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Environment friendly approach to remove unbound disperse dyes from polyester fabric

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Abstract

A conventional wash-off agent in disperse dyeing process with polyester fabrics is sodium dithionite ($Na_2S_2O_4$) which is environmentally unfavourable because theresulting by-products cause various problems to the disposal wastewaters. In this research, a new idea was investigated wherein the use of soap nut extraction was assessed in the wash-off process to remove unfixed disperse dyes from polyester fabrics. In this regard, dyed polyester fabric was subjected to wash-off process using soap nut extractionof various concentrations [5, 10,15 and 20%] at 80^0C for 20 minutes. Dyed fabrics were subjected to both conventional and new wash-off methods, and comparisons were made. The effectiveness of soap nut extraction-based wash-off was evaluated in terms of color fastness,color difference values, colorabsorbance, water absorbency and physical properties such as weight loss, bursting strength. The colorabsorbance was considered as indicative of the removal of unfixed dyes from the fabric.Soap nut extraction-based wash-off process shows better fastness properties, lower color difference, good water absorbency rate, higher color absorbance (color removal efficiency) than the conventional based wash-off process at 20% concentration. At 5% concentration of soap nut extraction-based wash-off process shows better physical propertiesin terms of higher bursting strength and low weight loss.Thus, in terms of fastness, colorimetric characteristics, color removal and physical properties, the conventional based wash-off process can be replaced by a single, non-dithionite-based wash-off process by soap nut extraction. This enabled a major reduction to be achieved in terms of the BOD, COD, and TDS that are typically generated as a result of the conventional based wash-off process of disperse dyes on polyester; it also offers the potential of avoiding the environmentally unacceptable generation of aromatic amines. Hence, soap nut extraction virtually has a potential to be used as a green wash-off agent in polyester disperse dyeing.

Keywords: Soap nut, wash-off, disperse dyes, unfixed dyes, colorfastness.

1. Introduction

Polyester (PolyEthyleneTerephthalate, PET) fibres have emerged as having a leading share among natural and synthetic fibres when production and consumption of different fibres in the world is compared [1 - 5]. It enjoys this dominant position due to its desirable properties, the most important of which are versatility and ease of use. Polyester is dyed exclusively with disperse dyes. Disperse dyes are non-ionic molecules of relatively small molecular size with limited solubility in water at room temperature. Dyeing is generally carried out at high temperatures, often around 130^{o}C, above a temperature referred to as the dyeing transition temperature, which is closely aligned with the glass transition temperature and where higher segmental mobility of the polymer chains enables the dye molecules to penetrate into the fibre. Because of the low solubility of disperse dyes in water and the tendency for particles in the dye dispersion to aggregate during the course of dyeing, some residual dye commonly remains on the fibre surface at the end of the dyeing phase. These surface deposits may have an adverse effect on the color and fastness properties of the dyed fabrics, if present, and an aftertreatment to remove them is generally introduced into the dyeing process.

The washing process which is used traditionally to remove the deposits of unfixed dye from the surface of the fabric after dyeing is referred to as reduction clearing. This process involves treatment of the dyed fabric with an aqueous solution of a reducing agent in alkaline conditions at temperatures below the boiling point of water. Commonly, reduction clearing employs a solution of sodium dithionite, sodium hydroxide at temperatures in the range 60-80°C for a period of 20-30 minutes.

Wash-off process is of technical importance in polyester dyeing in order to improve the brightness of the color and the fastness properties of the dyed fabric, especially to wet treatments [6]. There are, however, certain environmental, technological and economic disadvantages associated with the traditional reduction clearing process. The environmental disadvantage of the process is that it generates sulphur-containing degradation products derived from sodium dithionite which appear in the effluent with potentially toxic effects, notably sulphite (SO_3^{2-}), sulphate (SO_4^{2-}) and thiosulphate ($S_2O_3^{2-}$). Waste waters containing sulphites and sulphates are corrosive and can cause severe damage in waste lines [7, 8]. The oxidation products of sodium dithionite may also cause oxygen depletion in water streams resulting in an increase in chemical oxygen demand [4].

Stringent international laws and increased public concern about the environment have challenged the industries to develop eco-friendly processes [9]. In textile dyeing and finishing units, water is extensively used in almost every step of different processes [10]. One of the most expensive, time-consuming and polluting processes in textile coloration is the removal of loosely bound unfixed or hydrolyzed dyes from a textile substrate [11]. The wash-off process consumes large quantities of water and produces large volumes of wastewater containing residues of dyes, high temperature, turbidity, and COD & BOD concentration [12]. The direct discharge of such wastewater into water bodies without adequate treatment contaminates the water and adversely affects many species of aquatic flora and fauna [13, 14]. The wash-off step to remove unfixed dyes is critical to guarantee acceptable color fastness properties in the final dyed material [15].

A number of alternative organic and inorganic agents and some proprietary compounds have been proposed and marketed as replacements for sodium dithionite, but these do not appear as yet to have found significant commercial acceptance for a variety of reasons, associated mostly with environmental concerns, efficiency and cost [8, 16, and 17].Many industries have installed effluent treatment plants to meet the increasing stringent demands of environmental safety standards, but an approach to reduce the effluent and its hazards would have many advantages [18].

In the search of modified green technologies that meets the demand of green chemistry, the growing interest is on chemicals that are less pollutant, highly bio-degradable, safe towards ecology and of course easy to recycle or reproduce. Saponin is one of the most commonly known plant-based surfactants [19].

The fruits of *Sapindus mukorossi* (Family-Sapindaceae) also called as soapnut, contain Saponins about 6-10 % by weight [20]. Soapnut has been traditionally used for cleansing purposes and owes detergent action due to its saponin content. One of the important ingredients of soapnut is saponins which include triterpenoidal saponins, and triterpene saponin hederagenin [21]. When they come in contact with water, they provide surface activity and form soap-like foaming solutions. These saponins provide the functionality of surfactants, the ability to wet, emulsify, solubilize foam, disperse, clean, and condition.

Soap Nut Extract is a standardized, powdered extract prepared from the fruit (nuts) of Sapindus trifoliatus [22]. It is a source of saponins and functions as a mild detergent, cleanser, surfactant, antimicrobial and anti-inflammatory agent. The soapnut shell has a great potential as a natural surfactant for washing the soils polluted with organic compounds and crude oil [23]. The saponin from Soapnut acts as a natural bio-surfactant.

Using soap nut natural fabric scouring can be also helpful in maintaining BOD and COD of waste-water to a tolerable level[24].Soap nut has been used as an ingredient of shampoo and for cleaning wool fibers for many years[25].It shows better surface tension reduction and cleansing property. Moreover, it has good foaming power and creates stable emulsion at higher concentration [26] successfully also proved the improved tearing strength and color fastness properties of cotton woven fabric scoured with soap nut [27]. Soap nut appear to be an attractive alternative due to their environment friendly nature and their growing use on textiles.

This paper describes the use of soap nut extraction-based wash-off processes for polyester fabric which had been dyed with disperse dyes and compares the colorimetric and fastness data secured for the washed-off dyeing to those obtained using conventional wash-off methods.

2. Materials and Methods

2.1 Substrate

The textile substrate used in this study consisted of 100% polyester dyed knitted fabric of 170 gram per square meter.

2.2 Conventional Detergent

Most common detergents such as NaOH and $Na_2S_2O_4$ are used in this research work.

2.3 Soap nutextraction preparation

Commercially available, well-dried fruits of soap nut were used in this study and they were procured from a local market. Soap nut fruits were washed, dried, and ground to powder. Soap nut fruits were shelled and used only hulls to grind to powder.

Weighed quantity of soap nut fruit (100 g) was taken and dried in oven at 60 C for 1h, to dry the residual moist. The seeds were separated from the fruit and ground to powder form. A definite quantity of powder (50g) was taken and boiled with 100 ml water with continuous stirring and filtered and collected for further experimentation. Different concentrations of soap nut extractions based on own weight of fabric used in the study was shown in the Table.1.

Table.1 Concentrations of wash-off detergents used in the study

Samples Code	Wash-off method	
	Soap nut extraction method	Conventional method (NaOH and $Na_2S_2O_4$)
CN 3		3% each
SN 5	5%	
SN 10	10%	
SN 15	15%	
SN 20	20%	

2.4 Wash-off procedure

The dyed fabric swatch was given conventional wash-off treatment as shown in Table.2, and regarded as a reference sample. The other fabric sample was subjected to a new wash-off method containing soap nut extraction at various concentrations [5, 10, 15 and 20%] for 20 minutes at 80^{0}C and the samples treated was compared with the reference one. The soapnut based extractions were added directly in all wash-off steps the same way as detergents in the conventional wash-off.

Table.2 Conventional wash-off steps

Step	Operation	Temperature	Time
1	Cold rinse	30°C	20 minutes
2	Neutralization with acetic acid	30°C	
3	Warm Wash	50°C	
4	Hot wash	80°C	
5	Warm wash	50°C	
6	Cold wash	30°C	

3. Characterization

3.1 Color fastness

The conventional and soap nut extracted based wash-off samples were tested for their color fastness to wash, water, perspiration and rub fastness with their respective standards shown in the Table.3.

Table.3 Color fastness test and its standards

Test	Standard
color fastness to wash	ISO 105-C06: A2S
color fastness to water	ISO 105-E01
color fastness to perspiration	ISO 105-E04
color fastness to rub	ISO 105-X12

Color fastness is assessed generally by comparing any staining of specified adjacent fabrics during the test with a set of standards ‘grey scales. A numerical grading is given on a scale of 1-5, where 1 is very poor and 5 is excellent. Generally, a grade of 4 or above is deemed acceptable for commercial use [28].

3.2 Color Measurements

The color coordinates, L*, a*, b*, C*, h°, of the conventional treated and soapnut based extraction treated samples were measured using a Data color Spectra flash SF600 under illuminant D65,using a 10° standard observer. The CIELAB color space is organized in a cube form [29]. The L*axis runs from top to bottom. The maximum for L* is 100, which represents a perfect reflecting diffuser. The minimum for L* is zero, which represents black. The a*and b* axis have no specific numerical limits. Positive a* is red. Negative a* is green. Positive b* is yellow. Negative b* is blue. ΔE* is a single value which takes into account the differences between the L*, a* and b* of the sample and standard. ΔC* is the difference in chroma between the sample and standard. ΔH* is the difference in hue angle between the sample and standard.

3.3 Weight Loss

The weight loss percentage of the conventional treated and soapnut based extraction treated samples were determined by measuring the weights using weighing balance. The percentage weight loss of sample was determined according to the following formula:

$$\text{Weight loss } (\%) = [(W1 - W2)/ W2] \times 100$$

Where, W1 and W2 are the weights of control and treated samples, respectively.

3.4 Bursting strength

Bursting strength of fabric is the property of fabric to withstand the tension and stress when it is applied from both directions. To evaluate possible damages and weakening occurred to fabrics after both wash-offs, their bursting strength was determined according to the pneumatic method described in ISO 13938-2.

3.5Water Absorbency

Absorbency is used to describe the ability of a fabric to take in moisture - a very important property, which affects many other characteristics such as skin comfort, static build-up, shrinkage, water repellency and wrinkle recovery [30].

3.5.1 Immersion time

Standard TS EN 14697 was used to measure immersion time of both conventional treated and soapnut based extraction treated samples in the water. Five samples were cut from each type of samples with the dimensions of 100 mm × 100 mm, one side of each sample was spread on the water surface, and the time elapsed to thoroughly saturate the sample was recorded with a stopwatch.

3.5.2 Drop test

Water absorbency through drop test for both conventional treated and soapnut based extraction treated samples were measured according to AATCC TM 79 standard.

3.6 Measurement of color removal

Wastewater from both conventional and soapnut based extraction treated samples were measured at a wavelength corresponding to their maximum absorption (λ max) using a UV-visible spectrophotometer (thermo scientific evolution 201).

3.7 Assessment of the Environmental Impact

The residual liquors of both conventional and soapnut based extraction treated samples were sent to the measurement of biochemical oxygen demand (BOD) and chemical oxygen demand (COD), pH, total dissolved solids (TDS). COD and BOD analysis was carried out by open reflux and iodometric methods. TDS and pH were assessed using TDS (Make: ESICO, model 1601) and pH (Make: Eutech, model 510) meters respectively.

4. Results and Discussion

4.1 Effect on color fastness

A comparison of fastness properties of the conventional and soapnut based extraction treated samples is shown in Figure.1 and 2. Overall results show that both the conventional and soapnut based extraction treated samples exhibited similar fastness properties against washing, water and perspiration, clearly indicating that all the samples were found to be maintained at the same level of performance. The reason behind is high crystallinity nature of the polyester fibre can trap the dye molecules inside the fiber strongly, while the hydrophobicity nature restricts any entry of water molecules into the polymer system combined with a limited swelling during washing for the removal of excess dye molecules from fiber interior [31].

As far as the staining to adjacent cloth is concerned, the results were found to be very good in all case.

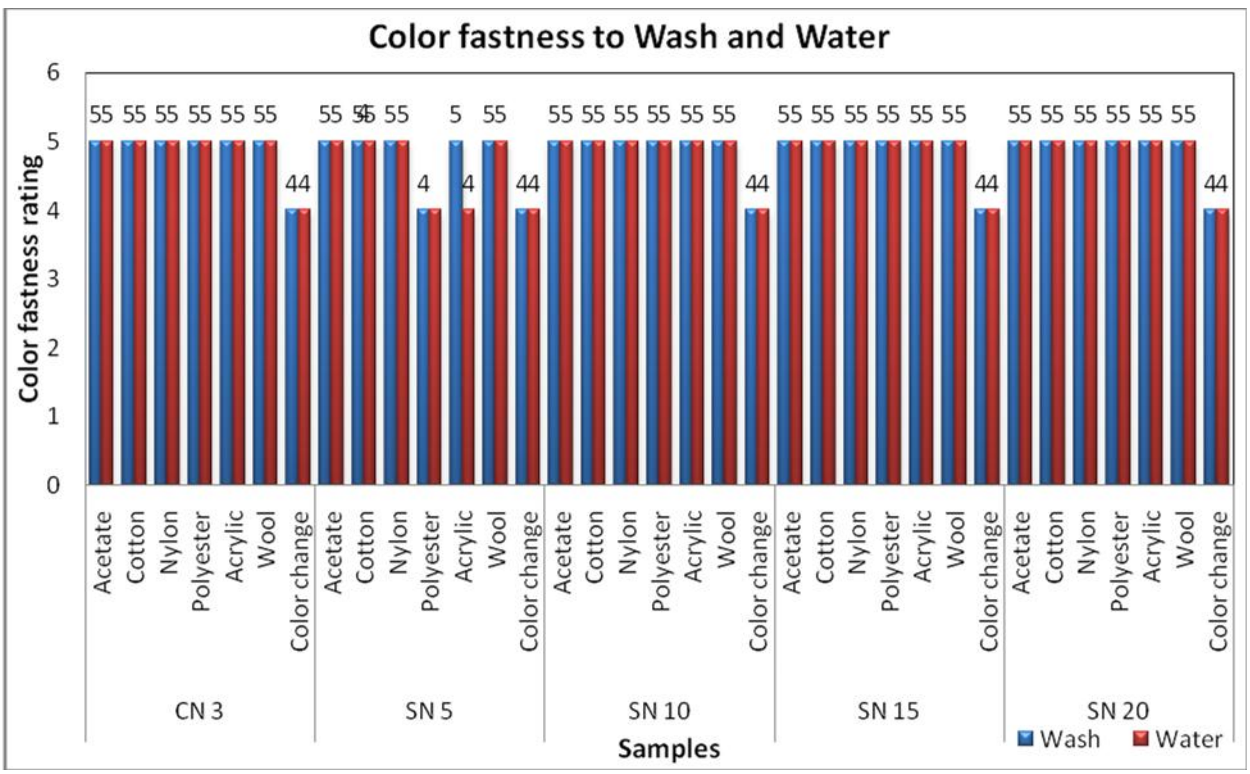

Figure.1 Effect of Soapnut Extraction wash-off on color fastness to wash and water

It is clear that all the samples have very good fastness properties, if judged solely on the basis of the change in colour. However, the staining on the adjacent multifibre fabric gives different results for the SN 5 samples. In this research, the adjacent fabric for used in fastness tests was a multifibre fabric (SDC). Multifibre fabric is composed of six different fibre types which are wool, acrylic, polyester, nylon, cotton and acetate in that order.

The results show that except polyester, all other fibre types are not stained by any of the dyed samples. This can be explained on the basis that the disperse dyes have higher affinity for polyester. Thus, any superficial dye removed which has not undergone wash-off process has more tendency to stain these fibres.

In fact, at soap nut concentrations 5% based wash-off, disperse dyes have higher affinity for polyester than other fibres and this is a major reason for the pronounced staining of polyester during the wash fastness test. Moreover, surplus dye had not been removed at the 5% concentration-based wash-off method that also leads to staining on the polyester fibre.

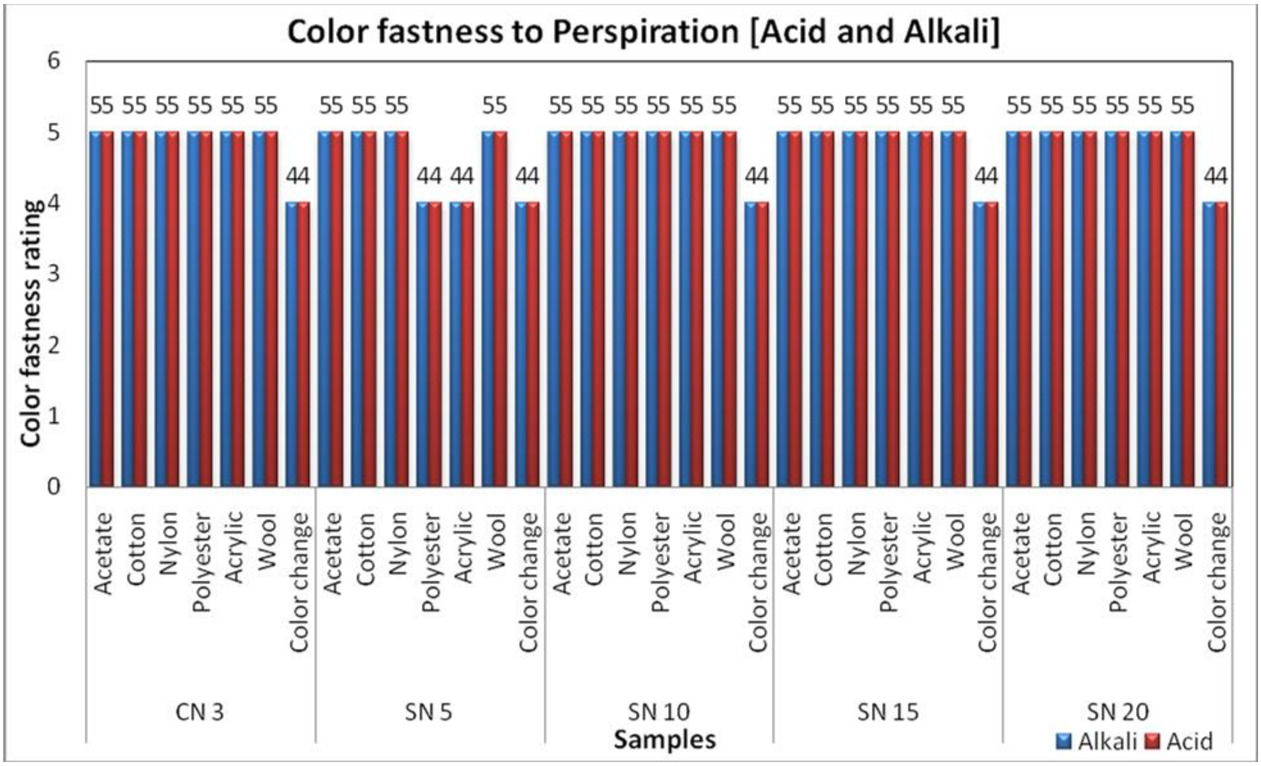

Figure.2 Effect of Soapnut Extraction wash-off on color fastness to perspiration

Fastness to acidic and alkaline perspiration of both the conventional and soapnut based extraction treated samples is shown in Figure.2. The perspiration fastness properties show similar trends to the wash fastness properties. Fastness to perspiration is also assessed by two parameters, change in colour of the dyed sample and staining of the adjacent fabric. In this case, the adjacent fabric is multifiber fabric.

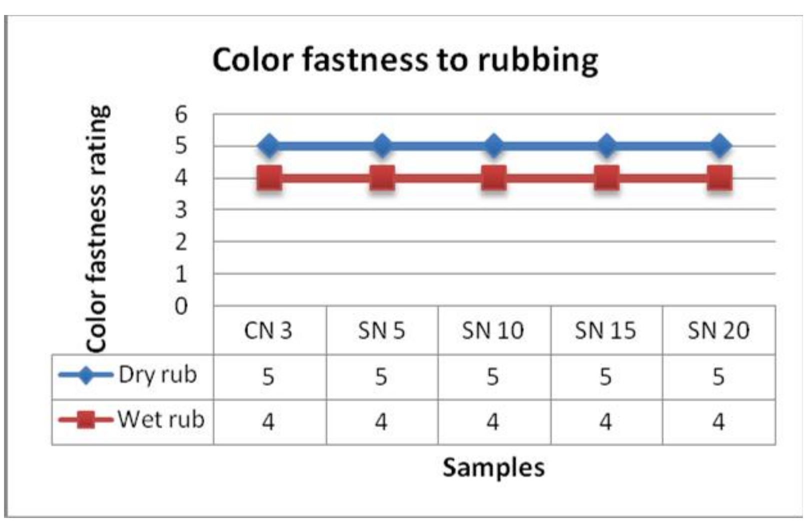

Figure.3 Effect of Soapnut Extraction wash-off on color fastness to Rubbing

The rubfastness of both the conventional and soapnut based extraction treated samples is shown in Figure.3. The results demonstrate that both methods show similar as far as rubbing is concerned. So, developed wash-off method does not result in a deterioration in the fastness properties.

4.2 Effect on Color difference

CIELAB colour differences between the soapnut extraction-based wash-off liquors compared to the standard as conventional method are summarized in Table.4. Overall results indicate that there is a change in shade of wash-off liquor with increasing soap nut concentration. The unit ΔE from this system was taken as a unit of measure of color deviations.

Table.4. CIELab colour differences of Soap nut treated fabrics

Samples	DL*	Da*	Db*	DC*	DH*	ΔE
SN 5	0.65	0.15	-0.43	0.55	-0.33	0.91
SN 10	0.27	0.05	-0.45	0.48	-0.43	0.70
SN 15	0.32	0.04	-0.36	0.37	-0.35	0.60
SN 20	-0.25	-0.03	-0.25	0.19	-0.30	0.43

With regard to colour difference, DE=1 is a useful assumption as the limit of significance of colour difference, although there is not a standard limit for DE. Lightness did not show a significant change (DL) for 5, 10 and 15% soapnut extraction-based wash-off liquors, although a change occurred at 20% concentration. This difference can be attributed to the difference of the soap nut concentration. The shade of soapnut extraction-based wash-off liquors became darker ($-\Delta L^*$) when the soap nut extraction concentration at 20%. and at that same time lower ΔE value indicates higher color removal efficiency, which clearly depicting that higher concentration will give better removal of unbound disperse dyes from the polyester fabric surface.

Again Da* and Db* value characterize the tone of the liquor color, positive values of a* and b* stand for redder and yellower tones while negative shows greener and bluer tones respectively.

4.3Weight loss

The weight loss of both the conventional based wash-off fabric and those washed-off with soapnut based extraction is shown in the Figure.4.It was observed that the weight loss% of fabric after wash-off with soapnut based extraction method was lesser than the weight loss% of the conventional method of wash-off. It is due to the hydrolytic reaction between sodium hydroxide solution and polyester that takes place at the electron-deficient carbonyl carbon of PET by attack of hydroxyl ions [32]. Hydrolytic scission of ester linkages of the polyester chains on the fibre surface takes place.

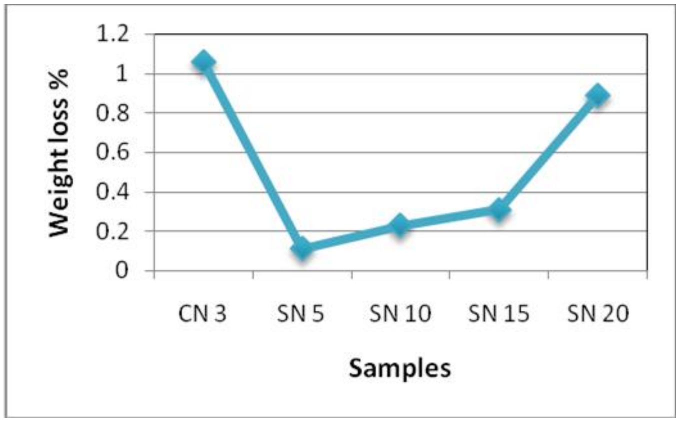

Figure.4 Effect of Soapnut Extraction wash-off on weight loss of the samples

4.4 Weight loss percentage

From the aforementioned table, it can be observed that the weight loss% of knit fabric after treatment with 5%, 10%, 15% and 20% soap nut was less than the weight loss% of the fabric after treated with synthetic chemicals. But the weight loss percentage for knit fabric, after treated with 20% soap nut was equal weight loss% found from the knit fabric after treatment with synthetic chemicals. It implied that at higher concentrations soap nut performs almost as same as or better than synthetic chemicals.

Samples	Concentration of soap nut	Weight loss %
SN 1	NaOH + Hydrose 3 + 3 gpl	1.06
SN 2	Soap nut extraction 5 %	0.11
SN 3	Soap nut extraction 10 %	0.23
SN 4	Soap nut extraction 15 %	0.31
SN 5	Soap nut extraction 20 %	0.89

4.5 GSM Measurement

As per the gsm measurement the initial grey fabric gsm was found as 170. After the after treatment with synthetic chemicals the fabric gsm came down to 162.6. On the other hand, the gsm found for different concentrations of soap nut were 167.2, 164.8, 165.6, 163.4 respectively. These results implied that the fiber loss or gsm drop was equal to synthetic chemical treated fabric in case of using 20% of soap nut after treatment sample.

<table>
<tr><th>Grey fabric GSM</th><th>GSM commercial reduction clearing (NaoH + Hydros)</th><th colspan="2">GSM after Washing with Natural detergent</th></tr>
<tr><td rowspan="5">170</td><td rowspan="5">162.6</td><td>Natural detergent concentration</td><td>GSM</td></tr>
<tr><td>Soap nut extraction 5 %</td><td>167.2</td></tr>
<tr><td>Soap nut extraction 10 %</td><td>164.8</td></tr>
<tr><td>Soap nut extraction 15 %</td><td>165.6</td></tr>
<tr><td>Soap nut extraction 20 %</td><td>163.4</td></tr>
</table>

4.6 The bursting strength

The bursting strength of the grey knit fabric was found 310Kpa while after treating with synthetic detergent came down to 300.6kpa. In case of treatment with the natural detergent better result was found, which might happen due to the loss of less fibre and strength. From the table it could be deducted that with the increase of the concentration of soap nut bursting strength decreased but was still higher than that of the fabric while treated with synthetic detergent.

Bursting strength of grey fabric	**Bursting strength of commercial reduction clearing**	**Bursting strength after Washing with Natural detergent**	
		Natural detergent concentration	Bursting Strength (Kpa)
310 kpa	300.6 kpa	Soap nut extraction 5 %	308
		Soap nut extraction 10 %	307
		Soap nut extraction 15 %	304.8
		Soap nut extraction 20 %	301.2

The bursting strength of both the conventional based wash-off fabric and those washed-off with soapnut based extraction is shown in the Figure.5.

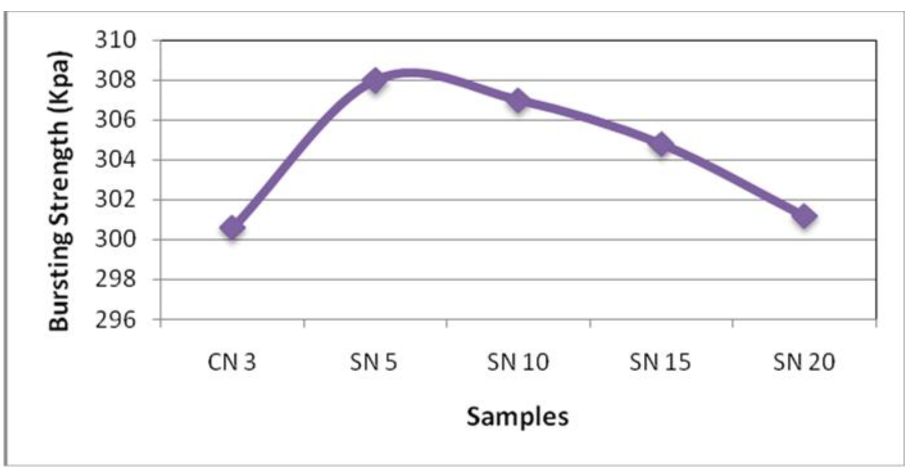

Figure.5 Effect of Soapnut Extraction wash-off on samples bursting strength

It was observed that the bursting strength of soapnut extraction-based wash-off shows higher strength than the conventional based wash-off method. It is due to the loss of lesser amount of fiber while treating with soapnut based extraction method. So, a greater number of fibres will be projected to withstand the stress caused by bursting strength tester. Moreover, with the increase of soap nut concentration was leading to decrement in strength, but strength was still higher than that of the conventional method.

4.7 Water absorbency

The water absorbency of both the conventional based wash-off fabric and those washed-off with soapnut based extraction is shown in the Figure.6.

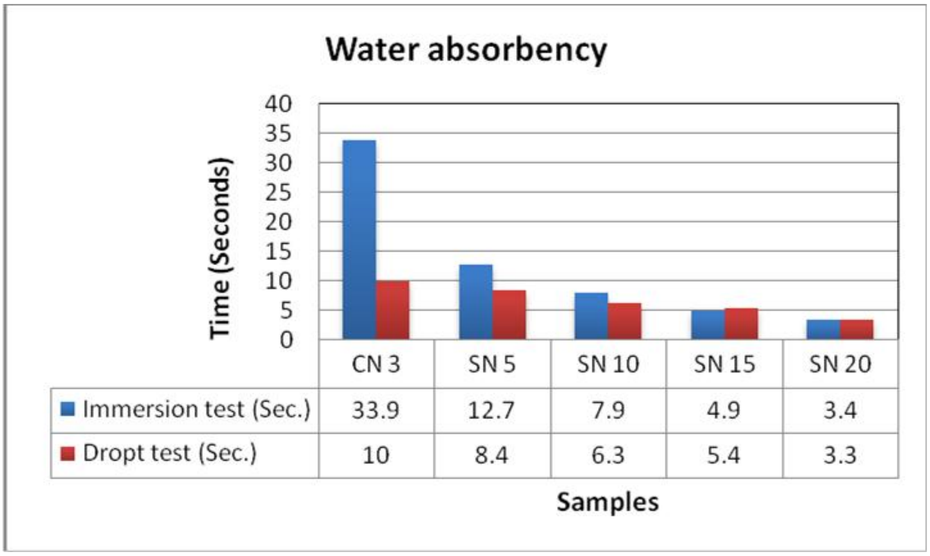

Figure.6 Effect of Soapnut Extraction wash-off on samples absorbency

The results show that the shortest immersion time observed for developed wash-off method (refer Figure. 6). This may be attributed to the OH groups arising from the surface hydroxyl groups, in the saponin moiety which are responsible for higher absorbency rate at a shorter time in the soapnut based extraction wash-off method [33].

4.8 Absorption Spectra

The amount of unfixed disperse dye for the evaluation of color removal for both conventional and soapnut extraction-based wash-off, was assessed by measuring the absorbance of the wash-off liquor in the wavelength range of 350 to 400 nm and the result is shown in the Figure.7.

The efficiency of soapnut extraction-based wash-off is comparable and in fact higher than the conventional wash-off method.

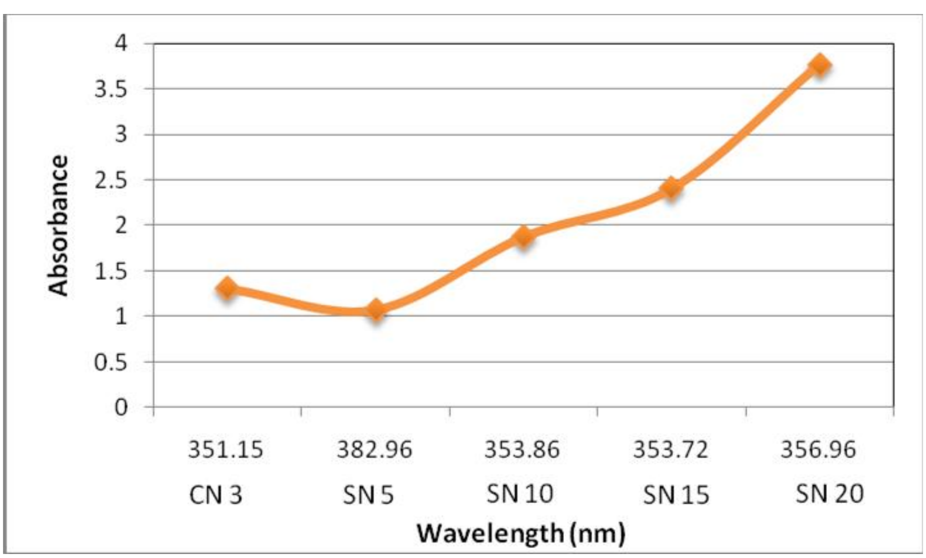

Figure.7 Absorbance Spectra of both conventional wash-off and soap nut wash-off samples

A high absorbance value indicates a greater amount of dye present in the wash-off liquor. Treatment with soap nut wash off method is most effective for the samples dyed with disperse dye giving a higher absorbance value of more than 3. A possible explanation for a higher efficiency of dye removal is due to that dye has a greater tendency to form aggregates on the fibre surface. Since soap nut composed of C=O functional groups of carboxylic acid or ester in the saponin moiety [33], it has the potential to hydrolyse the ester linkages of polyester superficially. This may facilitate the removal of the aggregated dye particles from the hydrolysed fibre surface. This results in relatively easier removal of dye aggregates present on the fibre surface as compared to conventional method.

4.9 Effluent characteristics

As previously mentioned, the use of conventional based wash-off process creates an environmentally unacceptable effluent. As an indication of the nature of the environmental impact of conventional based wash-off, Figure.8 shows the BOD, COD, TDS and pH data achieved for both the wash-off processes. Somewhat high values recorded for the conventional based wash-off process contrast, markedly, with those obtained for the wash-off treatment devised in this work.

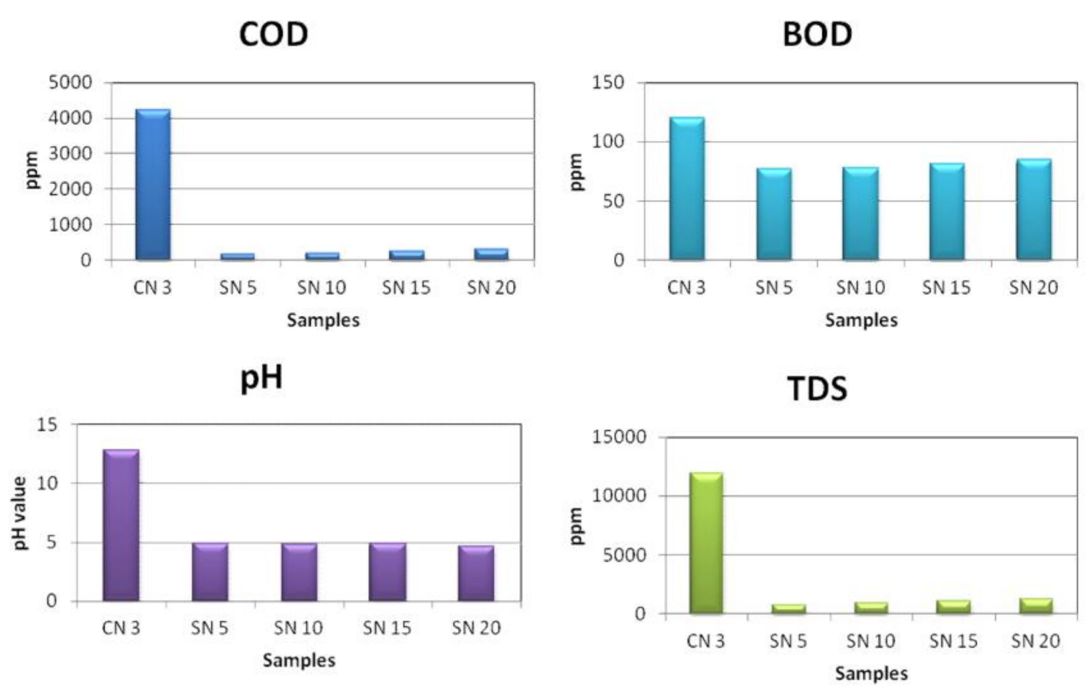

Figure. 8 COD, BOD, pH and TDS values of conventional and soap nut wash-off liquors

Environmentally, conventional method poses an additional problem due to the generation of aromatic amines in the case of azo disperse dyes. This developed soap nut extraction-based wash-off process clearly might be the replacement by negates this environmentally unacceptable problem.

Conclusion

The objectives of this research described in this article were to establish a more definitive understanding of the principles of the traditional reduction clearing process and its effect on the properties of polyester dyed with a range of disperse and, further, to explore a range of alternative clearing agents and processes which offer the potential to minimise the environmental consequences associated with the use of sodium dithionite. Polyester fabric was dyed with disperse dyes and then Soap nut were explored as alternative to sodium dithionite in the reduction clearing of dyed polyester. It has been demonstrated that the clearing of polyester dyed with the selected disperse dyes with the sodium dithionite efficient in terms of surface dye removal to an extent that provides good fastness properties, when used under the same treatment conditions as sodium dithionite. Reduction clearing with soap nut also improved the fastness and colour properties and better colour removal property to a comparable level to that of sodium dithionite.

Besides investigating the influence of reduction clearing on the fastness properties and colour properties of the dyed samples, the degree of surface dye removal was quantitatively determined by measuring the absorbance of the effluent. Generally, the degree of surface dye removal increased with the increasing concentration of soap nut which at first sight appears strange. Higher concentration of soap nut should be used in order to get the better performance compared to sodium dithionite. drop test and immersion test also exhibited that higher concentration of soapnut could ensure better absorbency performance than sodium dithionite. In physical properties fabric weight loss, GSM, bursting strength results implies that when increasing the soap nut concentration, it was slightly affected of the physical nature of the polyester fabric.

As indicators of the environmental consequence of the processes in terms of effluent production, the chemical and biochemical oxygen demand values for the residual liquors were established. The residual liquors from clearing of samples gave lower BOD and COD than sodium dithionite., although soap nut offer the environmental advantage of biodegradability. Although soap nut offer the advantage of lower environmental impact and they avoid the issue of the toxicity.

It can thus be concluded that soap nut washing agents perform better than sodium dithionite as far as the improvement of the fastness and colour properties is concerned. However, the acceptance, their cost and environmental effects have to be weighed. They also offer the advantage of sulphite and sulphate free effluent. Finally, when comparing the soap nut and sodium dithionite on the basis of their environmental issues, soapnut offers the most environmentally friendly alternative as well as better clearing efficiency.

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