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Sustainable Enzymatic Desizing of Cotton with Bio-surfactant Extracted from Soapnut

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Article

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ABSTRACT

Surfactant is one of the major consuming auxiliaries in textile processing. The rising demand for petroleum-based surfactants is in focus and it is tremendously utilized to fulfil the need for surfactants in textile industries. These petroleum-based surfactants are one of the major pollutants of textile wastewater. Many attempts have been made to replace this with low toxicity to make the process sustainable. The present investigation works on the same objective to replace the petroleum-based surfactant from desizing by using soapnut extract as a wetting agent. The process was optimised by using a modern statistical technique of Response Surface Methodology [RSM]. The initial designing was conducted using 10 g/l soapnut extract and 2% enzyme for 30 min at 75 °C and found satisfactory results. Additional desizing experiments were performed to optimize the process using RSM with weight loss as the primary outcome. An optimised desizing recipe provided by DOE numerical optimisation, viz., a concentration of 10 g/l soapnut extract and 2% enzyme at 75 °C for 40 min, was performed to validate. The findings demonstrate that optimum weight loss (6.58%) and desirable levels of absorbency (14 s), whiteness (73.52), yellowness (22.84 indices, bending length (2.1 cm), Flexural rigidity (98.13 mg.cm), while minimally affecting tensile strength (10.77). Enzymatic desizing with synthetic or soapnut-extracted wetting agents yields identical results and satisfies performance standards for industrial use. The Sustainable way of enzymatic desizing of cotton with bio-surfactant extracted from soapnut may be the green alternative to synthetic surfactant-based desizing.

KEYWORDS

enzymatic desizing, cotton, bio-surfactant, soapnut, sustainability

INTRODUCTION

Surfactants are crucial in textile processing, aiding wetting, emulsification, dispersion, and foaming. The demand for surfactants, often derived from petroleum-based sources, has been high due to their effectiveness in various applications within the textile industry. However, the environmental impact of these surfactants, particularly in textile wastewater, raises concerns about pollution and sustainability [1,2]. Petroleum-based surfactants can contribute to water pollution, affecting aquatic ecosystems and posing challenges for wastewater treatment [3]. The need for sustainable and environmentally friendly alternatives has led to efforts to replace these surfactants with low-toxicity options to make textile processing more eco-friendly [4].

Desizing is the first step during the woven fabric processing, preparing it for subsequent pretreatment and colouration by removing the size added to the yarn before weaving [5,6]. Desizing was traditionally accomplished with the use of acid and rot steeping techniques. The rot steeping process is not particularly polluting but is time-consuming, and the chances of uneven desizing are higher [7,8]. Using acids in desizing is harmful to working conditions and not environmentally friendly [9] Enzymatic desizing has been developed and is now commercially used in many cellulosic fibre treatments due to the significant limitations of conventional methods [2].

Several chemicals and auxiliaries are usually added during desizing to increase enzyme activity [10,11] The wetting agent, sometimes referred to as surfactant, is crucial to the desizing process because it facilitates the enzyme’s faster entry into the fabric. In desizing baths, ethoxylated surfactants or their blends are primarily used as wetting agents [12,13]. These surfactants are made from petroleum-based raw materials, and the ethoxylation reaction produces the potentially carcinogenic byproduct 4-dioxane, which is not biodegradable [14,15]. Synthetic surfactants are persistent in the environment due to their nonbiodegradable properties. However, in certain instances, it has been demonstrated that the results of partial breakdown are more hazardous than the original surfactant molecule. Additionally, surfactants facilitate the dispersal of other contaminants, such as heavy metals, exacerbating ecological issues [16].

Natural surfactants are "eco-surfactants" because they are environmentally friendly, biodegradable, affordable, less toxic, and biocompatible [17]. Natural surfactants, also called saponins and humic compounds, can be derived from either plants or animals [18]. The Sapindaceae family includes the *Sapindus mukorossi* plant, also known as soapnut, washnut, soapberry, reetha, and aritha. Three chemical substances—triterpenoid saponins, sesquiterpene glucoside, and fatty oil—combine to form soapnut saponin. Compared to synthetic surfactants, this bio-based non-ionic surfactant significantly lowers surface tension and offers strong detergency. Many researchers have tried to utilize saponin extracted from soapnut for textile processing such as degumming of silks and their varieties [19,20]. The utilization of soapnut extract as a natural surfactant for the leaching of toxic metal from the ashes of power plants is also studied, the bio-surfactant showed efficient removal of such hazardous substances from ash [21-23].

This research aimed to study bio-based surfactants for desizing to address the synthetic surfactants' downsides and environmental issues. Desizing cotton fabric using a combination of bacterial amylase enzyme and a bio-surfactant extracted from soapnut is an interesting and new area of research because it makes use of a complete non-synthetic chemical composition to achieve an eco-friendly process. The utilization of soapnut surfactant will help reduce the consumption of petroleum-based surfactants, which in turn will reduce the environmental impact.

EXPERIMENTAL

Material

Sized cotton greige fabric (15 x 15 cm of each sample) of 200 gsm with plain weave was purchased from Sanjay Shah and Associates, Vapi, India. Commercial amylase enzyme [Rexize LHT] and non-ionic wetting agent [Kleenox WLF Liq] were provided by Rossari Biotech Ltd, Mumbai, India. The soapnut fruits [Sapindus] were purchased from the general store in Mumbai. Potassium iodide and iodine crystals were purchased from SD Fine Chem Ltd, Mumbai.

Saponin extraction

As described by Patil et al., Saponin was extracted by aqueous extraction with a minor modification [17]. The soapnut cap was manually detached and ground into a fine powder using an electric grinder and sieved by using a mesh of 100 microns. 500 ml of distilled water was added to the beaker with 25 g of soapnut powder, and it was left to soak for 12 hours. The soapnut-soaked solution was heated at 90 °C for 60 minutes on a heating plate while constantly stirring to achieve the extraction. The mixture was filtered and evaporated at 90 °C to make 100 ml concentrated extract.

Desizing

The cotton-greige-sized fabric was desized using alpha-amylase enzyme [Rexize LHT] and a saponin solution of soapnut extract in a Rota-dyer machine [Rossari Labtech]. The pH of the bath was adjusted using acetic acid and soda ash at 6.5 - 7.5. The material-to-liquid ratio [MLR] and temperature were set at 1:20 and 75 °C for all experiments. The desized fabric was hot-washed, rinsed with cold water, and dried. Three independent desizing parameters, viz., enzyme concentration [% w/w], soapnut extract concentration [g/l], and time [min] and dependant variable weight loss (%) were optimised using RSM and Box-Behnken design. The levels of independent variables selected for the experiment's optimisation are shown in Table 1. Five centre points were used in all 17 experiments. The commercial synthetic wetting agent-based desizing was performed with a standard recipe consisting of 1% [w/w] enzyme and 2 g/l surfactant; the pH of 6.5 - 7.5 was adjusted by using acetic acid. Then, the bath temperature was raised to 75 °C, and desizing was continued for 30 minutes. The material-to-liquid ratio was set at 1:20. After desizing, the fabric was hot-washed, rinsed with cold water, and dried.

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Table 1. Variables for desizing experiments

Symbol	Name	Unit	Lower limit	Upper limit
A	Enzyme	%	1.0	3.0
B	Soapnut extract	g/l	10	20
C	Time	Minutes	30	60

Testing and characterization

Weight loss

Weight loss of fabric after desizing was estimated by equation [1]:

$$\text{Weight loss [\%]} = \frac{W_a - W_b}{W_a} \times 100 \qquad [1]$$

Where W_a and W_b are the weight of fabric before the desizing and after the desizing, respectively.

TEGEWA solution preparation

10 g potassium iodide and 1g iodine crystals were dissolved in 800 ml of distilled water by agitation and stirring. 200 ml of ethanol was added to raise the final volume to 1000 ml. Size removal was analysed by putting a drop of indicator solution on the fabric. The colour change was observed for judging size removal.

Yellowness, Whiteness, Brightness

Whiteness, brightness, and yellowness index of desized cotton fabric were tested using Hunter, TAPPI T452, and ASTM D1925 scales, respectively, on Spectrascan-5100+ [Datacolor International, USA] computerized colour matching system.

Absorbency

AATCC 39-1980 method was used to determine the absorbency of desized fabric. The time required for absorbing water drops was noted. An average of 5 readings was considered.

Bending length and Flexural rigidity

The bending length and flexural rigidity of desized fabric were investigated according to ASTM D1388-18 by a stiffness tester. Equations (2) and (3) were applied to calculate the bending length and flexural rigidity.

$$C = \frac{L}{2} \tag{2}$$

$$G = W * C^3 \tag{3}$$

C- Bending length [cm], L- Length of overhang [cm], G- Flexural rigidity [mg.cm], and W- Weight per unit area [mg/cm^2].

Tensile characteristics

Universal Tensile Tester [Tinius Olsen, H5K-S UTM, USA] were used for tensile testing as per ASTM D 5035 [Strip method]. The Gauge length, Elongation range, and Test speed were set at 100 mm, 0 - 200 mm, and 100 mm/min, respectively.

RESULT AND DISCUSSION

Optimization and validation of the experiment using RSM

The Box-Behnken design was used to optimize the desizing of cotton fabric. The design of experiments [DOE] was performed according to the BBD matrix, and the findings are summarized in Table 2.

Table 2. Design matrix of soapnut extract desizing

Run	Enzyme [%]	Soapnut extract [g/l]	Time [min]	Weight loss [%]
1	1.0	20	45	6.23
2	1.0	15	30	6.35
3	3.0	15	30	6.98
4	1.0	15	60	6.61
5	3.0	20	45	7.14
6	1.0	10	45	5.42
7	2.0	15	45	6.89
8	2.0	10	30	6.08
9	2.0	15	45	6.47
10	3.0	15	60	6.89
11	2.0	20	30	7.18
12	2.0	10	60	6.74
13	2.0	15	45	6.63
14	2.0	15	45	6.89
15	2.0	20	60	6.18
16	3.0	10	45	6.69
17	2.0	15	45	6.58

Table 3. Analysis of variance [ANOVA] model results of soapnut extract desizing

Source	Sum of Squares	df	Mean Square	F-value	F-crit (α=0.05)	P-value	
Model	1.56	3	0.5214	4.67	3.41	0.02	significant
A-Enzyme [%]	1.19	1	1.19	10.68		0.0061	
B-Soapnut Extract [g/l]	0.3698	1	0.3698	3.31		0.0919	
C-Time [min]	0.001	1	0.001	0.0091		0.9256	
Residual	1.45	13	0.1117				
Lack of Fit	1.31	9	0.1453	4.04		0.0961	not significant
Pure Error	0.1441	4	0.036				
Cor Total	3.02	16					

Table 3 displays the ANOVA results of the statistical analysis for Weight loss of the desized samples. The F value of the model is greater than the critical F value, which implies that the null hypothesis is rejected. The P value represents the probability of obtaining the observed findings. The null hypothesis is rejected with the lowest level of significance using the P value. A smaller P value of 0.020 indicates a higher probability of achieving the observed outcome. A p-value of less than 0.05 indicates that the model is significant [24]. As a result, applying this model to desizing parameter optimization could result in an exact and predictable response. The numerical optimisation was run in DOE software by choosing a minimum level for all variables (enzyme and soapnut concentration and time) and a maximum target for response (weight loss). The solution provided by the software with a higher desirability value of 0.700 - 0.760 was considered an optimised recipe and validated by experiment, and its weight loss was 6.58%.

Regression analysis

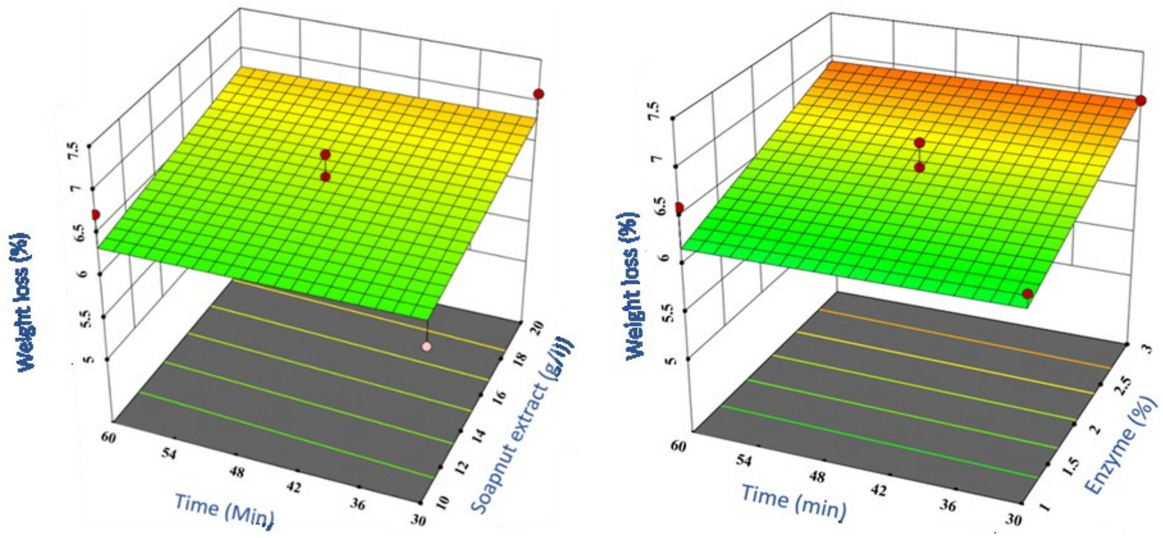
The execution of the design expert could be estimated using equation 4. Regression equation analysis was employed to forecast the outcomes of individual responses for desized samples. The R^2 0.5286 was obtained from the data analysis of the model. The weight loss of desized fabric may be predicted for each process parameter, such as the concentration of enzyme, the concentration of soapnut extract, and time, utilizing the equation below, which is described in a coded form.

$$\text{Weight loss} = +6.58+0.3863*A+0.215*B-0.0113*C \tag{4}$$

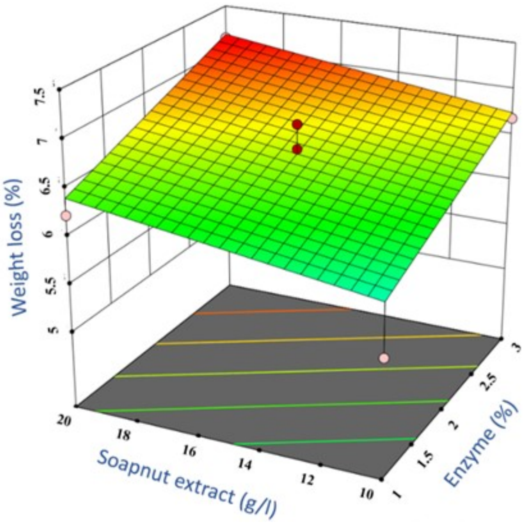
Where A, B and C stand for the concentration of enzyme, concentration of soapnut extract, and time, respectively.

Response Surface plot

The impact of enzyme concentration, soapnut extract concentration, and time on the weight loss of desized cloth is shown in Figures 1 a), b), and c). The combined effects of variables on the response (weight loss) of cotton fabric desized with enzyme and soapnut extract (as a wetting agent) were studied by the response surface plot of RSM. The impact of soapnut extract concentration and time on fabric weight reduction with a constant concentration of enzyme is shown in Figure 1 a). The enzyme's fixed concentration was 2%. Weight loss decreased to a deficient level when soapnut extract content rose. The soapnut extract concentration range of 10 - 15 g/l for 30 - 40 minutes resulted in significant weight loss. Figure 1 b) shows the weight loss of a cloth desized using a 15 g/l soapnut extract concentration as a function of time and enzyme concentration. Figure 1 b) shows that when the concentration of enzymes increases, the weight loss of the desized cloth increases; however, the desizing time does not lead to any significant loss in weight after 30–40 min. Most fabric weight loss occurred between 1 - 2 % enzyme for 30–40 minutes. The effect of soapnut extract and enzyme concentration at a constant desizing duration of 45 minutes is shown in Figure 1 c). The concentration of soapnut extract and enzyme causes more significant fabric weight loss. Maximum fabric weight loss was observed at a 2 - 3% enzyme concentration and a 10 - 15 g/l soapnut extract concentration.



a) b)



c)

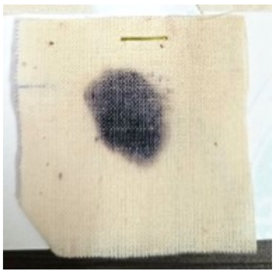
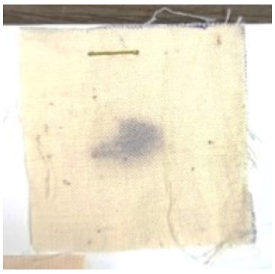
Figure 1. Response surface plots: (a) Soapnut extract concentration and time at a fixed enzyme concentration; (b) Enzyme concentration and time of desizing at a fixed concentration of soapnut extract; (c) concentration of Soapnut extract and enzyme at a fixed time of desizing.

Effect of desizing on physical properties of the fabric

Enzymatic desizing with both natural and synthetic wetting agents yielded identical results for all the characteristics listed in Table 4. The desizing process is essential in the woven fabric process as it determines the performance of all the succeeding processes [25]. The weight loss after desizing is the effect of size removal, which was found in the 6-7% range. The leading causes of the hydrophobicity of raw cotton fabric include hydrophobic contaminants in the cuticle of the fibre, a dried starch film, and hydrophobic fat found in the sized yarns. For fabrics treated with enzyme-soapnut extract and enzyme-syntenic wetting agent combinations, the absorbency values were between 13 and 14 seconds.

Table 4 Comparative assessment of desizing process

Parameters	Grey Fabric	Soapnut extract desized fabric	Synthetic surfactant desized fabric
Weight loss [%]	-	6.58	6.67
Absorbency [sec]	>60	14	13
Whiteness index	72.46	73.52	74.98
Yellowness index	31.32	22.84	23.12
Tensile strength [kgf]	15.93	10.77	10.91
Elongation [%]	19.70	18.24	19.20
Bending length [cm]	2.6	2.1	2.1
Flexural rigidity [mg.cm]	219.7	98.13	101.52

Parameters	Grey Fabric	Soapnut extract desized fabric	Synthetic surfactant desized fabric
TEGEWA			

Desizing significantly reduced the fabric's yellowness, but the whiteness barely changed. The tensile strength of the grey fabric is around 16 kgf due to the contribution of the yarn sizing to the improvement of strength; however, the removal of size during desizing lowered the fabric strength, as indicated in Table 4. The starch added to the yarn during sizing serves as a binder and reduces the mobility of the fibre within the yarn, stiffening it. As a result, the bending length and flexural rigidity of grey cloth are increased after desizing [26]. The yarn becomes more flexible as bending length and flexural rigidity are reduced because the size removal increases the yarn's internal fibre mobility [27]. There is no significant difference between the results of flexural rigidity of synthetic and natural surfactants. After desizing, the fabric's tensile and elongation characteristics barely changed. sizing additives act as binders to provide strength to the yarn during weaving. The removal of size reduces the binding between the fibres, and the fabric strength is affected after desizing [5,28]. TEGEWA rating 8-9 demonstrated the effective removal of size from the cloth; as can be seen, while the desized fabric displayed very light blue stains, the sized fabric revealed a dark violet. Weight loss and TEGEWA rating of desized fabric are the major parameters for the evaluation of desizing efficiency. Previous works on desizing of cotton report a 7-9 % loss in weight and an 8-9 TEGEWA rating of fabric, which is closer to our findings [29].

CONCLUSION

Desizing plays a vital role in the processing of woven cotton fabric as it aids in removing size from the fabric and enhances the effectiveness of subsequent processes. However, the uneven removal of starch often results in uneven quality in pretreatment, dyeing, and printing. This research utilizes soapnut extract as a wetting agent instead of synthetic wetting agents in the enzymatic desizing process of cotton. The primary design used 10 g/l soapnut extract and 2% enzyme for 30 min at 75 °C. Primary desizing experiment shows better outcomes and hence, further desizing trials were conducted to optimise the procedure using response surface methodology. An optimised desizing recipe provided by DOE numerical optimisation, viz., a concentration of 10 g/l soapnut extract and 2% enzyme at 75 °C for 40 min, was performed to validate the model. The findings demonstrate that optimum weight loss (6.58%) and desirable levels of absorbency (14 sec), whiteness (73.52), yellowness (22.84 indices, bending length (2.1 cm), Flexural rigidity (98.13 mg.cm), while minimally affecting tensile strength (10. 77). To identify the optimized conditions for desizing using natural wetting agents, a statistical model based on response surface methodology [RSM] was employed. The results indicate that the most favourable outcomes were obtained with a soapnut extract concentration of 10 - 15 g/l and an enzyme concentration of 1% - 2%, at a temperature of 75 °C for 30 - 40 min. These outcomes are comparable to those achieved with commonly used industry wetting agents. Thus, by adopting the optimized biosurfactant-based desizing approach, it is feasible to reduce the reliance on synthetic chemicals and ensure the environmental sustainability of this procedure. Utilizing bio-surfactant extracted from soapnut in other textile processes like scouring of wool and cotton, soaping of dyed fabric, and washing of synthetic fibres will be an interesting area for future research.

Author Contributions

Conceptualization –Athalye A and Patil H; Methodology – Athalye A and Patil H; Formal analysis – Patil H; Investigation –Patil H; Writing-original draft preparation – Patil H; Writing-review and editing – Athalye A; Visualization – Patil H; Supervision – Athalye A. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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