SiO_3 and Temp have the main contribution on the Whiteness Index as it is concluded from the equation (3.1).

Table 7: ANOVA for Whiteness Index

Source	DF	Adj SS	Adj MS	F- Value	P-Value
Model	14	664.282	47.449	21.09	0.000 *
Linear	4	410.766	102.692	45.64	0.000 *
TIME	1	35.363	35.363	15.72	0.002 *
H_2O_2	1	13.547	13.547	6.02	0.030 *
Na_2SiO_3	1	79.877	79.877	35.50	0.000 *
TEM	1	281.979	281.979	125.34	0.000 *
Square	4	217.568	54.392	24.18	0.000
TIME*TIME	1	5.445	5.445	2.42	0.146 **
H_2O_2* H_2O_2	1	71.411	71.411	31.74	0.000 *
Na_2SiO_3* Na_2SiO_3	1	176.819	176.819	78.59	0.000 *
TEM*TEM	1	60.630	60.630	26.95	0.000 *
2-Way Interaction	6	35.948	5.991	2.66	0.070
TIME* H_2O_2	1	6.150	6.150	2.73	0.124 **
TIME* Na_2SiO_3	1	1.769	1.769	0.79	0.393 **
TIME*TEM	1	0.014	0.014	0.01	0.938 **
H_2O_2* Na_2SiO_3	1	22.278	22.278	9.90	0.008 *
H_2O_2*TEM	1	2.387	2.387	1.06	0.323 **
Na_2SiO_3*TEM	1	3.349	3.349	1.49	0.246 **
Error	12	26.997	2.250		
Lack-of-Fit	10	20.985	2.099	0.70	0.716 **
Pure Error	2	6.012	3.006		
Total	26	691.280			
Model Summary					
S	**R-sq**	**R-sq (adj)**	**R-sq (pred)**		
1.49993	96.09%	91.54%	80.56%		

* Significant at 95% confidence level ** Not Significant at 95% confidence level

In Table 7, the observation of Whiteness Index experiments and statistical analysis via ANOVA are depicted. The F value of the Model is 21.09 which signify that the model used in this experiment is significant. P-Value less than 0.0500 at 95% confidence level signifies that the model terms are significant. Hence, the values of TIME, H_2O_2, Na_2SiO_3, TEM, H_2O_2*H_2O_2, Na_2SiO_3*Na_2SiO_3, TEM*TEM are showing significance in the model terms. In case of Two- way interaction only H_2O_2*Na_2SiO_3 is significant in comparison to the other variables. The Lack of Fit F-value of 0.70 implies that it is not significant relative to the pure error. The standard deviation (S) of the distance between the data values and the fitted values is 1.499. The co-efficient of determination (R^2) of Whiteness Index is 0.9609.

From the value of R^2 it can be inferred that more than 96% of variability can be explained by the chosen variables for the variation in Whiteness Index. The difference between Adjusted R^2 and Predicted R^2 is 0.1098.

The interactive effects of Sodium Silicate and Temperature (Fig 3.a & Fig 4.a), Hydrogen Peroxide and Temperature (Fig 3.b & 4.b), Hydrogen Peroxide and Sodium Silicate (Fig 3.c & 4.c), Time and Sodium Silicate (Fig 3.d & 4.d), Time

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and Hydrogen Peroxide (Fig 3.e & Fig 4.e), Time and Temperature (Fig 3.f & Fig 4.f) on Whiteness Index (WI) of bleached Tasar wet reeled yarn are shown in the Contour Plot (Fig 3) and Response Surface Plot (Fig 4).

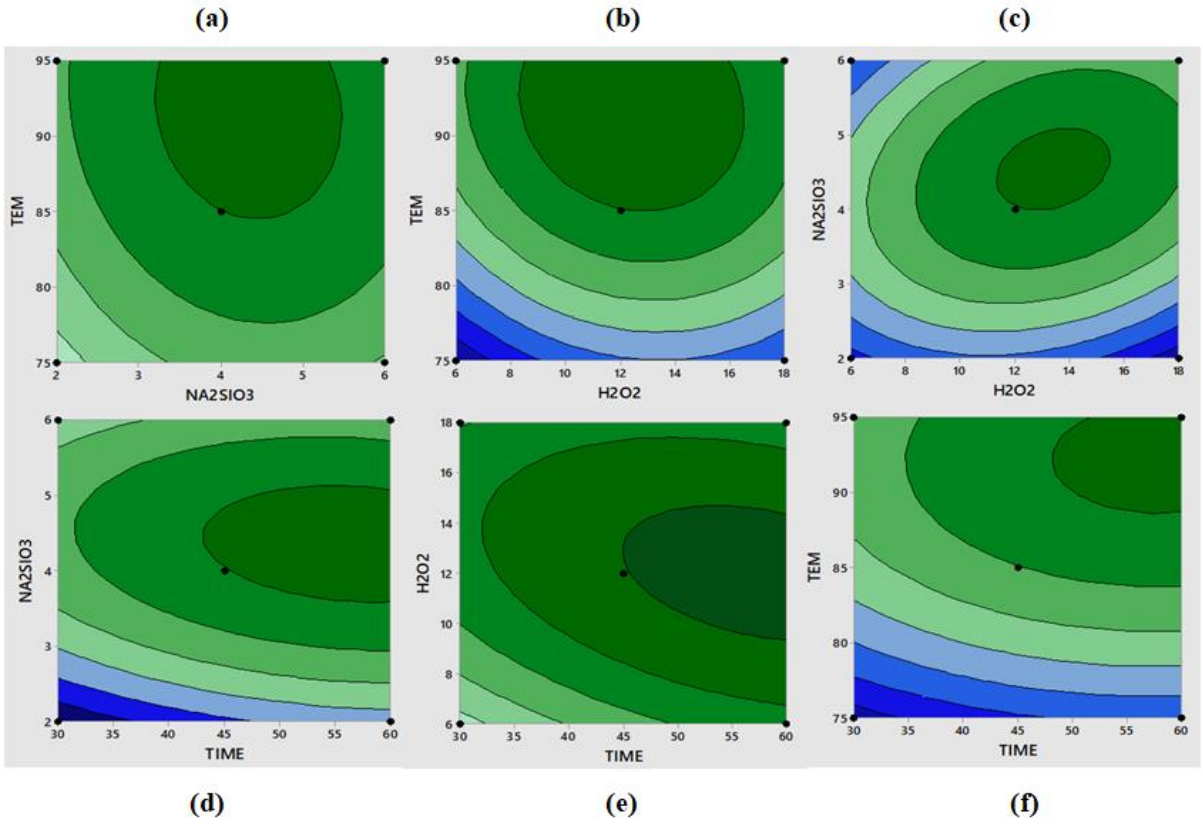

Fig 3: Contour Plot- Influence of different independent variables on Whiteness Index for Bleached Wet Reeled Tasar Silk Yarn. (a) Na_2SiO_3 Concentration (g/l) vs TEM (ºC) (b) H_2O_2 Concentration (cc/l) vs TEM (ºC) (c) H_2O_2 Concentration (cc/l) vs Na_2SiO_3 Concentration (g/l) (d) TIME (minutes) vs Na_2SiO_3 Concentration (g/l) (e) TIME (minutes) vs H_2O_2 Concentration (g/l) (f) TIME (minutes) vs TEM (ºC)

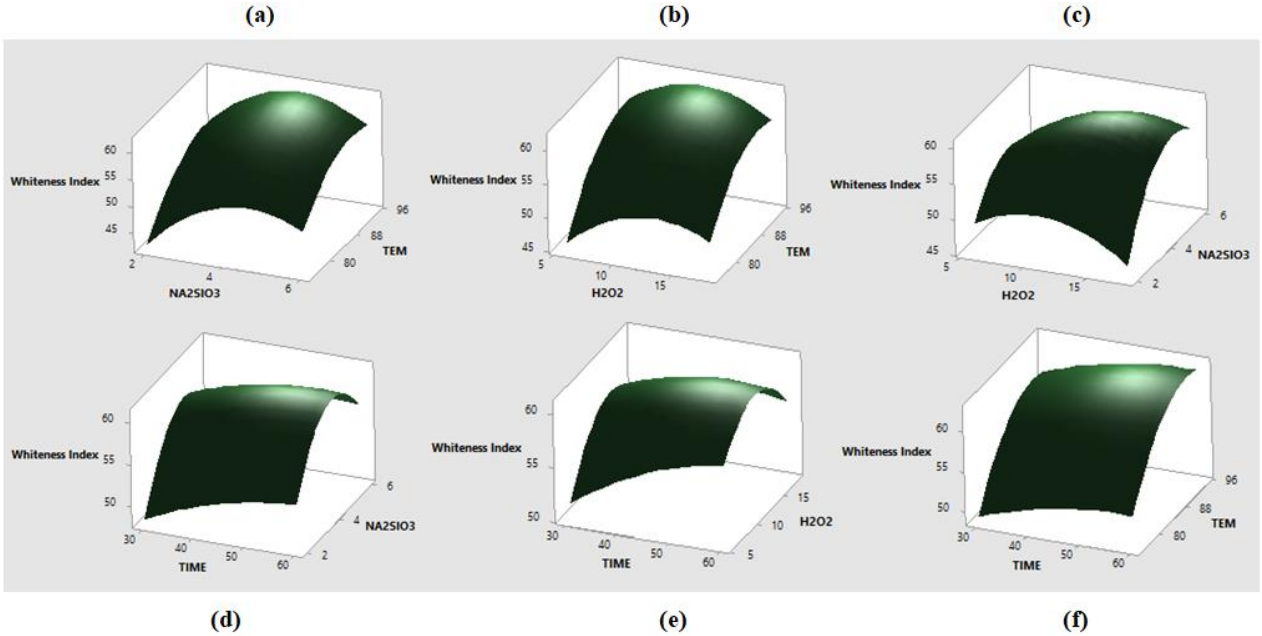

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Fig 4: Response Surface Plot- Influence of different independent variables on Whiteness Index for Bleached Wet Reeled Tasar Silk Yarn. (a) Na_2SiO_3 Concentration (g/l) vs TEM (ºC) (b) H_2O_2 Concentration (cc/l) vs TEM (ºC) (c) H_2O_2 Concentration (cc/l) vs Na_2SiO_3 Concentration (g/l) (d) TIME (minutes) vs Na_2SiO_3 Concentration (g/l) (e) TIME (minutes) vs H_2O_2 Concentration (g/l) (f) TIME (minutes) vs TEM (ºC)

In acidic medium H_2O_2 was stable but in strong alkaline medium with the presence of Sodium Carbonate (Na_2CO_3) solution, good bleaching action occurred that had a direct impact on the whiteness values of Tasar wet reeled yarn. $Na_2SiO_3$is used as a stabilizing agent to prevent the faster degradation of H_2O_2 in alkaline medium. Stabilization is the process of controlling per hydroxyl (HO_2-) ions to reduce deterioration of silk fibres and to prevent fast disintegration. The nature of Na_2SiO_3 is somewhat alkaline. During the bleaching process, Na_2SiO_3 combines to form a complex molecule containing per hydroxyl (HO_2-) ions, which are slowly released at higher temperatures.

It was observed from the Fig 3 & Fig 4 that under the condition of 85ºC, treatment time above 45 min, concentration of stabilizing agent (Na_2SiO_3) 4-5 g/l and hydrogen peroxide (H_2O_2) concentration of 12-16 cc/l give the maximum Whiteness Index (WI). With increase in the concentration of H_2O_2andincrease in temperature, the dwell time of the bleaching process reduces. As concentration of H_2O_2 increases, Whiteness Index and weight loss increases compared to the standard due to loss of sericin along with wax, colouring matter. With an increase in the temperature the rate of bleaching increases & process time reduces. The values of Whiteness Index increase from 34.20 (Raw wet reeled Tasar Silk Yarn) to 61.43 (degummed cum bleached wet reeled Tasar silk yarn) i.e. 79.6% increased which is significant.

3.2.2 Tenacity (gm/den)

The regression equation (3.2) in terms of coded factors obtained for Tenacity (gm/den) as follows:

Tenacity (gm/den) = -4.21 + 0.0592 TIME - 0.1687 H_2O_2 + 0.232 Na_2SiO_3 + 0.1367 TEM + 0.000467 TIME*TIME + 0.003631 H_2O_2*H_2O_2 + 0.01638 Na_2SiO_3*Na_2SiO_3- 0.000501 TEM*TEM - 0.000833 TIME*H_2O_2 + 0.00008 TIME*Na_2SiO_3- 0.001076 TIME*TEM + 0.00208 H_2O_2*Na_2SiO_3 + 0.001291 H_2O_2*TEM- 0.00498 Na_2SiO_3*TEM (3.2)

Table 8: ANOVA for Tenacity (gm/den)

Source	DF	Adj SS	Adj MS	F- Value	P-Value
Model	14	0.438190	0.031299	8.36	0.000 *
Linear	4	0.050068	0.012517	3.34	0.047
TIME	1	0.000020	0.000020	0.01	0.944 **
H_2O_2	1	0.000406	0.000406	0.11	0.748 **
Na_2SiO_3	1	0.047515	0.047515	12.7	0.004 *
TEM	1	0.002128	0.002128	0.57	0.465 **
Square	4	0.195259	0.048815	13.04	0.000
TIME*TIME	1	0.058823	0.058823	15.72	0.002 *
H_2O_2* H_2O_2	1	0.091118	0.091118	24.35	0.000 *
Na_2SiO_3* Na_2SiO_3	1	0.022896	0.022896	6.12	0.029 *
TEM*TEM	1	0.013396	0.013396	3.58	0.083 **
2-Way Interaction	6	0.192862	0.032144	8.59	0.001
TIME* H_2O_2	1	0.022500	0.022500	6.01	0.030 *
TIME* Na_2SiO_3	1	0.000025	0.000025	0.01	0.936 **
TIME*TEM	1	0.104103	0.104103	27.82	0.000 *
H_2O_2* Na_2SiO_3	1	0.002500	0.002500	0.67	0.430 **
H_2O_2*TEM	1	0.023994	0.023994	6.41	0.026 *
Na_2SiO_3*TEM	1	0.039740	0.039740	10.62	0.007 *
Error	12	0.044909	0.003742		
Lack-of-Fit	10	0.042042	0.004204	2.93	0.281 **
Pure Error	2	0.002867	0.001433		
Total	26	0.483098			
Model Summary					
S	**R-sq**	**R-sq (adj)**	**R-sq (pred)**		
0.061175	90.70%	79.86%	48.54%		

* Significant at 95% confidence level ** Not Significant at 95% confidence level

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From the regression equation (3.2) it is observed that time duration has less effect on Tenacity (gm/den) as compared to the other independent variables during degumming cum bleaching process of wet reeled Tasar yarn. This is attributed to the fact that the value of positive co-efficient of time is showing less i.e. 0.0592. The equation (3.2) signifies that the value of negative co-efficient of H_2O_2 is 0.1687 whereas the values of positive co-efficient of Na_2SiO_3 and Temperature are 0.232 and 0.1367 respectively. So, the concentrations of H2O2 negatively effects on the Tenacity (gm/den) as it is concluded from the Equation (3.2).

In Table 8, the observation of Tenacity (gm/den) experiments and statistical analysis via ANOVA are depicted. The F value of the Model is 8.36 which signify that the model used in this experiment is significant. P-Value less than 0.0500 at 95% confidence level signifies that the model terms are significant. Hence, the values of Na_2SiO_3, TIME*TIME, H_2O_2*H_2O_2, Na_2SiO_3*Na_2SiO_3 are showing significance in the model terms. In case of Two- way interaction only TIME*H_2O_2, TIME*TEM, H_2O_2*TEM, Na_2SiO_3*TEM are significant in comparison to the other variables.

The Lack of Fit F-value of 2.93 implies that it is not significant relative to the pure error. The standard deviation (S) of the distance between the data values and the fitted values is 0.0611. The co-efficient of determination (R^2) of Tenacity is 0.9070. From the value of R^2 it can be inferred that more than 90% of variability can be explained by the chosen variables for the variation in Tenacity. The difference between Adjusted R^2 and Predicted R^2 is 0.3132.

The interactive effects Sodium Silicate and Temperature (Fig 5.a & Fig 6.a), Hydrogen Peroxide and Temperature (Fig 5.b & 6.b), Hydrogen Peroxide and Sodium Silicate (Fig 5.c & 6.c), Time and Sodium Silicate (Fig 5.d & 6.d), Time and Hydrogen Peroxide (Fig 5.e & Fig 6.e), Time and Temperature (Fig 5.f & Fig 6.f) on Tenacity (gm/den) of bleached Tasar wet reeled yarn are shown in the Contour Plot (Fig 5) and Response Surface Plot (Fig 6).

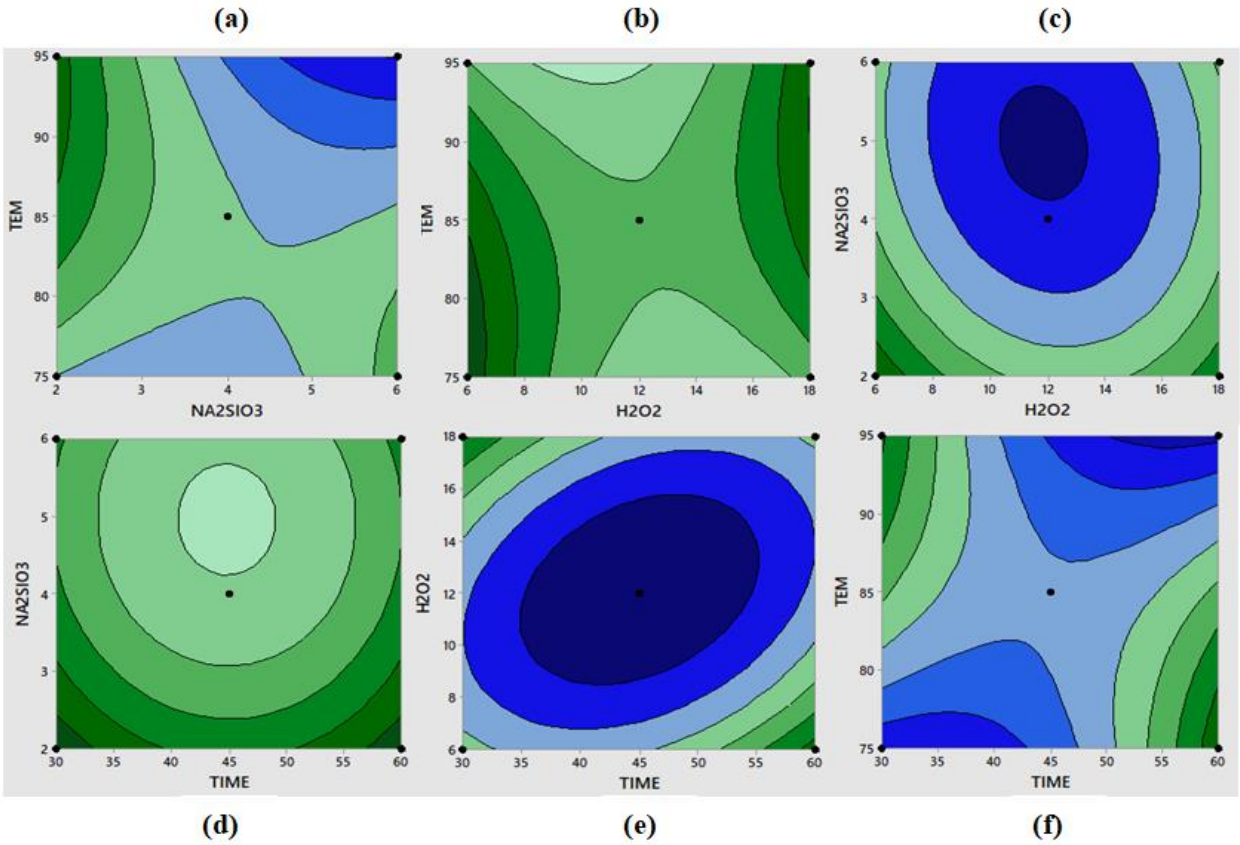

Fig 5: Contour Plot- Influence of different independent variables on Tenacity (gm/den) for Bleached Wet Reeled Tasar Silk Yarn. (a) Na_2SiO_3 Concentration (g/l) vs TEM (ºC) (b) H_2O_2 Concentration (cc/l) vs TEM (ºC) (c) H_2O_2 Concentration (cc/l) vs Na_2SiO_3 Concentration (g/l) (d) TIME (minutes) vs Na_2SiO_3 Concentration (g/l) (e) TIME (minutes) vs H_2O_2 Concentration (g/l) (f) TIME (minutes) vs TEM (ºC)

From the Fig 5 and Fig 6 it was observed that the yarn strength loss is higher, at higher concentration of H_2O_2 and Na_2SiO_3 at boiling temp, with longer duration due to the loss of sericin and the structure of Tasar silk was destroyed by degumming

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and bleaching process. The tenacity values for the raw sample (Raw Wet Tasar Silk) decreased from 2.92 gm/den to 1.97 gm/den i.e. 32.5% after bleaching process. High temperature, high dwell time damages the original structure of the wet reeled Tasar silk yarn which resulted in significant loss of tenacity.

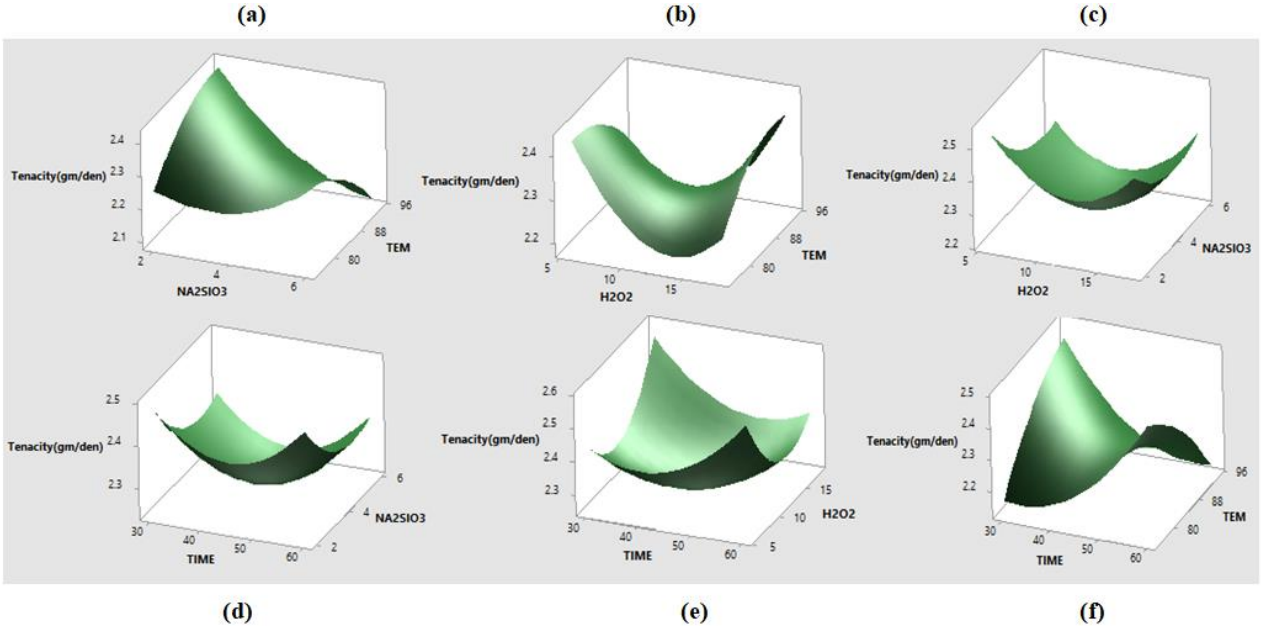

Fig 6: Response Surface Plot- Influence of different independent variables on Tenacity (gm/den) for Bleached Wet Reeled Tasar Silk Yarn. (a) Na_2SiO_3 Concentration (g/l) vs TEM (ºC) (b) H_2O_2 Concentration (cc/l) vs TEM (ºC) (c) H_2O_2 Concentration (cc/l) vs Na_2SiO_3 Concentration (g/l) (d) TIME (minutes) vs Na_2SiO_3 Concentration (g/l) (e) TIME (minutes) vs H_2O_2 Concentration (g/l) (f) TIME (minutes) vs TEM (ºC)

3.2.3 Elongation (%)

The Regression Equation (3.3) in terms of coded factors obtained for Elongation (%) as follows:

Elongation (%) = 205.7 - 0.666 TIME - 3.70 H_2O_2 - 12.66 Na_2SiO_3 - 2.58 TEM+ 0.01668 TIME*TIME + 0.0541 H_2O_2*H_2O_2 + 0.550 Na_2SiO_3*Na_2SiO_3+ 0.01302 TEM*TEM - 0.0061 TIME*H_2O_2 - 0.0300 TIME*Na_2SiO_3 - 0.00828 TIME*TEM + 0.0958 H_2O_2*Na_2SiO_3 + 0.0283 H_2O_2*TEM+ 0.0925 Na_2SiO_3*TEM (3.3)

From the regression equation (3.3) it is observed that time duration has less effect on Tenacity (gm/den) as compared to the other independent variables during degumming cum bleaching process of wet reeled Tasar yarn. This is attributed to the fact that the value of positive co-efficient of time is showing less i.e. 0.666. The equation (3.3) signifies that the value of negative co-efficient of H_2O_2 is 3.70 whereas Na_2SiO_3 and Temperature are 12.66 and 2.58 respectively so all the independent variables negatively effect on the Elongation (%) as it is concluded from the equation (3.3).

In Table 9, the observation of breaking Elongation (%) experiments and statistical analysis via ANOVA are depicted. The F value of the Model is 2.75 which signify that the model used in this experiment is significant. P-Value less than 0.0500 at 95% confidence level signifies that the model terms are significant. Hence, only the values of TIME*TIME, H_2O_2*H_2O_2, Na_2SiO_3*Na_2SiO_3 are showing significance in the model terms. In case of two- way interaction none variables are showing significance. The Lack of Fit F-value of 2.75 implies that it is not significant relative to the pure error. The standard deviation (S) of the distance between the data values and the fitted value is 2.0227. The co-efficient of determination (R^2) of Elongation is 0.7624. From the value of R^2 it can be inferred that more than 76% of variability can be explained by the chosen variables for the variation in Tenacity. The difference between Adjusted R^2 and Predicted R^2 is 0.4852.

The interactive effects of Sodium Silicate and Temperature (Fig 7.a & Fig 8.a), Hydrogen Peroxide and Temperature (Fig 7.b & 8.b), Hydrogen Peroxide and Sodium Silicate (Fig 7.c & 8.c), Time and Sodium Silicate (Fig 7.d & 8.d), Time and Hydrogen Peroxide (Fig 7.e & Fig 8.e), Time and Temperature (Fig 7.f & Fig 8.f) on Elongation (%) of bleached Tasar wet reeled yarn are shown in the Contour Plot (Fig 7) and Response Surface Plot (Fig 8).

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Table 9: ANOVA for Elongation (%)

Source	DF	Adj SS	Adj MS	F- Value	P-Value
Model	14	157.522	11.2516	2.75	0.043 *
Linear	4	33.992	8.4980	2.08	0.147
TIME	1	10.509	10.5094	2.57	0.135 **
H_2O_2	1	5.333	5.3333	1.30	0.276 **
Na_2SiO_3	1	17.280	17.2800	4.22	0.062 **
TEM	1	0.869	0.8694	0.21	0.653 **
Square	4	82.364	20.5911	5.03	0.013
TIME*TIME	1	75.100	75.1000	18.36	0.001 *
H_2O_2* H_2O_2	1	20.254	20.2540	4.95	0.046 *
Na_2SiO_3* Na_2SiO_3	1	25.784	25.7840	6.30	0.027 *
TEM*TEM	1	9.048	9.0480	2.21	0.163 **
2-Way Interaction	6	41.165	6.8609	1.68	0.210
TIME* H_2O_2	1	1.210	1.2100	0.30	0.597 **
TIME* Na_2SiO_3	1	3.240	3.2400	0.79	0.391 **
TIME*TEM	1	6.175	6.1752	1.51	0.243 **
H_2O_2* Na_2SiO_3	1	5.290	5.2900	1.29	0.278 **
H_2O_2*TEM	1	11.560	11.5600	2.83	0.119 **
Na_2SiO_3*TEM	1	13.690	13.6900	3.35	0.092 **
Error	12	49.096	4.0914		
Lack-of-Fit	10	48.110	4.8110	9.75	0.097 **
Pure Error	2	0.987	0.4933		
Total	26	206.618			
Model Summary					
S	**R-sq**	**R-sq (adj)**	**R-sq (pred)**		
2.02271	76.24%	48.52%	0.00%		

* Significant at 95% confidence level ** Not Significant at 95% confidence level

It is known that Tasar silk has a higher elongation at break than Mulberry Silk. The incapacity of molecular chains to assemble tightly during fibre growth is linked to the absence of orientation and organization. The compact packing of chains in fibroin can be influenced by the amino acid makeup and sequencing. It has been shown that a larger proportion of bulky side group amino acids, such as alanine, are present in Tasar silk. The molecular segments in the amorphous regions will mobilize easily if they contain more bulky side groups. During bleaching and degumming process, the orientation of the chain molecules gets deteriorated resulting in loss of ability to return to its original shape. From the Fig 7 and Fig 8 it was observed that at higher concentration of H_2O_2 and Na_2SO3 at boiling temp with longer duration shows the maximum breaking elongation (%). The values of breaking elongation (%) of bleached wet reeled Tasar yarn increased 39.6 % from the standard sample 32%.

3.3 Optimization of Bleaching Process

Under the optimum values of the independent variables as shown in the Table 10, the best desirability of 0.933650 is achieved. The optimization curves of degumming cum bleaching experiments are shown in the Fig 9. From the different experimental trials, it was observed that single bath degumming cum bleaching process of wet reeled Tasar silk yarn using 18 cc/l H_2O_2 concentration and 5 g/l Na_2SiO_3 concentration with duration of 30 mins at 90 ºC facilitates uniform degumming plus bleaching. The whiteness Index is observed to be around 58.15 without significant deterioration of tensile strength characteristics.

Table 10: Variable Ranges and Solution for the Desirability

Composite Solution	TIME	H_2O_2	Na_2SiO_3	TEM	Whiteness Index	Elongation (%)	Tenacity (gm/den)	Desirability
1	30	18	5	90	58.1484	41.4866	2.63175	0.933650

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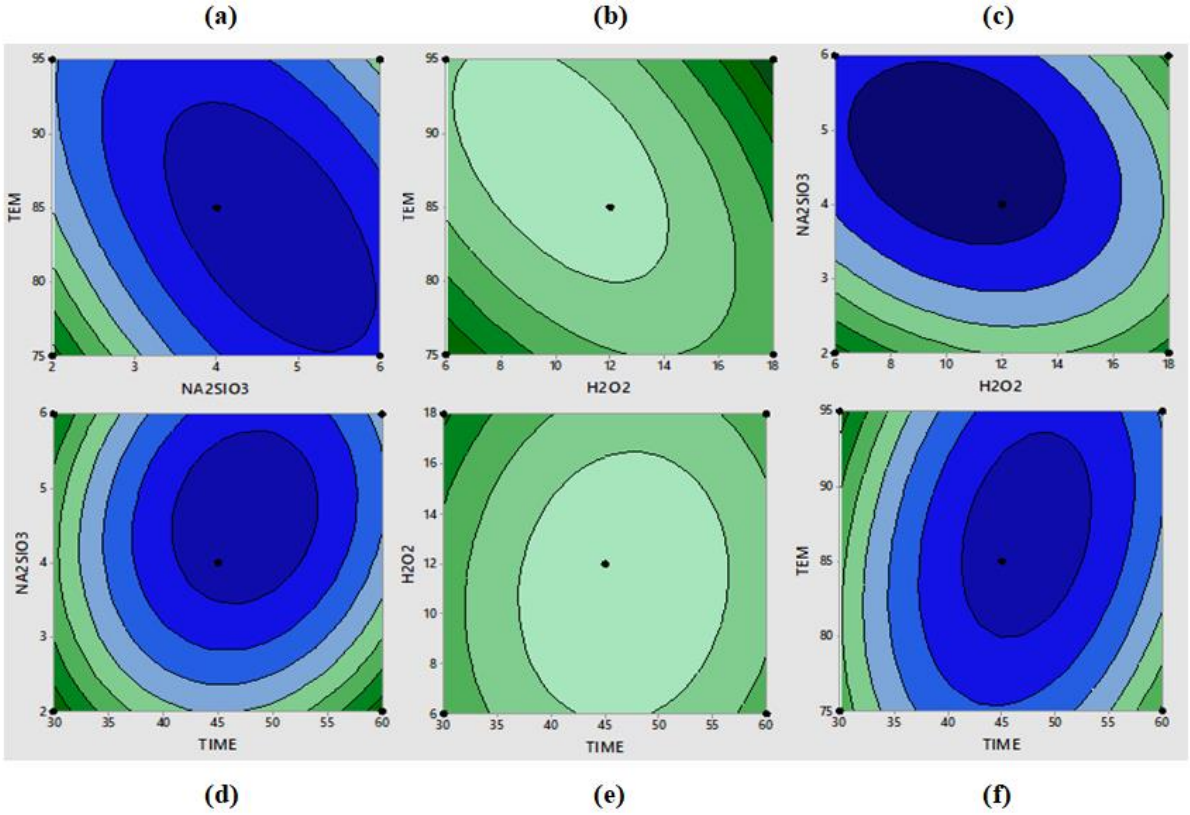

Fig 7: Contour Plot- Influence of different independent variables on Elongation (%) for Bleached Wet Reeled Tasar Silk Yarn. (a) Na_2SiO_3 Concentration (g/l) vs TEM (ºC) (b) H_2O_2 Concentration (cc/l) vs TEM (ºC) (c) H_2O_2 Concentration (cc/l) vs Na_2SiO_3 Concentration (g/l) (d) TIME (minutes) vs Na_2SiO_3 Concentration (g/l) (e) TIME (minutes) vs H_2O_2 Concentration (g/l) (f) TIME (minutes) vs TEM (ºC)

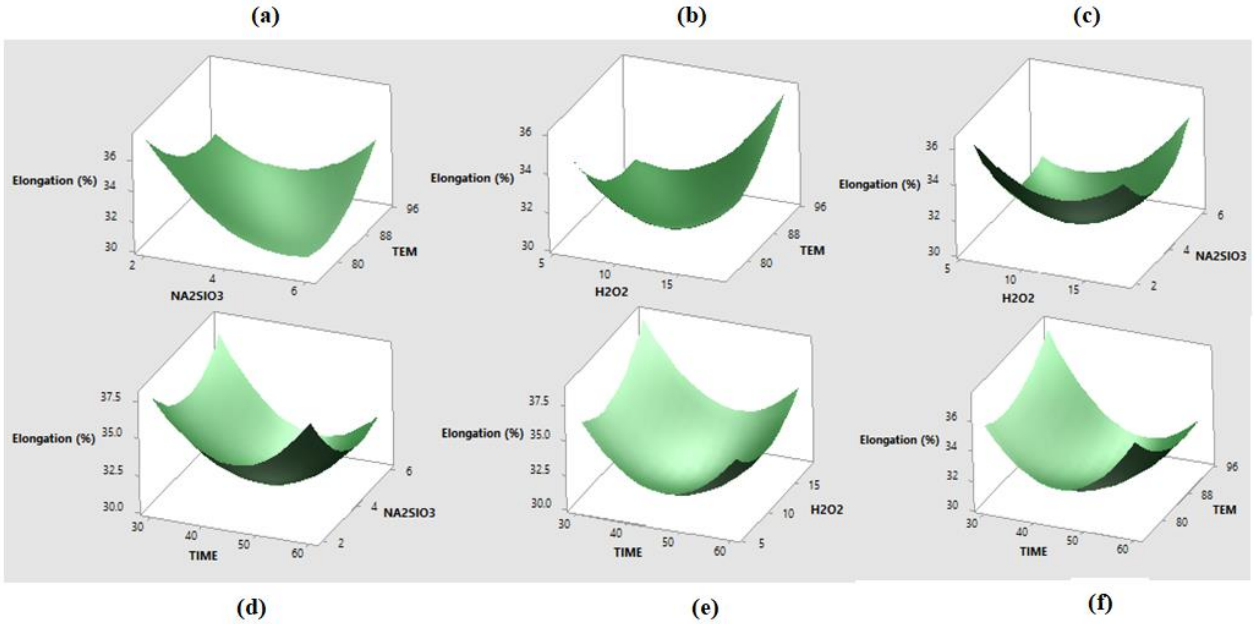

Fig 8: Response Surface Plot- Influence of different independent variables on Elongation (%) for Bleached Wet Reeled Tasar Silk Yarn. (a) Na_2SiO_3 Concentration (g/l) vs TEM (ºC) (b) H_2O_2 Concentration (cc/l) vs TEM (ºC) (c) H_2O_2

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Concentration (cc/l) vs Na_2SiO_3 Concentration (g/l) (d) TIME (minutes) vs Na_2SiO_3 Concentration (g/l) (e) TIME (minutes) vs H_2O_2 Concentration (g/l) (f) TIME (minutes) vs TEM (°C)

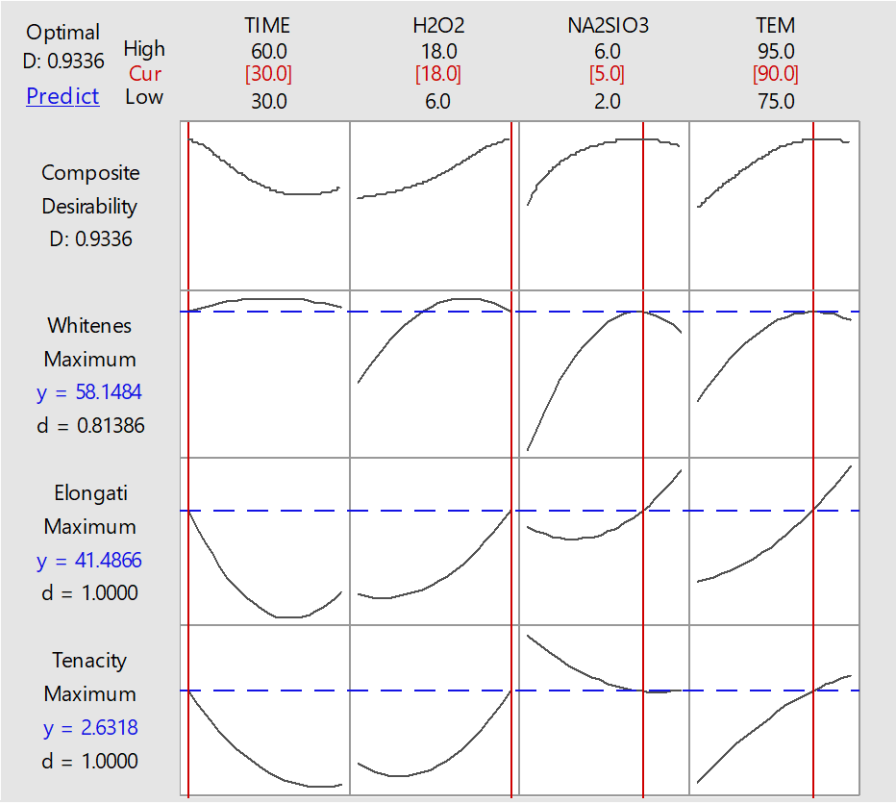

Fig 9: Optimization curves of Degumming cum Bleaching Experiments

IV. CONCLUSION

It is concluded from the different experimental conditions that the single bath degumming cum bleaching of wet reeled Tasar silk yarn using 18 cc/l Hydrogen Peroxide (H_2O_2) of 30% w/v, 5 g/l Sodium Silicate (Na_2SiO_3) at 90^0C with 30 minutes time duration facilitates uniform degumming plus bleaching. The weight loss is observed about 8.90% for wet reeled Tasar silk yarn by using the optimized degumming cum bleaching recipe without significant deterioration in tensile properties. The actual value of Whiteness Index shows 58.28 which is significant.

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Under the approved research project CFC 07025 SI, "Studies on dyeing of Tasar silk yarn, fabric, and Tasar blended fabric," the experimental study described in this paper is being conducted. The research was funded by the Central Silk Board, Ministry of Textiles, Government of India, for which the authors are extremely grateful. They also thank the director of the Central Silk Technological Research Institute, Central Silk Board, for his gracious support of the project.

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