

Replenishment of Pretreatment Bath- A Measure for Minimizing Effluent Load

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Abstract: Water scarcity is becoming a global concern. Textile industry uses large amount of water in their production processes, with subsequent generation of large quantity of wastewater. The major operations performed in a typical textile processing industry are desizing, scouring, mercerizing, bleaching, neutralizing, dyeing, printing and finishing. As the cost of water supplied to industry keeps increasing, textile industry needs recycling of waste water generated and conservation of water to reduce the water requirements and also dependency on other water sources. Reduction in water consumption can be done by process modification, machine modification and replenishment of the treatment baths. In this work, attempt has been made to reduce water consumption by replenishment of pretreatment baths of various processes. After replenishing the fresh bath of desizing, decrease in desizing efficiency in terms of % weight loss and TEGEWA ratings with the increasing number of replenishments was observed. Similar results were obtained in the case of the scouring efficiency in terms of % weight loss, % wax content and absorbency. From bleaching replenishments it was found that there was no remarkable difference in whiteness indices of samples from fresh and replenished baths. No significant difference was observed due to replenishments of desizing, scouring and bleaching baths, on the performance of reactive dyeing in terms of K/S value, colour difference (ΔE), washing fastness and rubbing fastness.

Keywords: Desizing, Scouring, Bleaching, Replenishment of bath, etc

I. Introduction

Global situation is, less than 3% of the world's water is fresh, the rest is seawater and undrinkable. Out of this 3% over 2.5% is frozen, locked up in Antarctica, the Arctic and glaciers, and not available to man. Thus humanity must rely on this 0.5% for all of man's and ecosystem's fresh water needs. Water scarcity is becoming a global concern. An increase of industrialization reduces freshwater sources because of the large amount of water required by manufacturing facilities and because pollutants from manufacturing processes pollute the remaining freshwater sources. Textile industry uses large amount of water in their production processes, with subsequent generation of large quantity of wastewater. Over 60 to 70% of water is used for wet processing of textile. The major operations performed in a typical textile processing industry are desizing, scouring, mercerizing, bleaching, neutralizing, dyeing, printing and finishing. Water is a vital natural reserve for sustaining life and environment, which is always thought to be available in abundance and free gift of nature. Now a days water scarcity is becoming a global concern. Due to increase in industrialization, the water quality and quantity both are now on the verge of getting depleted. Indian Textile Industry has great potential to dominate the Global Textile Market. The textile industry has to strive and put in some real work towards fulfilling such expectations. Hence, wet processing of the future should be cost effective, environmentally friendly and gentle to the textile material.

Table no. 1.1 Water usage in Textile Mills:

Sr. No.	Purpose	Water usage
1.	Steam generation	5.3 %
2.	Cooling water	6.4 %
3.	RO water for specific purpose	7.8 %
4.	Process water	72.3 %
5.	Sanitary use	7.6 %
6.	Miscellaneous and fire fighting	0.6 %

Textile sector is one of the high water consuming industries and disposes a large quantity of effluent in the environment. According to USEPA (United States Environment Protection Agency) a unit producing 20,000 lb / day of fabric consume 36000 liters of water. There is need to implement cost- effective practices for the use of water in textile industries. It has been estimated that 3.5 % of the total cost of running the industry is required for water utilization in textile industry.

Table 1.2 Total Water Consumption during Wet Processing:

Sr. No.	Process	% water consumed
1.	Bleaching	38%
2.	Dyeing	16%
3.	Printing	8%
4.	Boiler	14%
5.	Other uses	24%

The high consumption of water for textile wet processing sector emphasizes the urge for improvement of process. There should be a cut down in the chemical usage wherever possible and recycling of wastewater before releasing it into the environment. The effluent from wet processing sector, if discharged without proper treatments, is harmful to the environment. On an average about one million liters of effluent is discharged per day by an average sized textile mill having a daily production of 8000 kg.

1.1 Process Water Re-Use Options in Textile Industry: Significant savings can be made in textile processing industries by recovering and re-using of water at processes itself. Few areas where these options can be examined by the units are out lined and as follows.

- Recycling of final wash water after H₂O₂ bleaching as a wash water for second scouring step or for earlier bleaching steps.
- Reusing bleaching wash water to start another bleaching batch.
- Re-use of hot bleach water for starting optical brightening batch.
- Re-use of optical brightening wash water to start another batch of optical brightening batch.
- Final wash water of cone scouring and bleaching can be used as wash water for scouring and bleaching.
- Cold rinse water used after scouring step for sulphur black dyeing can be used for the reduction step.
- Re-use of hydrosulphite wash water for another batch of hydrosulphite bath.
- Re-use of clarified print washes water in washing blankets and screens of the print machine.

The present work deals with developing a process which can minimize the load on the effluent treatment plant by reusing the water in the wet processing sector. In accordance with this, the work was carried out to reuse each pre-treatment bath i.e. desizing, scouring and bleaching separately by standing bath method with replenishment of chemicals and water but altering the weight of substrate to maintain the required MLR. The process efficiencies after each reuse of bath were evaluated followed by reactive dyeing to check the effect of replenishment of pretreatment bath on dyeing in terms of depth. Thus efforts were made towards reusing the pretreatment bath not only to reduce the water intake and minimize the intermediate washings but also to minimize the intake of chemicals wherever possible to make existing process, 'more sustainable' and 'environment friendly process'.

II. Plan Of Experiment

2.1 Materials : 32^s Ne x 32^s Ne / 82x50 - 100% plain woven cotton fabric of gsm 85 gm/m² with 15% size add on was used in this study.

2.2 Chemicals and Auxiliaries:- Sodium hydroxide, Sodium carbonate, Sequestering agent (sodium hexameta phosphate), Detergent, Acetic acid, Hydrogen peroxide, Stabilizer, Peroxide Killer, etc all of LR grade and Amylase enzyme of commercial grade.

CI Reactive Red 2 (Procion Reactive Red M5B) dye was used for dyeing the fabric.

III. Methodology

3.1 Fresh bath Enzyme Desizing: Initially weighed grey cotton woven fabric was treated with 5gpl Amylase enzyme, 1gpl wetting agent and 10gpl salt at 60°C for 2 hrs with 1:30 MLR. After desizing the sample was given 2 hot washes followed by 1 cold wash and dried. The desizing efficiency in terms of % weight loss and TEGEWA ratings were determined.

3.1.1 Desizing Bath - First Replenishment: In this chemical addition was done in fresh used bath by 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80% and 90% chemicals. The time and temperature were maintained same as that of fresh bath and desizing was carried out. Then hot washes and cold wash were given in the same used bath. Optimum results were obtained by 60% addition of chemicals. The desizing efficiency in terms of % weight loss and TEGEWA ratings were determined.

3.1.2 Desizing Bath - Second Replenishment: Second replenishment of desizing bath was done by addition of 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80% and 90% chemicals in first replenished bath. The time and temperature were maintained same as that of fresh bath and desizing was carried out. Then hot washes and cold

washes were given in the same used bath. Optimum results were obtained by 40% addition of chemicals. The desizing efficiency in terms of % weight loss and TEGEWA ratings were determined.

3.2 Fresh bath Scouring: Initially weighed desized cotton woven fabric samples were treated with 2.5% (owf) Sodium Hydroxide, 1.0% Sodium Carbonate, and 0.5% sequestering agent (Sodium hexa meta Phosphate) at boil for 2 hours at MLR of 1:30. After scouring the samples were given two hot washes followed by one cold wash and neutralized with Acetic Acid followed by drying. The weight loss, absorbency, wax content were calculated.

3.2.1 Scouring Bath - First Replenishment: In this, initially alkali present in scouring bath was obtained by titration method. There was presence of 17% of alkali (of 2.5% owf) in the fresh scoured bath hence for 1st replenishment, addition of 83% of alkali (NaOH) was done in fresh scoured bath. Scouring was carried out as per fresh bath scouring followed by after treatments in above used fresh baths.

3.2.2 Scouring Bath- Second Replenishment: The alkali present in the 1st replenished scoured bath was calculated by titration. It was found that there was presence of 22% alkali (of 2.5% owf) in the bath hence for 2nd replenishment 78% addition of alkali (NaOH) was done. Then scouring was carried out as per fresh bath scouring and then given same after treatments in above used 1st replenished bath.

3.3 Fresh Bath Bleaching: Initially weighed desized and scoured cotton woven fabric samples were treated with 5 gpl hydrogen peroxide (45 %), 2-5 gpl Sodium silicate and 2-5 gpl sodium carbonate at 80- 85°C for 1 hr at 1:30 MLR. Residual peroxide present on the fabric was removed by using 0.25% owf peroxide killer at 85°C for 10 minutes. After this the samples were given cold wash followed by a hot wash and neutralized by using acetic acid. The samples were dried and Whiteness Indices were measured to ensure complete decolourization of coloured impurities.

3.3.1 Bleaching Bath - First Replenishment: Before using the fresh bleaching bath, the strength of peroxide in the used bath was determined by titration method. There was 28% residual peroxide present in bath hence addition of 72% hydrogen peroxide was done. Then pH was adjusted to 10.5 by using sodium carbonate and 2gpl sodium silicate was also added to the used bath. The process was carried out followed by after treatments as mentioned earlier.

3.3.2 Bleaching Bath - Second Replenishment: In this also strength of peroxide in the used bath was tested by using titration method. There was 30% of peroxide present in the bath and then the addition of 70% hydrogen peroxide was done. Then pH was adjusted to 10.5 by using sodium carbonate and 2gpl sodium silicate was also added to the used bath. The process was carried out followed by after treatments as mentioned earlier.

3.4 Dyeing: The desized, scoured and bleached cotton fabrics were dyed with cold brand CI Reactive Red 2 with 2% shade.

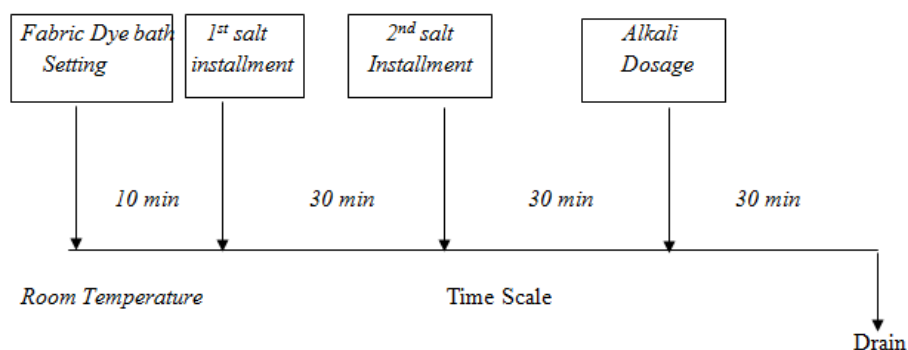


Figure 1: Reactive dyeing cycle

After dyeing, the fabric was washed with cold water followed by one hot wash (60°C), soaping with 2 gpl non-ionic soap at boil, cold wash and drying.

3.5 Testing and Analysis: The following tests were carried out for analyzing the efficiency of pretreatment combinations.

1. Weight loss by using electronic balance.
2. Testing of starch size on fabric by TEGEWA scale.

3. Absorbency by IS1560-1963 method.
4. Weight per square meter by IS1964-1970 method.
5. Residual wax content by IS4390-197.
6. Washing fastness by ISO105 C03 1982.
7. Fastness to rubbing by IS766-1956 using crock meter.
8. Whiteness Indices, K/S values and colour difference values by Macbeth colour eye 3000 Spectrophotometer.
9. Estimation of residual starch in cotton fabric after desizing by IS1967:1961.
10. Determination of alkalinity of given water sample.
11. Determination of residual peroxide in bleaching bath.

3.6 Sampling: Set of 10 samples was processed for each pretreatment combination to get the error free results.

3.6.1 Desizing Samples:

E0	E1	E2
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3.6.2 Scouring Samples

E0S0	E1S0	E2S0
E0S1	E1S1	E2S1
E0S2	E1S2	E2S2

3.6.3 Bleaching Samples

E0S0B0	E1S0B0	E2S0B0
E0S0B1	E1S0B1	E2S0B1
E0S0B2	E1S0B2	E2S0B2
E0S1B0	E1S1B0	E2S1B0
E0S1B1	E1S1B1	E2S1B1
E0S1B2	E1S1B2	E2S1B2
E0S2B0	E1S2B0	E2S2B0
E0S2B1	E1S2B1	E2S2B1
E0S2B2	E1S2B2	E2S2B2

Where - E0: Fresh bath desizing, E1: First replenishment of desizing bath, E2: Second replenishment of desizing bath.

S0: Fresh bath scouring, S1: First replenishment of scouring bath, S2: Second replenishment of scouring bath.

B0: Fresh bath bleaching, B1: First replenishment of bleaching bath, B2: Second replenishment of bleaching bath.

IV. Results And Discussion

Initially desizing of 10 samples was carried out and the desizing efficiency of all was determined in terms of weight loss and Tegewa ratings. The desized bath was replenished twice with required doses of enzymes for processing set of 10 samples each time. Similarly desizing efficiency of all samples was determined and the results are shown in table no. 4.1.

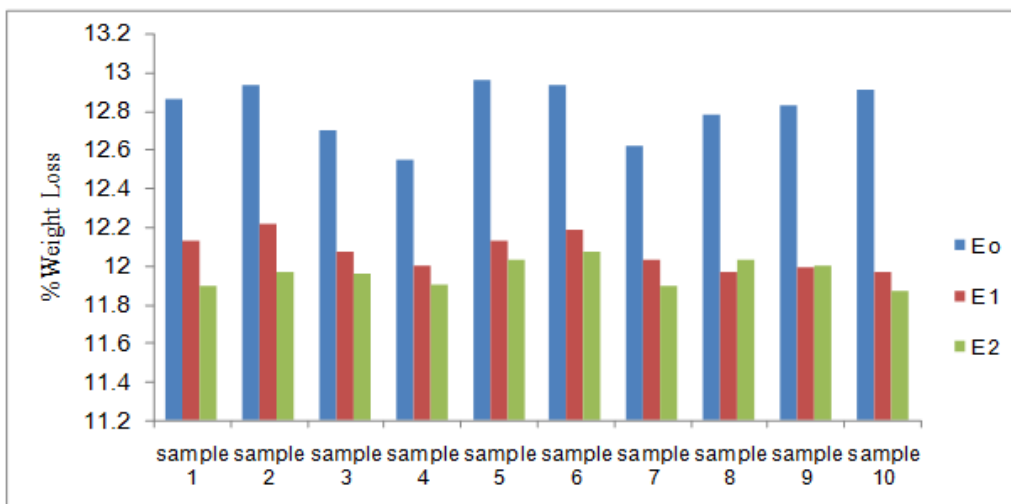
Table No. 4.1 Effect of Desizing Replenishments on Desizing Efficiency:

Sample	Sr. No.	Weight Loss		TEGEWA Rating
		% Weight Loss	Average %	
E0	1	12.86	12.807	8
	2	12.93		8
	3	12.7		8
	4	12.55		7
	5	12.96		8
	6	12.93		8
	7	12.62		8
	8	12.78		8
	9	12.83		8
	10	12.91		8
E1	1	12.13	12.075	7
	2	12.22		7
	3	12.07		7
	4	12		7
	5	12.13		7
	6	12.18		7
	7	12.03		7
	8	11.97		7
	9	11.99		7
	10	11.97		7

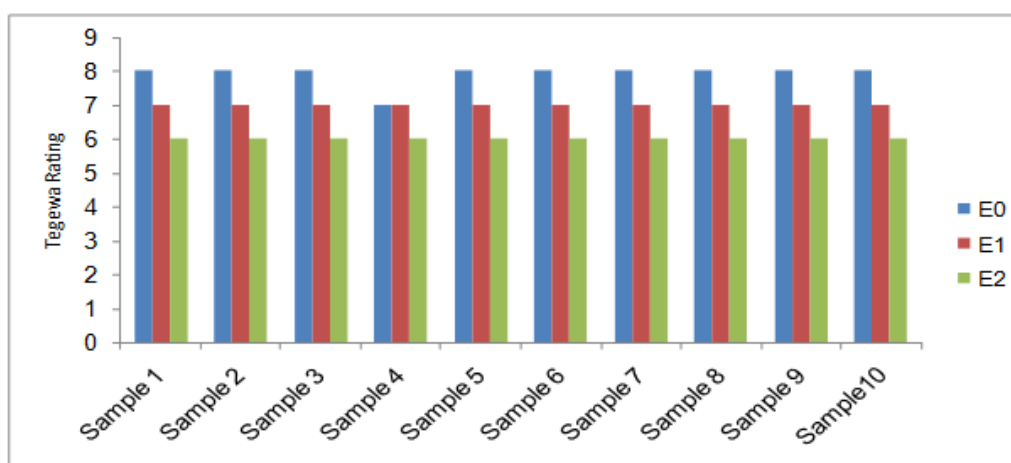
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E2	1	11.89	11.977	6
	2	11.97		6
	3	11.96		6
	4	11.9		7
	5	12.03		7
	6	12.07		7
	7	11.89		7
	8	12.03		7
	9	12		7
	10	11.87		6

Where - E0: Fresh bath desizing, E1: First replenishment of desizing bath, E2: Second replenishment of desizing bath.



Graph 4.1.1 Effect of Replenishments on desizing efficiency in terms of % Weight Loss



Graph 4.1.2 Effect of Replenishments on TEGEWA ratings after desizing

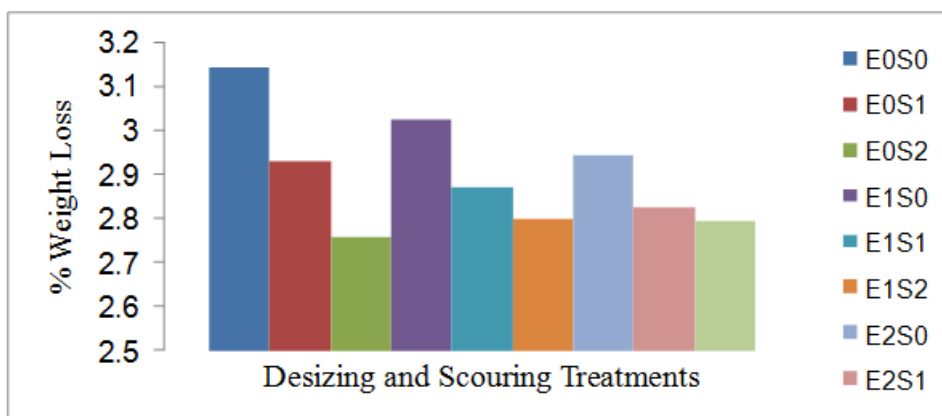
Results from the table no. 4.1 and graph no. 4.1.1 indicate that the desizing efficiency in terms of % weight loss is 12.807% for fresh desizing bath, 12.075% for 1st replenishment of desizing bath and 11.977% for 2nd replenishment of desizing bath.

Also results from table no. 4.1 and graph no. 4.1.2 show that the TEGEWA rating for fresh bath desizing was 8, for 1st replenishment of desizing bath was 7 and for 2nd replenishment of desizing bath is 6.

From above results it can be stated that, the reduction in desizing efficiency occurred may be because of increased inactivation of the enzyme and the saturation of added impurities in desizing bath due to replenishments.

Table No. 4.2 Effect of Desizing and scouring replenishments on Scouring Efficiency:

Process	Average Weight Loss %	Average Wax content %	Average Absorbancy (Sec)
E0S0 (set of 10 samples)	3.142	0.118	0.98
E0S1 (set of 10 samples)	2.928	0.34	1.117
E0S2 (set of 10 samples)	2.759	0.501	1.263
E1S0 (set of 10 samples)	3.023	0.237	1.025
E1S1 (set of 10 samples)	2.87	0.39	1.342
E1S2 (set of 10 samples)	2.8	0.46	1.362
E2S0 (set of 10 samples)	2.942	0.318	1.347
E2S1 (set of 10 samples)	2.824	0.436	1.419
E2S2 (set of 10 samples)	2.792	0.468	1.456



Graph no. 4.2.1 Effect of Deizing, Scouring replenishments on scouring efficiency



Graph no.4.2.2 Effect of Desizing, Scouring Replenishments on % Wax Content.



Graph no. 4.2.3 Effect of Desizing, Scouring Replenishments on Absorbency

Results from table no. 4.2 and graph no. 4.2.1 indicate that the scouring efficiency in terms of % weight loss for fresh bath scouring is 3.142% with fresh bath desizing and 3.023%, 2.942% after 1st replenishment, 2nd replenishment of desizing bath respectively. After 1st replenishment of scouring bath it is 2.928% and that of 2.87%, 2.824% after 1st replenishment, 2nd replenishment of desizing bath respectively. After 2nd replenishment of scouring bath % weight loss is 2.759% and 2.80%, 2.792% after 1st replenishment, 2nd replenishment of desizing bath respectively.

Results from table no. 4.2 and graph no. 4.2.2 indicate that the scouring efficiency in terms of % wax content for fresh bath scouring is 0.118% with fresh bath desizing and 0.237%, 0.318% after 1st replenishment, 2nd replenishment of desizing bath respectively. After 1st replenishment of scouring bath it is 0.34% and that of 0.39%, 0.436% after 1st replenishment, 2nd replenishment of desizing bath respectively. After 2nd replenishment of scouring bath it is 0.501% and 0.46%, 0.468% after 1st replenishment, 2nd replenishment of desizing bath respectively.

Results from table no. 4.2 and graph no. 4.2.3 indicate that the scouring efficiency in terms of absorbency for fresh bath scouring is 0.98 sec with fresh bath desizing and 1.025 sec, 1.347 sec after 1st replenishment, 2nd replenishment of desizing bath respectively. After 1st replenishment of scouring bath it is 1.117 sec and that of 1.342 sec, 1.419 sec after 1st replenishment, 2nd replenishment of desizing bath respectively. After 2nd replenishment of scouring bath it is 1.263 sec and 1.362 sec, 1.456 sec after 1st replenishment, 2nd replenishment of desizing bath respectively.

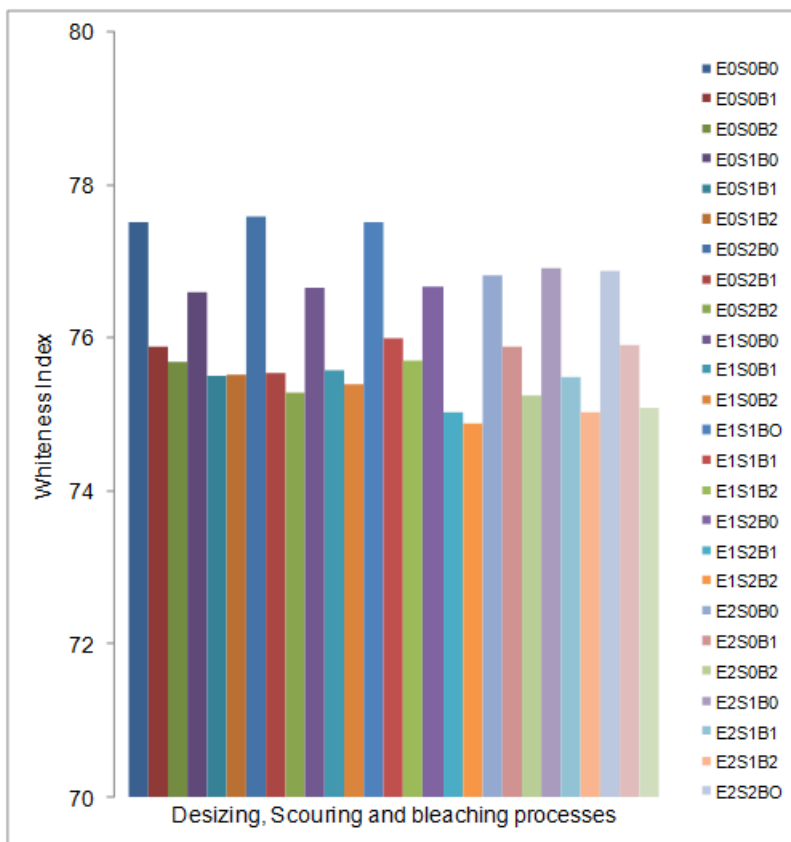
From these results it can be stated that, the scouring efficiency in terms of % weigh loss, % wax content and absorbency decreases with increased number of replenishments. This may be attributed to the fact that saturation of natural and added impurities in the replenished baths which are removed from fabric during desizing and scouring.

Table No. 4.3 Effect of Desizing, Scouring, Bleaching Replenishments on Whiteness Index:

Process	Average Whiteness Index (CIE)	Process	Average Index (CIE)	Whiteness
E0S0B0 (set of 10 samples)	77.5125	E1S1B2 (set of 10 samples)	75.7086	
E0S0B1 (set of 10 samples)	75.8909	E1S2B0 (set of 10 samples)	76.6688	
E0S0B2 (set of 10 samples)	75.6883	E1S2B1 (set of 10 samples)	75.0276	
E0S1B0 (set of 10 samples)	76.6102	E1S2B2 (set of 10 samples)	74.8952	
E0S1B1 (set of 10 samples)	75.5071	E2S0B0 (set of 10 samples)	76.8159	
E0S1B2 (set of 10 samples)	75.5298	E2S0B1 (set of 10 samples)	75.8867	
E0S2B0 (set of 10 samples)	77.5881	E2S0B2 (set of 10 samples)	75.2567	
E0S2B1 (set of 10 samples)	75.5508	E2S1B0 (set of 10 samples)	76.9166	
E0S2B2 (set of 10 samples)	75.2976	E2S1B1 (set of 10 samples)	75.4966	

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E1S0B1 (set of 10 samples)	76.6674	E2S1B2 (set of 10 samples)	75.0326
E1S0B1 (set of 10 samples)	75.5732	E2S2B0 (set of 10 samples)	76.8172
E1S0B2 (set of 10 samples)	75.4032	E2S2B1 (set of 10 samples)	75.9191
E1S1B0 (set of 10 samples)	77.518	E2S2B2 (set of 10 samples)	75.0808
E1S1B1 (set of 10 samples)	76.0023		



Graph no. 4.3 Effect of Desizing, Scouring & Bleaching Replenishments on Whiteness Index.

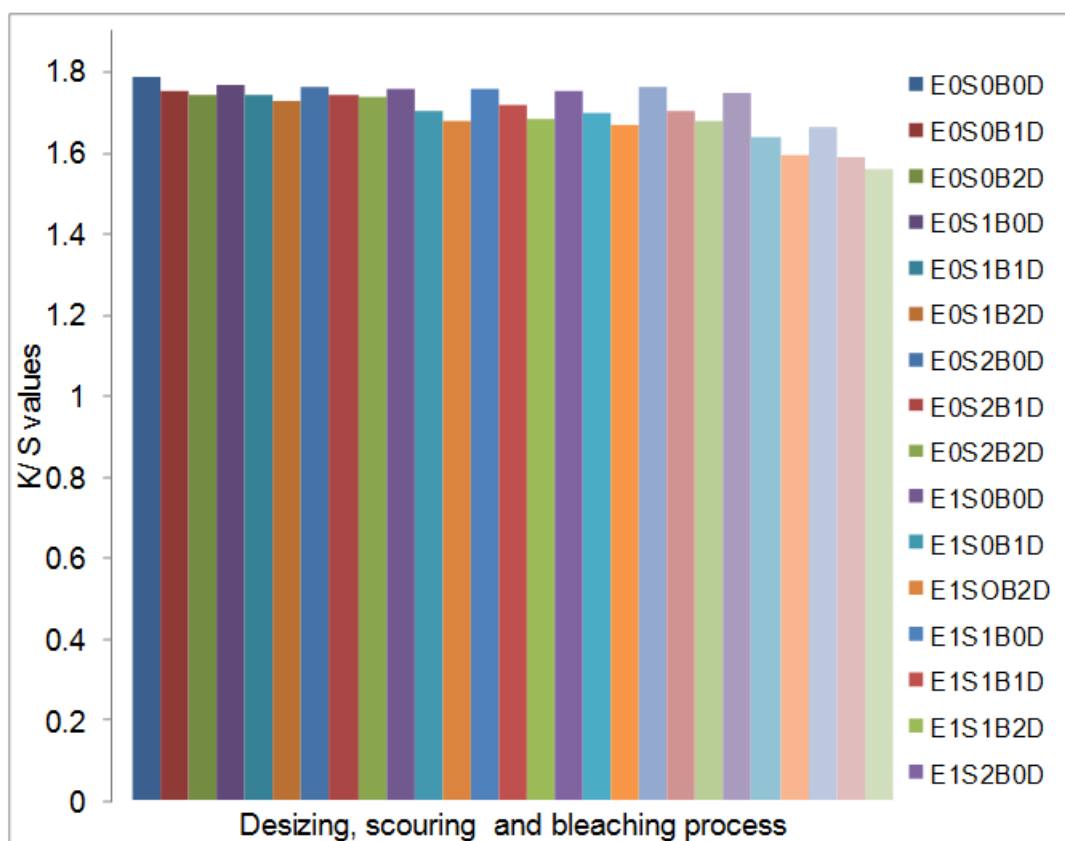
Results from table no. 4.3 and graph no. 4.3 indicate that the average bleaching efficiency in terms of whiteness index obtained after fresh bath desizing, scouring and bleaching is 77.51 and that of after first replenishment of desizing, scouring and bleaching baths is 76.0023. After second replenishment of desizing, scouring and bleaching baths it is 75.08. Thus no significant difference in the whiteness index was observed due to replenishments of desizing, scouring and bleaching baths.

From these results it can be stated that, the replenishment of bleaching can be carried out successfully.

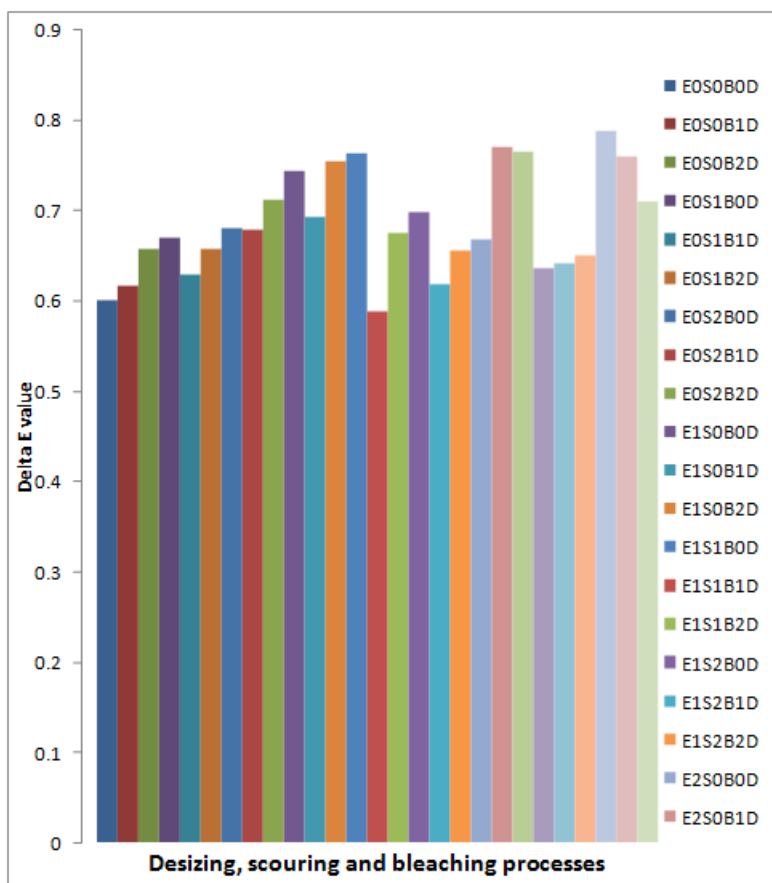
Table no. 4.4 Effect of Desizing, Scouring and Bleaching replenishments on performance of dyeing with CI Reactive Red 2 Dye:

Sample	Average K/S values for set of 10 samples	Average Colour Difference for set of 10 samples □ E	Washing Fastness		Rubbing Fastness	
			Staining on Cotton	Staining on Wool	Dry Rubbing	Wet Rubbing
E0S0B0D	1.7868	Standard	4	4-5	4	4
E0S0B1D	1.7932	0.617	4	4	4-5	4
E0S0B2D	1.7448	0.657	4	4	4	3-4
E0S1B0D	1.765	0.671	4	3-4	4	3-4
E0S1B1D	1.7438	0.629	4	4	4	3-4
E0S1B2D	1.7258	0.658	4	4	3-4	4
E0S2B0D	1.762	0.681	4	4	4	3-4
E0S2B1D	1.74	0.679	4	3-4	4	3-4
E0S2B2D	1.7382	0.713	3-4	4	3-4	3-4
E1S0B0D	1.7578	0.745	4-5	4	4	4

E1S0B1D	1.7034	0.693	4	4	4	4
E1S0B2D	1.6768	0.755	4	4	4	3-4
E1S1B0D	1.7572	0.763	4	4	4	4
E1S1B1D	1.7158	0.589	4	3-4	4	3-4
E1S1B2D	1.6816	0.676	4	4	4	3-4
E1S2B0D	1.7522	0.683	4	4	4	3-4
E1S2B1D	1.7002	0.618	4	4	4	4
E1S2B2D	1.669	0.656	4	4	4	3-4
E2S0B0D	1.7626	0.668	4	4	4	3-4
E2S0B1D	1.704	0.771	4	4	4	4
E2S0B2D	1.6776	0.765	4	4	4	4
E2S1B0D	1.7456	0.636	4	3-4	4	3-4
E2S1B1D	1.6386	0.642	4	4	4	4
E2S1B2D	1.5952	0.651	3-4	4	3-4	4
E2S2B0D	1.6604	0.788	4	4	4	3-4
E2S2B1D	1.5902	0.761	3-4	4	4	3-4
E2S2B2D	1.5598	0.711	4	4	3-4	3-4



Graph no. 4.4.1 Effect of Desizing, Scouring and Bleaching replenishments on performance of dyeing with CI Reactive Red 2 dye in terms of K/S value:



Graph no. 4.4.2 Effect of Desizing, Scouring and Bleaching replenishments on performance of dyeing with CI Reactive Red 2 Dye in terms of colour Difference (ΔE)

The results from table 4.4 and graph no. 4.4.1 indicate the K/S values of samples dyed with CI Reactive Red 2 dye after fresh bath desizing, scouring and bleaching and also after 1st and 2nd replenishments of these baths. The sample dyed after fresh desizing, scouring and bleaching is referred as standard and K/S value of the standard sample is 1.7868. The K/S value of samples dyed after 1st replenishment of desizing, scouring and bleaching baths is 1.7158 and that of after 2nd replenishment of pretreatment bath is 1.5598.

It was observed that the K/S values of dyed samples are decreasing with increasing number of replenishments which can be attributed to the decreased desizing, scouring efficiency in terms of % weight loss and absorbency.

Table no. 4.4 and graph no. 4.4.2 indicate the results of colour difference (ΔE) values. The colour difference value for sample dyed after 1st replenishment of pretreatment bath (E1S1B1D) is 0.589 and that of 0.711 after 2nd replenishment of pretreatment bath.

Similar results were obtained with combination process replenishments also from which it can be stated that the colour difference values (ΔE) of all the dyed samples lie below 1 which reveals that no significant colour difference can be observed by visual method which is the normal requirement of the customer for approval of production.

Also results from table no. 4.4 indicate the washing fastness and rubbing fastness ratings of samples dyed with CI Reactive Red 2 dye after fresh bath desizing, scouring and bleaching and that of after replenishments of each process. There is no significant difference found in the fastness ratings of washing and rubbing fastness after dyeing with CI Reactive Red 2 dye.

From the above results it can be stated that the performance of reactive dyeing for the samples processed in replenished desizing, scouring and bleaching baths is good and comparable with the standard processed fabric.

V. Conclusion

From above results and discussions it can be concluded that:

- The pretreatment baths of desizing, scouring and bleaching can be replenished.
- Desizing bath can be replenished by addition of 60% and 40% chemicals for 1st & 2nd replenishment respectively.
- With increasing number of replenishments, the efficiency of desizing in terms of % weight loss & TEGEWA rating and scouring efficiency in terms of % weight loss, % wax content & absorbency also decreases.
- No significant difference was obtained in case of whiteness index due to desizing bath replenishments and scouring bath replenishments for two times.
- No remarkable difference was observed in whiteness index due to replenishing the bleaching bath twice.
- Dyeing performance using CI Reactive Dye 2 exhibited that the K/S values of samples dyed after desizing, scouring and bleaching replenishment combinations were comparable with fresh dyed samples and colour difference (ΔE) values of all samples lie below 1 which is normal requirement of the customer for approval.
- The washing fastness and rubbing fastness properties of samples dyed after replenishment were comparable with fresh dyed samples.
- As per normal water consumption by textile industry for desizing, scouring and bleaching is 60%, due to replenishments of pretreatment baths, water consumption can be minimized upto 70% since same bath can be reutilized for processing.
- For reutilization of the desizing, scouring and bleaching baths, provision of storage tanks near the machines in processing department is recommended.
- There is greater scope to reduce the water consumption and effluent load on Effluent Treatment Plant (ETP) in textile industry by replenishing the pretreatment baths.

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