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Sodium Lignosulfonate: an Industrial Bio-Waste for the Colouration and UV Protective Finish of Nylon Fabric

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Abstract
Sodium lignosulfonate (NLS), a biomass waste procured from paper and pulp industries as brown colour water soluble powder, is explored in this article as dye for nylon 6 fabrics in aqueous media by the exhaustion method. The fastness and functional properties of the dyed nylon 6 fabrics were studied. The dyeing process parameters were optimised using Box–Behnken response surface design and the ANOVA technique. The NLS treated dyed nylon fabric exhibited an excellent ultraviolet protection factor (UPF), as high as 62.13. The NLS dyed nylon fabrics were characterised using FTIR, FESEM, EDX and instruments. The ultraviolet protection factor (UPF) and colour strength (K/S) values are significantly increased with an increase in the concentration of NLS without any loss of tensile properties and thermal stability. Moreover, NLS treatment has excellent wash fastness. This dyeing process for synthetic fibre like nylon can be a cleaner and more eco-friendly approach by utilising waste biomass of paper from the pulp industries.

Key words: *sodium lignosulfonate, lignin, ultraviolet protection, nylon-6, protective clothing, eco-friendly dyeing.*

Introduction

The utilization of agro-waste and agro-residue for the modification of textile fibers, fertilisers, building materials, polymers, etc. is being adopted throughout the world to reduce carbon footprints and the dependency on precious virgin resources. Since agro-residues are available in huge quantities at a low price, there is ample scope in this area, and many research attempts are being made to derive novel sources and value-added products [1]. The extraction of natural dyes from various agro-processing residues viz., groundnut testa, chickpea husk, pomegranate rind, walnut husk, green coconut shell, etc. have been reported [1]. Lignin, which is a constituent material in most cellulosic fibre and wood, is isolated as biomass waste (black liquor) by using sulfuric acid 25% and sodium hydroxide solutions (2N) during the manufacturing of pulp in paper mills [2]. The lignin obtained was reacted with sodium bisulfite to get sodium lignosulfonate (NLS). Sodium lignosulphonate is a brown colour powder with a molecular mass of 534 g/mol. It has a wide range of industrial applications *viz*., pesticides, surfactants, additives in oil drilling, metal adsorbents, stabilisers in colloidal suspensions, plasticisers, and dispersant [3-5]. Qana et. al. studied the use of lignin as a raw material/reactive component in a UV curable system and also the effect of the chemical modification of lignin on the film properties produced by UV radiation [6]. The existence of an abundance of the aromatic skeleton and hydrogen bonds helps NLS to absorb ultraviolet radiation [7-8]. Due to the presence of the sulfonated group, lignosulfonates are anionically charged and water-soluble [9]. As water-soluble colorant material, NLS has not been explored for colouring textile fibres, which may provide both aesthetic appeal and UV protection.

Nylon 6 is one of the most useful synthetic fibres, with applications produced in the form of filament and staple fiber. It has good strength, heat, and chemical resistance. However, it cannot be dyed with any water soluble natural dye by the exhaustion method, and its existing dyeing process requires a harsh environment of high temperature and hazardous chemicals. Moreover, the dyeing processes are not eco-friendly. In the search for the eco-friendly dyeing of nylon with natural dye in aqueous media, NLS treatment may be a useful approach.

Moreover, nylon cannot provide protection from UV radiation. Therefore, suitable UV absorbers are used to improve the UPF of nylon fabrics. Saravanan reported that the application of 1.5% TiO_2 on microfine nylon fabrics provides good resistance against UV radiation capable of giving UPF > 50 [10]. The studies until now have been conducted on the modification of textile fibres including nylon with various inorganic substances, dye molecules, and pigments. There is a lack of studies available in the litera-

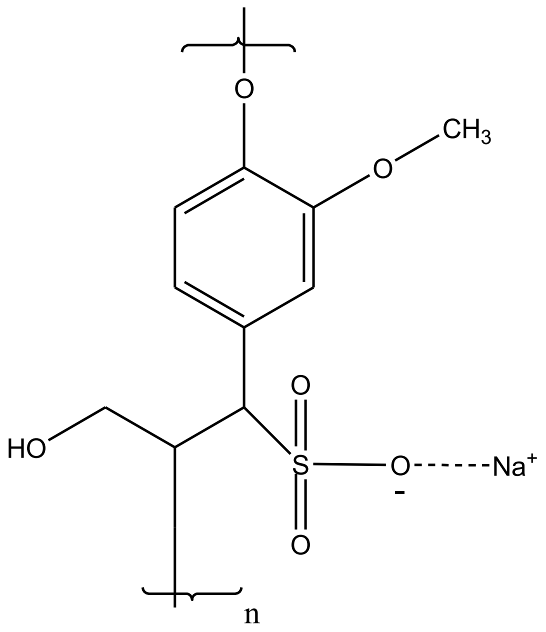

Figure 1. Chemical structure of sodium lignosulphonate (NLS).

Table 1. Process parameters and their levels.

Name of parameter	Label	Low level (-1)	Medium level (0)	High level (+1)
NLS, %	A	10	15	20
Temperature	B	60	75	90
pH	C	5.5	7	8.5

ture on the modification of nylon fabrics with organic waste material like sodium lignosulfonate (NLS).

In this study, NLS solutions of various concentrations were prepared and treatment on nylon 6 fabrics done by the exhaustion method. Process parameters of the treatment *i.e*. concentration of NLS, temperature, and pH of NLS solution were optimised using Box–Behnken response surface design. The dyed fabrics were characterised by the FTIR, FESEM, EDX, and thermo-gravimetric analysis (TGA) techniques. The NLS dyed nylon fabric samples were evaluated for UPF, K/S, tensile properties and luster in detail.

Experimental

Materials

Nylon filament with a linear density of 149 denier was procured from M/s JCT Ltd. Hoshiarpur, Punjab, India. Sodium lignosulphonate (NLS) powder was obtained from M/s Lignotech, Umkomass, South Africa. NLS is a brown colour by-product obtained from pulp and paper mills. During pulping, the lignin of the cellulosic biomass is dissolved in a high alkali solution (black liquor) and precipitated later as NLS, which has a molecular mass of 534 g/mol. The chemical structure of NLS is shown in ***Figure 1***. It has functional –OH groups which are supposed to be responsible for the formation of intermolecular hydrogen bonds with nylon fibre.

Preparation of nylon fabric and treatment with NLS

Nylon filament was woven into a fabric of 90.8 g/m^2 areal density with 26 threads per cm in the warp and 31 threads per cm in the weft direction using a handloom. The fabric was scoured with 5 g/L non-ionic detergent for 30 min at 60 °C before NLS treatment. Various concentrations of NLS solution (10, 15, and 20%) were prepared in an aqueous medium, and the nylon fabric was dyed in an infrared heating chamber at an elevated

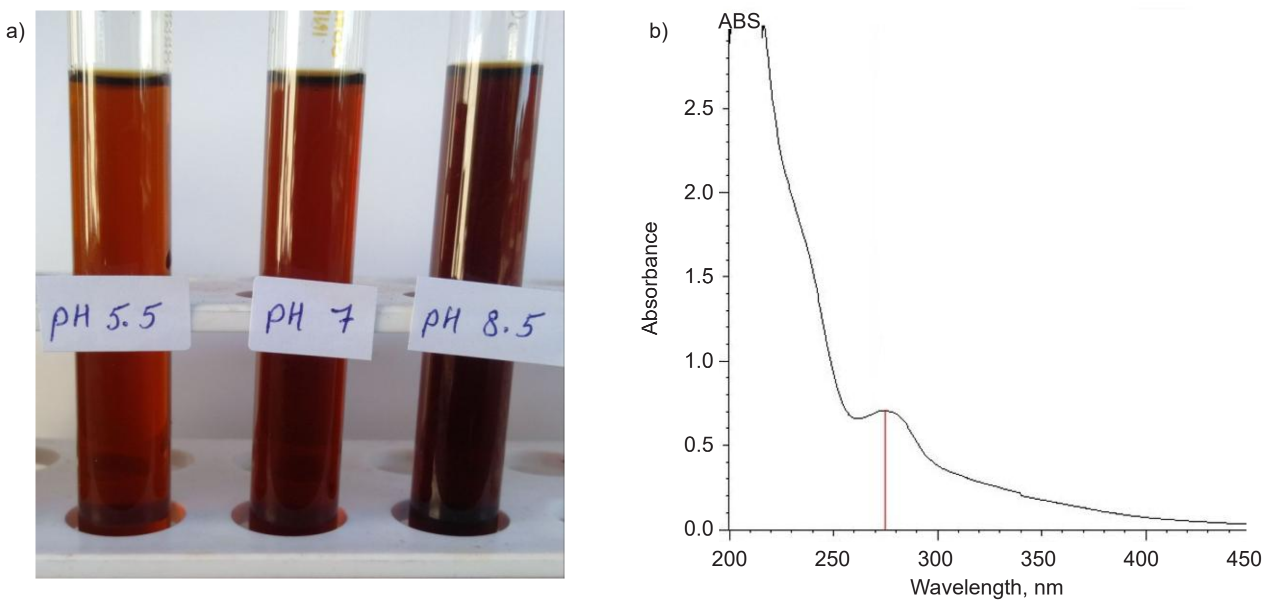

Figure 2. a) Colour of NLS solution at different pH (left hand side) and b) UV – vis spectra of NLS solution (right hand side).

temperature and predetermined pH for 1 hour. The pH of the solution was maintained by adding acetic acid. No extra chemicals or salts were used during the dyeing process. Therefore, the dyeing process is eco-friendly, non-hazardous and driven by natural exhaustion.

Design of experiment

The effects of the three main parameters: NLS concentrations, treatment temperature, and pH on the colour strength of the dyed nylon fabrics were investigated by employing a 3^3 Box–Behnken response surface design of the experiment and the ANOVA technique. The levels of these parameters are shown in ***Table 1***, selected based on a preliminary experiment, where satisfactory results were obtained. The coating of NLS over the textile surface depends on the temperature and pH of the solution. Depending on the pH of the solution, the colour of NLS varies, which was evidenced by UV-VIS spectroscopy analysis. The colour of the NLS dyed sample may ultimately be affected by the pH of the solution in which it is dyed. The difference in the colour of NLS at different pH is shown ***Figure 2.a***. It is observed from the figure that the colour of NLS is sensitive to a change in pH. The colour of the NLS solution is pale brown in the slightly acidic and neutral pH, while it turned dark brown in the alkaline pH. The main cause of the dark brown colour of lignin is the groups of quinonoid structures [11]. As mentioned earlier, the catechol structure in NSL is easy to oxidise to obtain quinonoid structures, which render the lignin strongly coloured [12]. ***Figure 2.b*** shows the UV vis spectra of NLS solution. The solution shows absorbency at 280 nm. The peak is due to the content of guaiacyl compound. Similar observations were reported by Yang et al.[12].

Experimental design software (Design-Expert version 6.0.8) was used for the design of test runs and the statistical evaluation of responses.

Characterisation

FTIR spectra of the untreated and NLS dyed nylon samples were recorded using a Bruker Alfa FTIR at a scan rate of 4 cm^{-1} in the wave range of 4000 to 500 cm^{-1}. The surface topography of the control and NLS dyed fabric samples was investigated by a Sigma field emission scanning electron microscope – Carl Zeiss 300. Gold coating of the samples was done using a sputter coating set-up.

Table 2. *Responses of process parameters to K/S value.*

Sample	NLS, %	Temp, °C	pH	K/S
	A	B	C	
1	15	75	7.0	2.42
2	15	75	7.0	2.33
3	20	60	7.0	2.95
4	20	75	5.5	4.81
5	15	60	5.5	1.52
6	10	90	7.0	0.91
7	15	90	8.5	1.17
8	10	75	8.5	0.83
9	20	90	7.0	1.63
10	10	75	5.5	1.1
11	15	60	8.5	1.58
12	20	75	8.5	1.74
13	10	60	7.0	1.65
14	15	75	7.0	2.52
15	15	75	7.0	2.75
16	15	90	5.5	1.77
17	15	75	7.0	2.48

An energy dispersive X-ray (EDX) study of the control and NLS dyed fabric samples was performed on the same FESEM instrument with an EDX attachment. The thermal stability of the NLS coated nylon fabrics was investigated by means of a Perkin Elmer 4000 TGA instrument in a temperature range of 40 °C to 400 °C, at a heating rate of 10 °C per min.

Measurement of ultraviolet protection factor (UPF), colour strength (K/S), luster and tensile properties.

The UPF of the fabric samples was estimated with a Gester GT-C30 UPF tester according to AS/NZS4399 standards within a radiation range of 290-400 nm. Ten samples of 2.5 x 2.5 cm were prepared and ten observations made for each sample. The colour values of the NLS dyed samples were evaluated with the aid of a computer color matching system (Konica Minolta, model no-4808), using JAY pack software. A gloss meter (CHN Spec technology, Hangzhou model no: CS300S) was used to analyse the lustrous appearance of the fabric samples by estimating the amount of light reflected at an angle of 60°. The tensile strength and elongation of untreated and NLS treated fabric samples were measured by an Instron 4200 tester following the ASTM standard.

Analysis of fastness properties

A Q-Sun weather-o-meter was used to evaluate the light fastness property of the fabric samples as per ISO 105-B02. The fabric samples were exposed for 10 h while keeping an irradiance of 1.10 W/m^2, a wavelength of 420 nm, and temperature of the chamber of 50 °C. The exposed portion of the test specimen was compared with the masked portion for any colour change using the Grey scale for colour change and then graded accordingly on a 5 point scale. The washing fastness and colour staining were as per ISO 105:C06. The experimental samples were washed in a solution of 4 g of ECE detergent and 1g of sodium perborate mix with 1 litre of water at 50 °C for 40 minutes duration.

Results and discussion

Investigation of the effects of process parameters of NLS treatment on nylon fabric for colour strength (K/S)

Response surface methodology was used to establish an empirical relationship between the response variables i.e. K/S value and a set of experimental factors. Process parameters such as the concentration, temperature, and pH of NLS solutions were optimised using 3^3 Box–Behnken response surface design. The concentration of the NLS solution was kept at 10% (-1), 15% (0) and 20% (+1) levels, the temperature of the NLS solution at 60 °C (-1), 75 °C (0) and 90 °C (+1), and the pH of the NLS solution at 5.5 (-1), 7.0 (0) and 8.5 (+1), respectively. According to this design, a total of 17 samples were prepared. K/S values of all 17 fabric samples were measured and are reported in ***Table 2***.

Effect of the concentration of NLS solution and temperature on K/S

The effect of the concentration and temperature of the NLS solution on the fabric's K/S is shown in ***Figure 3***. The con-

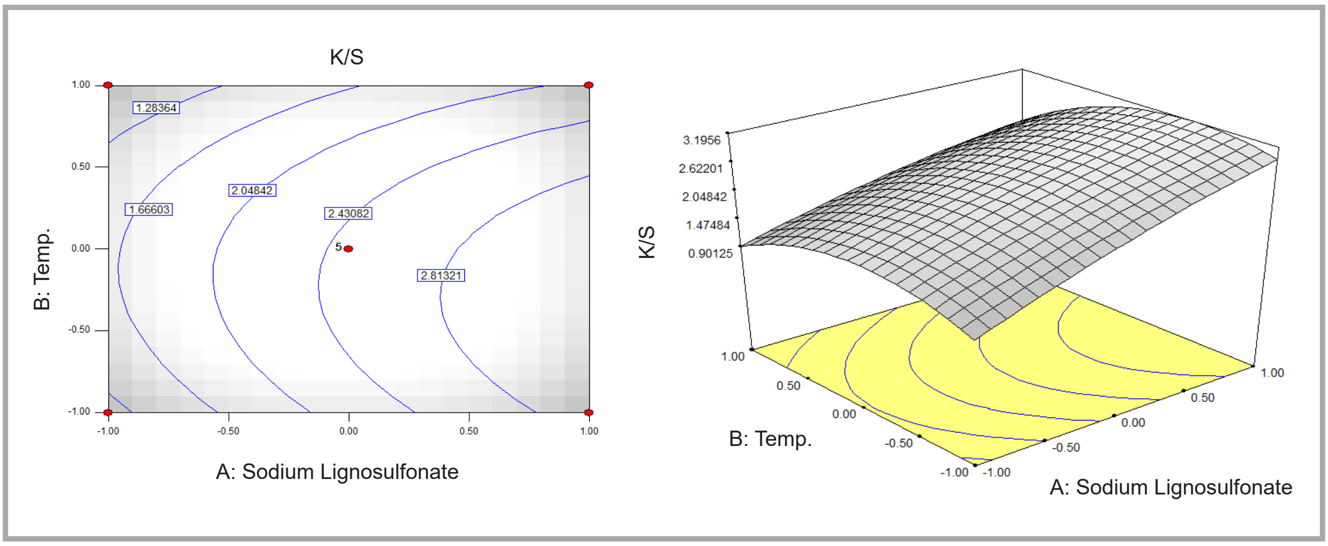

Figure 3. Effect of the temperature and concentration of NLS on the K/S value of NLS dyed nylon fabric.

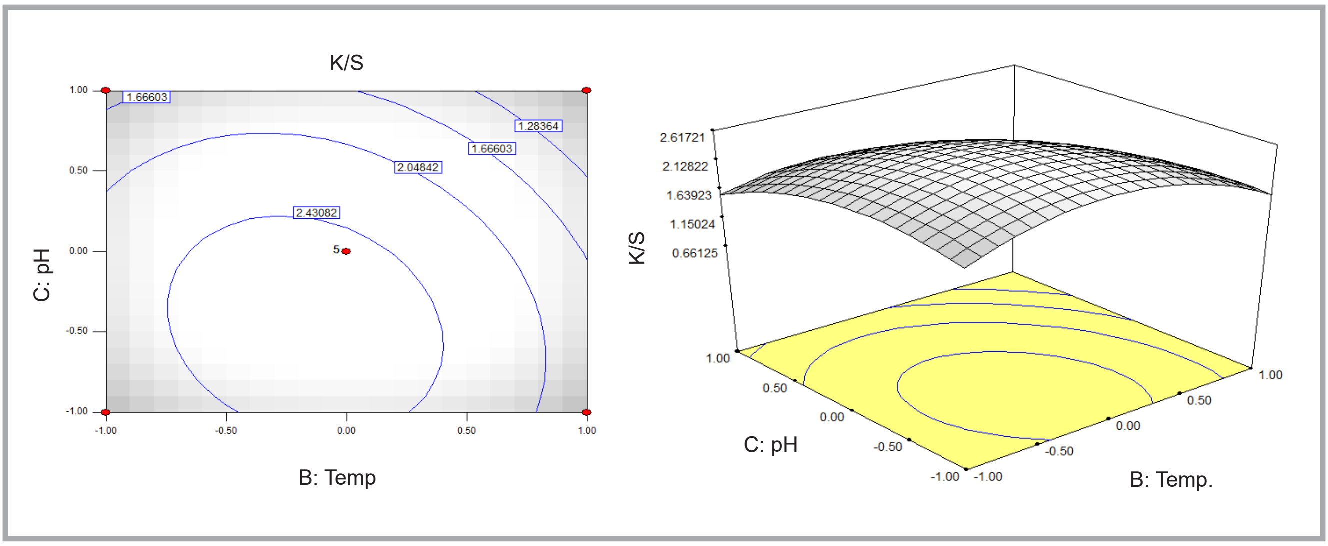

Figure 4. Effect of temperature and pH on the K/S value of NLS dyed nylon fabric.

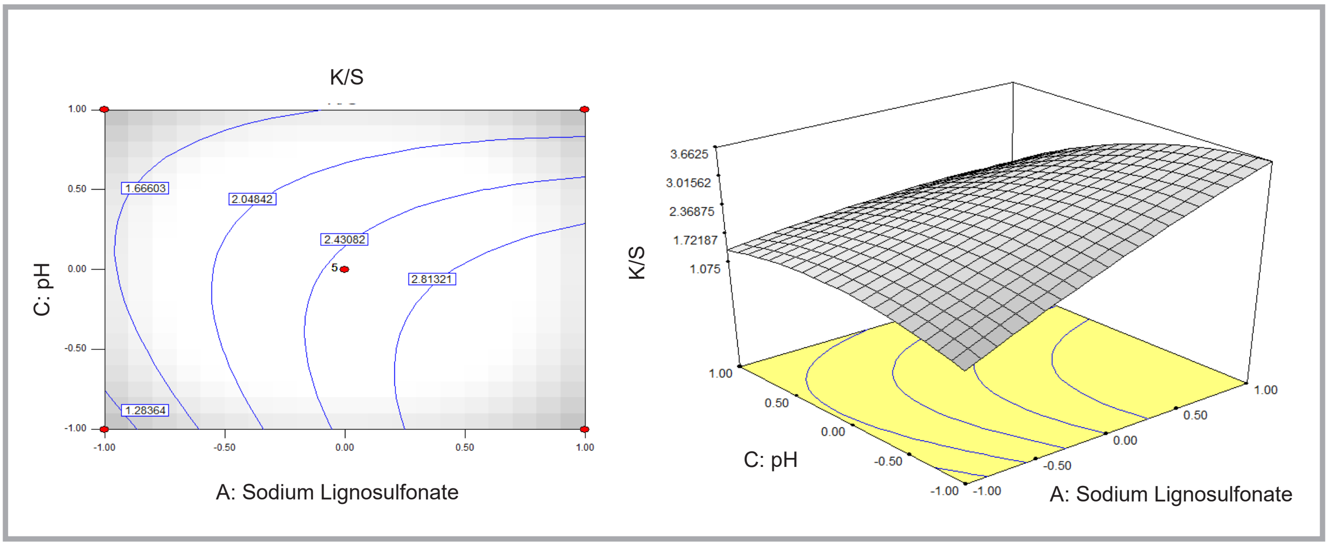

Figure 5. Effect of a) pH and b) concentration of NLS on the K/S value of nylon fabric.

tour plot shows that K/S increases with an increase in the concentration of the NLS solution. K/S initially increases and then decreases with an increase in the temperature of the NLS solution. However, the maximum value of K/S is observed at the highest concentration of the NLS solution and around the mid-value of the temperature of the NLS solution.

Effect of temperature and pH on K/S

Figure 4 displays a contour diagram showing the K/S factor against the temperature and pH of the NLS solution. It can be seen from the contour diagram that the K/S factor initially increases marginally and then decreases with an increase in the temperature and pH of the NLS solution. The trend shows the best result of K/S at a lower temperature and acidic medium of the solution.

Effect of concentration of NLS solution and pH on K/S

A contour diagram of the influence of the concentration and pH of the NLS solution on colour values of the dyes fabric is shown in ***Figure 5***. The graph clearly shows that K/S increases with an increase in the concentration of the NLS solution. It initially increases and then decreases with an increase in the pH of the solution. The increase in K/S with an increase in the concentration of the NLS solution is attributed to the increase in lignin in the fabric. It is also indicated that the acidic medium leads to an increase in the fabric's K/S as opposed to a basic medium. During these experimental runs, the highest value of colour strength (K/S) achieved was 4.18 at 20% NLS concentration, 75 °C temp, and 5.5 pH.

ANOVA analysis of colour strength (K/S)

ANOVA analysis was conducted to understand the influence of the process parameters on the K/S value. The main effects and interaction effects of the process parameters on K/S are shown in ANOVA ***Table 3***. The Model F-value of 4.89 implies the model is significant. There is only a 2.41% chance that a Model F-value this large could occur due to noise. Values of Prob > F less than 0.05 indicate model terms are significant. In this case A, C & B^2 are significant model terms. The Lack of Fit F-value of 20.62 implies the Lack of Fit is significant. There is only a 0.68% chance that a Lack of Fit F-value this large could occur due to noise.

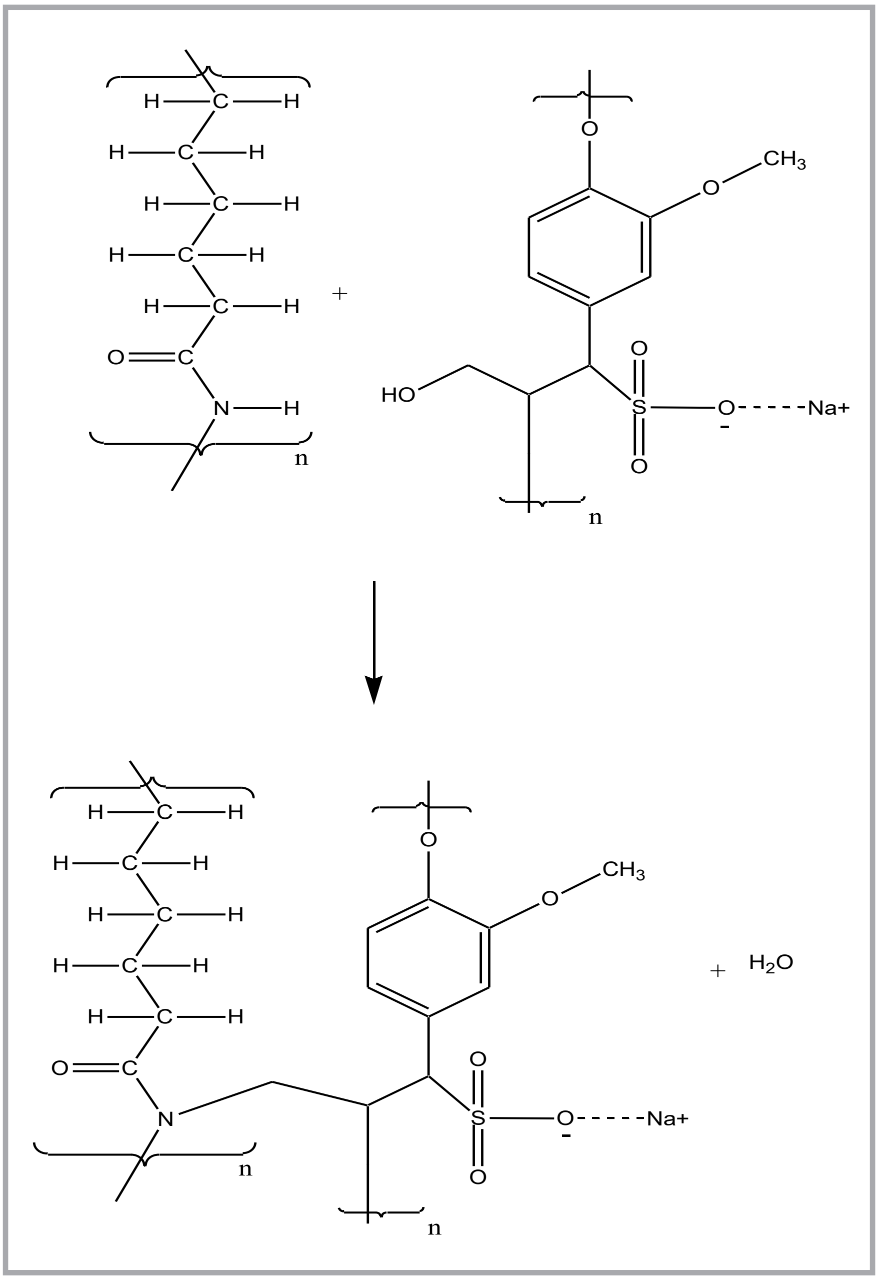

Figure 6. *Reaction mechanism of NLS with Nylon 6.*

Table 3. *ANOVA of colour strength (K/S).* ***Note:*** ** Indicates statistically significant at 95% confidence level.*

Source	Sum of squares	DF	Mean square	F-value	Prob > F
Model	10.20	9	1.13	4.89	0.024*
A	4.51	1	4.51	19.46	0.003*
B	0.61	1	0.61	2.65	0.147
C	1.32	1	1.32	5.69	0.048*
A^2	0.072	1	0.072	0.31	0.593
B^2	1.43	1	1.43	6.18	0.041
C^2	0.69	1	0.69	2.99	0.127
AB	0.084	1	0.084	0.36	0.566
AC	1.17	1	1.17	5.075	0.058
BC	0.108	1	0.108	0.46	0.515
Residual	1.62	7	0.23		
Lack of Fit	1.52	3	0.50	20.62	0.006*
Pure Error	0.098	4	0.024		
Cor Total	11.83	16			

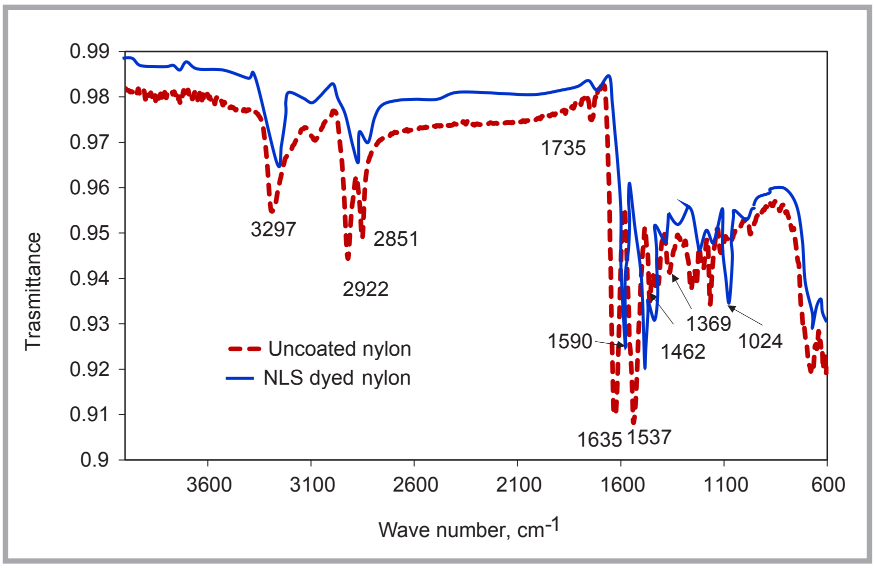

Figure 7. *FTIR spectra of untreated and NLS dyed nylon*

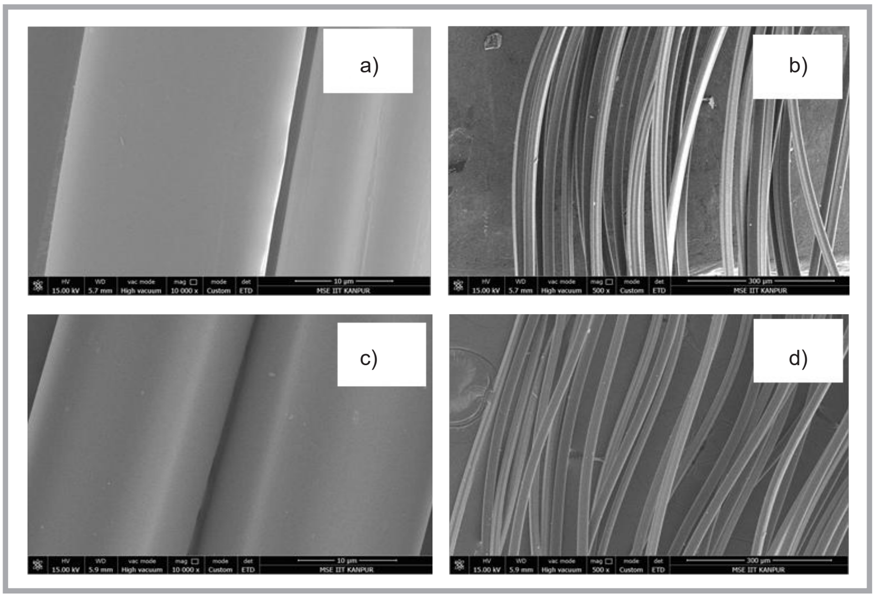

Figure 8. *SEM Images of a) NLS dyed nylon (1000 X), b) NLS dyed nylon (500 X), c) uncoated nylon (1000 X), d) uncoated nylon (500 X).*

A model equation of the coding unit is shown in ***Equation (1)***. It can be seen that the fabric's ultraviolet protection factor has a quadratic relationship with the concentration, temperature, and pH of the NLS solution, which can be shown by the following equation:

$$Y(K/S) = 2.50 + 0.75A - 0.28B - 0.41C - 0.13A^2 - 0.58B^2 - 0.41C^2 - 0.15AB - 0.54AC - 0.17BC \quad (1)$$

The ***Equation (1)*** can predict well the K/S value for process parameters with a coefficient of determination (R^2) of 0.8628. This shows that the model is quite significant. The correlation coefficient, R^2, is important for validation of the model developed. High R^2 values indicate that the responses predicted are close to the experimental values and that the models are suitable to correlate with the experiment data.

Mechanism of reaction between NLS and nylon

The mechanism of the reaction between nylon molecules and sodium lignosulphonate proposed is shown in ***Figure 6***. Nylon 6 and sodium lignosulphonate molecules possess –NH- and – free radical group –OH-, respectively. The free radical of the OH group of NLS attaches with the –NH- group of nylon 6, consequently releasing H_2O molecules during the reaction.

FTIR Analysis

FTIR spectra of uncoated nylon and NLS coated nylon are shown in ***Figure 7***. FTIR spectra of pure nylon fibre show an absorption band at 3297 cm^{-1} that may be attributed to the vibration of –OH groups, the band at 2922 cm^{-1} to –CH asymmetric stretching, the band at 1635 cm^{-1} to amide I, the band, at 1537 cm^{-1} to $–CH_2$ asymmetric deformation of amide II, the band at 1462 cm^{-1} to $–CH_2$ scissoring and –NH deformation, and the band at 1369 cm^{-1} to $–CH_2$ wagging of amide III. FTIR spectra of the NLS dyed nylon show all vital peaks of nylon fibre as well as characteristic peaks of lignin. Shifting of the peak of 1635 cm^{-1} of nylon to a new position at 1590 cm^{-1} after NLS treatment indicates the intermolecular interaction between the –NH- group of nylon with the –OH group of lignin [13]. The prominent peak at 1024 cm^{-1} indicates the C-H plane deformation of lignin [14-16].

FESEM and EDX analysis

Surface topographic FESEM images of the control and NLS dyed nylon 6 fabric samples are shown in ***Figure 8***. It is evident from this that there is no prominent deposition visible on the fiber surface, which may be because of the chemical reaction between the nylon and NLS rather than the physical coating on the surface.

For evidencing the presence of NLS on the surface of nylon, EDX analysis was carried out for all samples. EDX spectra of untreated and 20% NLS dyed nylon fabric are shown in ***Figure 9***. It is visible from the EDX analysis that the control nylon fabric showed three peaks for carbon, nitrogen, and oxygen elements. In addition to the said peaks, the NLS dyed fabric sample showed extra peaks for sulphur and sodium due to NLS treatment. From ***Table 4***, it is observed that the pure nylon fabric sample has 64.78% carbon, 17.56% nitrogen, and 17.65% oxygen. The element composition is changed after NLS treatment of nylon fabric in terms of sodium and sulphur. The NLS dyed nylon fabric sample has 63.87% carbon, 16.77% nitrogen, 19.04% oxygen, 0.24% sodium and 0.07% sulphur.

Colour Strength of NLS dyed fabrics

The fabric colour strength of the control and NLS dyed fabric at different NLS concentrations was measured, the results of which are shown in ***Table 5***. These samples were prepared with an optimum condition of acidic pH (5.5) and 75 °C

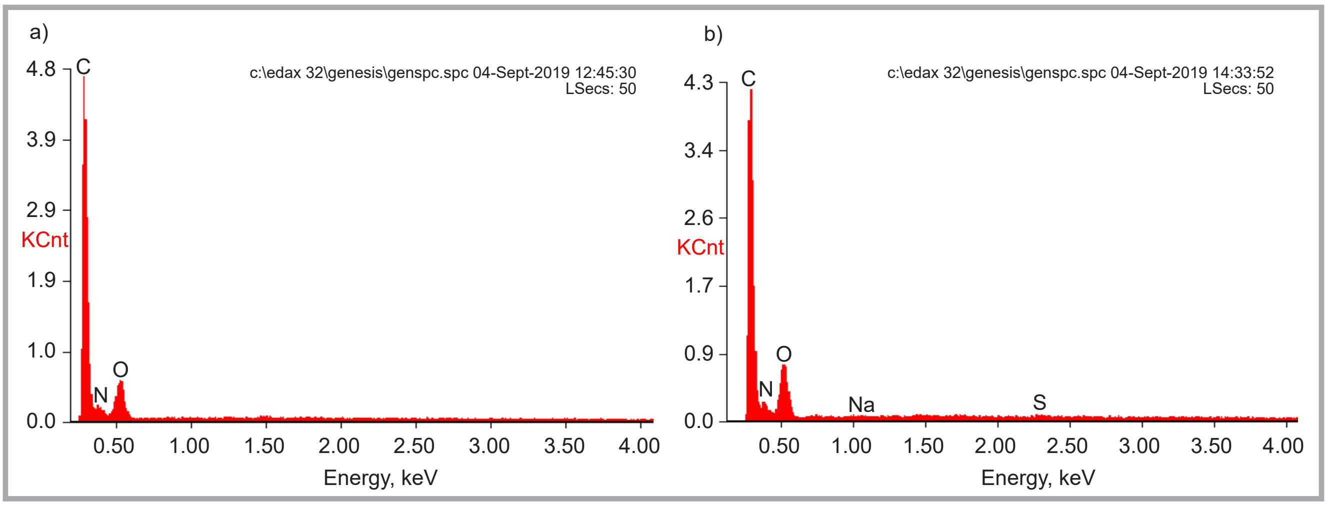

Figure 9. *EDX spectra of a) control nylon and b) NLS dyed nylon.*

temperature, and the NLS concentration kept on being increased to obtain greater values of K/S. Lignin is brown due to the presence of chromophoric groups, such as catechols, aromatic ketones, coniferaldehyde, stilbenes, and conjugated phenolics [17-18]. These chromophores can interact with nylon to impart a yellow colour to the fabric (***Figure 10***). It is visible from ***Table 5*** that an increase in the concentration of sodium lignosulfonate induces a linear increase in the value of the colour strength. These increases in the colour strength values of the NLS dyed fabric samples were found statistically significant, as proven by the ANOVA test. It is reported that the UPF of the fabric has a positive relationship with the colour values of the fabric [16] because dark colour absorbs higher UV radiation than light shades. In our study we also observed a direct positive correlation between the UPF and K/S.

Ultraviolet protection factor (UPF) of NLS dyed nylon fabrics

UPF values of control and NLS dyed fabric samples are shown in ***Table 5***. It is depicted that the presence of sodium lignosulfonate (NLS) on nylon fabric substantially enhances the ultraviolet protection ability of nylon fabric up to a UPF of 64.54 in comparison to 10.96 of the control sample. The UPF value of the NLS dyed fabric is excellent, in a much safer range of ultraviolet protection. This significant enhancement in UPF is due to the presence of lignin, which contains an aromatic ring and methoxyl $-OCH_3$, carbonyl –CO, and hydroxyl –OH groups and is known for its ability to absorb ultraviolet rays [7-8].

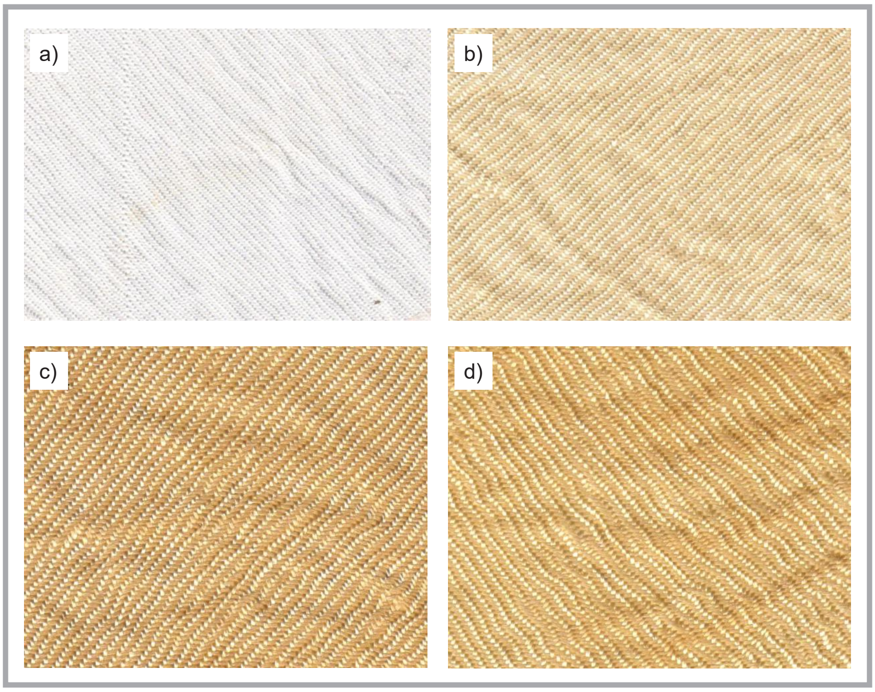

Figure 10. *Images of a) untreated nylon fabric, b) nylon dyed with 10% NLS solution, c) nylon dyed with 15% NLS solution, and d) nylon dyed with 20% NLS solution.*

Table 4. *EDX analysis of control and NLS dyed nylon samples*

Sample	C (A.W%)	N (A.W%)	O (A.W%)	Na (A.W%)	S (A.W%)	Total
Untreated nylon	64.79	17.56	17.65	–	–	100
20% NLS dyed nylon	63.87	16.79	19.04	0.24	0.07	100

Table 5. *Colour strength (K/S), lustre, washing fastness, and light fastness.* ***Note:*** *N – nylon, NLS – sodium lignosulfonate*

Sample code	NLS Con, %	Colour strength				UPF	Lustre	Washing fastness	Light fastness
		K/S	L	A	B				
N	0	0.10	91.9	0.76	3.01	10.96	4.55	5	4
NNLS-10	10	2.24	80.4	4.28	21.14	44.36	3.67	5	4
NNLS-15	15	4.10	75.62	5.53	25.31	52.72	2.88	5	4
NNLS-20	20	4.81	73.0	6.19	25.72	64.98	2.05	5	3

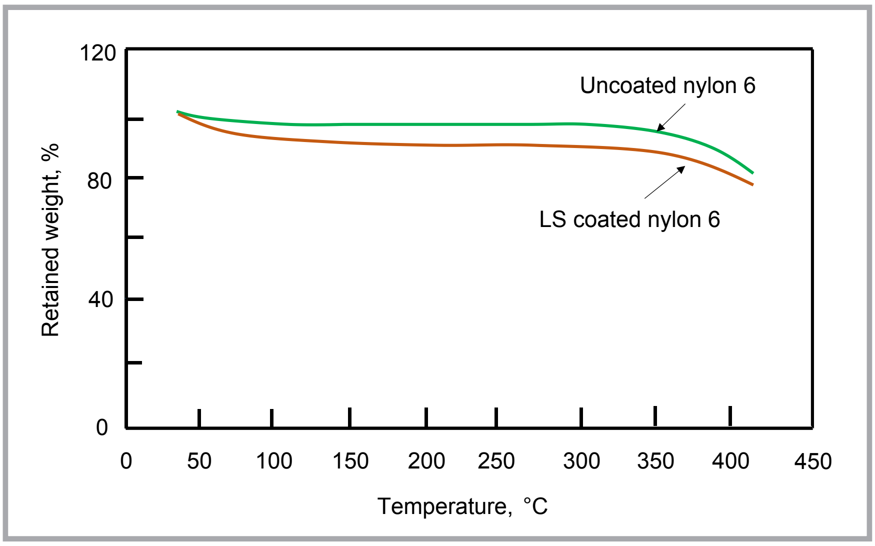

***Figure 11.** TGA analysis of control and NLS dyed nylon fabrics.*

Table 6.

Sample code	NLS Con, %	Tensile strength, MPa Mean (SD)	Elongation, mm Mean (SD)	Tensile modulus, MPa Mean (SD)
N	0	62.36 (2.10)	35.62 (1.55)	277.90 (22.18)
NNLS-10	10	67.29(2.51)	40.54 (1.35)	276.18 (33.37)
NNLS-15	15	69.67 (4.32)	39.96 (1.32)	297.85 (21.85)
NNLS-20	20	67.56 (1.71)	39.39 (1.77)	300.69 (9.90)

Fastness properties of NLS dyed fabrics

Lightfastness properties of the NLS dyed fabric samples tested under an xenon arc lamp are shown in ***Table 5***. All NLS dyed fabric samples reveal a very good lightfastness rating. It is evident from light fastness data that sodium lignosulfonate has excellent affinity with the nylon fiber surface under xenon arc light. The washing fastness of the NLS dyed nylon samples is excellent and does not exhibit any staining on either cotton or scoured nylon fabric after washing. This may be attributed to the fact that sodium lignosulfonate has an effective affinity with nylon fabrics. It is also noted that the soaping process undertaken after the NLS treatment is one of the reasons for getting good fastness properties.

Lustre of NLS dyed fabrics

Fabric luster is one of the attributes which affects the visual appearance of a fabric. It is the amount of specular light the fabric reflects. The luster behaviour of the control and NLS dyed fabrics at different concentrations is shown in ***Table 5***. The untreated nylon fabric was found to be highly lustrous (4.55 Gloss 60°). The presence of NLS on dyed nylon fabric samples suppressed the luster intensity, and the concentration of NLS is inversely proportional to the luster intensity. The reduction in luster intensity in the presence of sodium NLS is attributed to its colouring potential, which exhibits higher absorption of light in the visible region.

Tensile properties of untreated and NLS dyed fabrics

The tensile strength, modulus, and elongation of untreated nylon fabrics as well as all NLS dyed nylon fabrics are shown in ***Table 6***. It can be seen that after NLS treatment there is no significant change in any of these tensile properties of nylon fabric. Therefore, it can be concluded that NLS treatment is very safe for the modification of nylon fabric for enhancement of the aesthetic value as well as UV protection properties.

Thermo-gravimetric analysis of NLS dyed nylon samples

TGA analysis of untreated nylon and the 20% NLS solution dyed nylon sample was conducted to understand the thermal degradation behaviour, the TGA thermogram of which is shown in ***Figure 11***. It can be seen that NLS treatment does not cause any significant deterioration of the thermal stability of nylon. The insignificant weight loss at 400 °C may be due to the loss of NLS molecules from the nylon surface, but without affecting the nylon 6 polymer.

Conclusions

Box-Behnken response surface methodology reveals that NLS concentration (A) is the most significant main effect influencing the colour strength of NLS dyed nylon fabric. The maximum K/S value achieved was 4.81 when NLS treatment was carried out 20% NLS concentration, 75 °C temperature and 5.5 pH. It was observed that the colour value can be achieved with a high level of NLS concentration, and a medium level of temperature and pH. A chemical reaction between the free radical of the OH group of NLS and the –NH- group of nylon 6 was suspected during the dyeing process, which is evidenced by the FTIR study. FESEM and EDX results provide evidence of the chemical reaction of NLS with nylon fabrics. UV-VIS spectroscopy results show a deeper colour strength of the samples with concentrated NLS treatment, due to which the lusture of the fabrics decreased when dyed with concentrated solution. The tensile modulus, strength, elongation and thermal stability of nylon fabrics do not deteriorate after NLS treatment. The wash fastness of all NLS dyed fabrics was found to be excellent.

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