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Scopes of Acid Washing with varying Concentrations of Phosphoric Acid vis-à-vis Bleach Wash

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ABSTRACT

To acquire different fading effect and aesthetic outlook several techniques are applied in order that bleach wash is mostly used. As we know many limitations of bleach wash so our main motive is to find out an alternative method which can deliver same or better positive result than bleach wash. For this instance, we intend different concentrated acid wash and draw a comparison between acid and bleach wash on 100% cotton indigo dyed denim fabric. During our research, denim garments were developed using three parameters; bleach concentration 10 gm/L, temperature 40℃, time 20 minutes where acid concentration 0.5 to 2.5 ml/L, temperature 50° C, time 15 minutes. Due to change in acid concentration, the variation of its physical and mechanical properties like strength, weight loss, GSM, EPI & PPI and absorbency are observed. Furthermore, we focus on the color change properties such as wash, rubbing and pers piration fastness, CMC, K/S value and whiteness index. At the end of our study, we noticed that acid washed garments exhibit a promising disparity in almost all properties than bleach washed garments including same fading effects of bleach wash is accomplished by using 2% phosphoric acid in case of acid washing on denim fabric.

Keywords: denim washing, bleach wash, acid wash, phosphoric acid

1. Introduction

Denim garments have seen a revival of interest among all age groups because of their worn-out look, modified appearance, and comfort [1]. It is a warp-faced cotton fabric where weft yarn passes under two or more warp yarns fabricated under a twill design so that the lengthwise yarn is dyed with indigo dye, but the horizontal yarns remain white [2]. There are lots of technological factors

that go into making denim one of the most iconic fashion items such as huge improvements in spinning, weaving, washing, and finishing. Of these, washing plays a crucial part during denim production due to the multiple effects that consumers look for on their jeans. Without finishing treatments, denim garments are uncomfortable to wear because of the weaving and dyeing effects [3]. Moreover, industrial washing makes the fabric soft including removing dust, dirt, and hazardous materials from the garments [4]. Usually, for the washing of denim garments, bleaching, enzymatic, acid, and silicone treatments are used [7–9]. Bleach wash is mostly used to fade a higher degree of color, whereas without bleaching it is difficult to get the desired fading effect over the whole garment at one wash [10]. Shi et al. mentioned that good bleaching effects, more savings of energy and of raw materials, as well as a higher washing speed, can be achieved by using ultrasonic-assisted chlorine bleaching [11]. On the other hand, Khan et al. found that chlorine is a harsh chemical that is harmful to human health, causes corrosion to the washing machine, and destroys cotton cellulose. They stated that during bleaching, many decomposed products are produced which pass into the effluents and negatively affect the aquatic environment [12]. Faced with the problem of effluent discharge, washing industries are struggling to come up with an environmentally friendly technique that will discharge no effluent [15]. As a result, enzyme wash has become useful for consumers, retailers, fashion marketers, as well as denim apparel designers and producers, as it is eco-friendly and can improve softness, impart a new look, and promote different physical and mechanical properties of denim products [16]. But the cellulose structure is attacked by the enzyme, resulting in degradation particularly in the crystalline region, which reduces the tensile strength of the cellulose fiber and the service ability of the garments [17–18]. On the other hand, fading effects are becoming a crucial factor nowadays, which is why Pazarlioglu et al. used immobilized acid cellulases for performing an efficient fading of indigo-dyed denim fabrics, though this could not ensure the ability to prevent back-staining of the denim fabric [20]. In another work, Tarhan et al. found that back-staining can be removed with the aid of laccase washing applied after normal washing with a low concentration for a short period of time [21]. Hasan et al. mentioned that color fading of denim can be achieved by using a natural reducing agent, which is environmentally friendly. Moreover, it can reduce the amount of effluent output and of waste water; however, this technique has not been applied yet in bulk production [22]. In addition, simple softening and enzyme washing affects fabric thickness and damages the yarn [23]. However, acid wash has become popular among consumers because of its irregular fading effects on denim garments. Being a chemical process, it can strip the color from the top layer and make the surface white, while the lower layer retains the color [5]. Phosphoric acid (H_3PO_4), combined with potassium permanganate ($KMnO_4$), acts as a color-discharging agent on the surface of denim apparel [6]. Jiming Yao et al. (2013) investigated the effects of potassium permanganate decoloration on the denim shade. Their results showed that the brightness of denim was generally enhanced by increasing processing time, KMnO4, and H_3PO_4 concentrations [14–15]. So, the treatment of denim garments has been modified chemically by changing the concentration of phosphoric acid with potassium permanganate in case of acid wash, and using sodium or calcium hypochlorite in case of bleach wash. As a result, the desired fading effect of the denim garment can be achieved, and the physical and mechanical properties can be developed.

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2. Experiment

2.1 Fabric and chemicals

In the present experiment, 100% indigo-dyed, cotton-woven denim fabrics were used. These comprised GSM (grams/meter^2) 340, 3/1 warp-faced twill, and the construction of

$\frac{83 \times 55}{10 \times 12}$ x 56″ was collected randomly from the local market.

An enzyme (Cellzyme mxl-200, Dysin) was used as a de-sizing agent. Furthermore, a wetting agent, i.e. an anionic chemical (DYNOTEX MH-40) having pH of 6–7, was used to assist the quick penetration of liquor to the fiber, and a sequestering agent, hydroxyethylidene diphosphonic acid (MASQUOL P210N), was used to mitigate the hardness of the water. For washing, different concentrations of phosphoric acid (Lab Scan, Thailand) (samples 1, 2, 3, 4, and 5 are respectively treated with 0.5%, 1%, 1.5%, 2%, and 2.5% phosphoric acid in case of acid wash and sample 6 is treated with bleach.) were used separately and compared with bleach wash.

2.2 Desizing and washing

The fabrics were desized using detergent (1 g/l), Cellzyme (2 g/l), wetting agent (1 g/l), sequestering agent (2.5 g/l), and a material-to-liquor ratio of 1:12 in a sample stone washing machine (NISHO, Model-NH-WH-25, Singapore). This treatment was carried out at 60ºC for 30 minutes. After that, the liquor was dropped out. The denim fabrics were then washed with hot water at 70°C for 10 minutes. Then the samples were washed twice with cold water for three minutes each. Figure 1. shows the process curve of the desizing. Thereafter, the samples were put into a hydro-extractor (Model-NH-EX-10, Singapore) for hydro-extraction and hydro-extracting was done for three minutes. After that, the samples were left in a tumble dryer (Mel-HX-30, England) for 25 minutes.

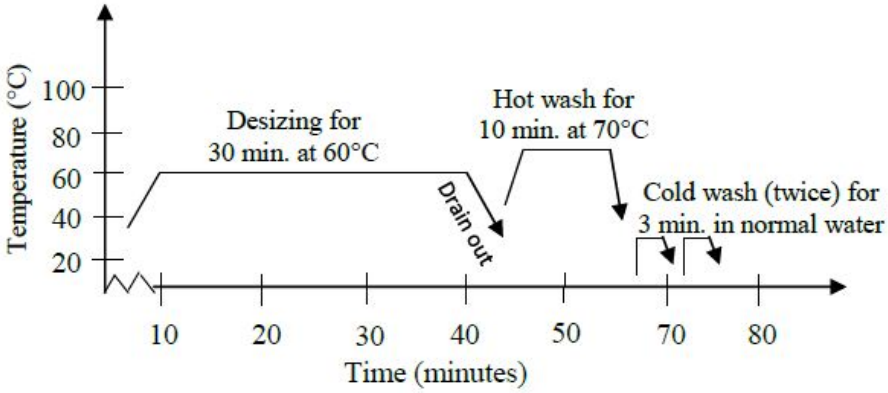


Figure 1: Desizing process curve

The samples were then taken out of the tumble dryer. The desized denim fabrics were treated using a soaked pumice stone. To make the soaked pumice stone, pumice stones were soaked with the mixture of potassium permanganate (Merck, Germany) (4g/l) and phosphoric acid (0.5 g/l) of a material-to-liquor ratio of 1:2 for 30 minutes at room temperature. Then the soaked stone was kept in air contact for 30 minutes. At first, the soaked pumice stone was put into the washing machine and run for three minutes for uniform chemical distribution of stones. After that, the pretreated garments were put into the washing machine and the machine was run for 15 minutes. After that, the treated pumice stones were unloaded from the machine. Neutralization was done using sodium meta-bi-sulfate (4g/l) and detergent (2g/l) at 60ºC for five minutes with a liquor ratio of 1:2. The liquor was then drained out. After that, cold wash was carried out each for three minutes. After the completion of the washing process, the samples were treated for three minutes in the hydro-extractor to remove the excess water. After that, the tumble dryer was used for drying the samples. Different samples were treated by changing the concentration of phosphoric acid (1 g/l, 1.5 g/l, 2 g/l, and 2.5 g/l) from the above recipe.

The same number of desized fabrics were also washed with calcium hypochlorite (10 g/l), sequestering agent (2.5 g/l), wetting agent (1 g/l), sodium carbonate (3 g/l), pH of 10.5, and a material-to-liquor ratio of 1:10 at 40°C for 20 minutes. The required amount of water and chemicals was added to the washing machine, and the machine was run for one minute for uniform chemical distribution. Then the samples were again put into the washing machine and the machine was run for 20 minutes at 40^{0}C. After that, the liquor was drained out. Then hot wash was done at 70°C for 10 minutes with acetic acid (1 g/l) and the liquor was drained out. After that, cold wash was done twice for three minutes each and the liquor was drained out. Figure 2. shows the bleach washing process curve. After the completion of the washing process, the samples were treated in the hydro-extractor to remove excess water. Then the tumble dryer was used for drying the samples. After washing and drying, the dry weight of the bleached samples was measured using an electronic balance.

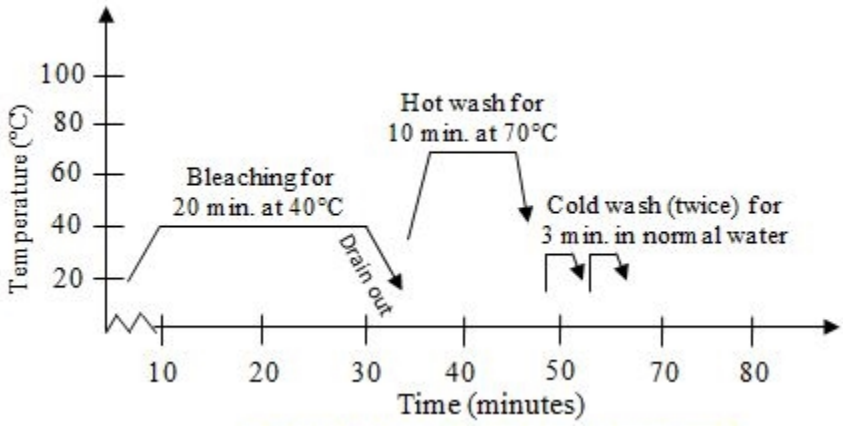


Figure 2: Bleach washing process curve

2.3 Testing methods

For testing purposes, the treated denim fabrics were conditioned in 65% RH at 20°C for 24 hours before testing according to ASTM D1776 [24]. Tensile strength (breaking force) was determined by a strength tester (Testo Metric M250-3CT, England) using the US standard grab test method according to ASTM D 5034 [25]. The weight change (%) in fabric/GSM was calculated from the difference in fabric weight before and after the treatment according to ASTM D 3776 [26]. Dimensional change/shrinkage (%) was calculated from the difference in fabric length before and after washing the garment according to AATCC test methods 135 and 150, ASTM D 2724, BS4931. GSM was calculated from the differences in the denim fabric weight between before and after the wash treatment of the denim according to ASTM D 3776 [27]. The ends per inch (EPI) and picks per inch (PPI) were measured using a counting glass. For the absorbency test, 1% direct red (Congo red) was taken in a pipette and a droplet of the solution was put on the fabric. Then the shape of the absorption area on the fabric was noted. A spectrophotometer (data color 650, USA) was used to measure the CIE whiteness indexes, color differences, and reflectance values of the samples in a D65-10 degree illuminant. The color strengths of the samples were calculated with the Kubelka–Munk equation:

$$\frac{K}{S} = \frac{(1-\mathrm{R})^2}{2R}$$

where K/S is the color strength and R represents the reflectance of the sample.

AATCC test method 8, colorfastness to crocking: The AATCC crock-meter method was used for testing the rubbing fastness test [28]. Color fastness to washing was tested according to ISO 105-C06:2010. The AATCC 15:2002 method was used for testing color fastness to perspiration [29].

RESULTS AND DISCUSSION

3.1 Effect of washing on tensile strength of the samples:

Tensile strength of a fabric is a crucial factor. It is related to the fiber, yarn, fabric properties, and the finishing process used in treating the fabric. It is a necessary performance property especially of denim fabrics, which are used frequently.

Table 1: Tensile strength of the samples

Sample no.	Force at peak (N)		Elongation at break (mm)	
	Warp	Weft	Warp	Weft
Original	761	658.75	26.336	67.2405
1	941.45	347.90	37.467	18.58
2	950.70	345.6	39.8185	18.764
3	930.10	346.55	40.448	18.159
4	950.25	327.75	37.2755	18.0165
5	882.80	397.60	38.1175	18.245
6	964.80	412	40.0895	18.567

The above table shows that, after acid and bleach wash, force increased in the warp direction compared to the original sample. But in the weft direction, the force decreased compared to the original sample. The acid-washed samples (1–4) were almost similar to the bleach-washed sample, where sample 5 was relatively lower than bleach-washed sample. It could have happened due to the yarn getting more shrinkage after washing toward the warp direction. Consequently, the higher density of the yarn helped to achieve the ability to share the loading and stand against the tensile force [30]. Again, we notice that, after both washes, the elongation moved upward in the warp direction compared with the original sample, but it declined in the weft direction compared with the original sample. Samples 2 and 3 were almost similar to the bleach-washed sample, while samples 4 and 5 declined in the warp direction compared with the bleach-washed sample. But samples 1–6 looked similar in the weft direction.

3.2 Finding weight loss of the treated samples:

The loss of weight of the washed samples during washing is an important factor. Moreover, it is related to profit and the washed fabric's quality, durability, comfortability, and other properties.

Table 2: Weight loss of samples

Sample no.	Weight Before - Washing (gm)	Weight After Washing (gm)	Weight Loss %
1	84.79	82.60	2.58
2	84.79	82.50	2.70
3	84.79	82.21	3.04
4	83.72	81.10	3.12
5	83.71	80.60	3.71
6	82.28	79.67	3.17

The above table shows that the fabrics began to lose weight with increasing concentration. This could be due to the use of as much acid as the removal of dye molecules, as well as to the loss of hairy fiber and some yarn due to the rubbing action of the pumice stone. It was observed that the weight loss of samples 6 and 4 was almost similar.

3.3 Comparison of shrinkage after washing of the samples:

Denim shrinkage after washing is closely related to the change in its structural characteristics. It is also related to the dimension of the washed sample.

Table 3: Shrinkage of different washed samples

Sample no.	Shrinkage %	
	Warp	Weft
1	-1.01	-0.75
2	-1.25	-0.80
3	-1.45	-0.89
4	-1.60	-0.92
5	-1.73	-1.21
6	-1.64	-0.96

Cotton fabrics are generally subjected to considerable tension during weaving, especially in the warp direction. Owing to subsequent finishing processes such as calendaring, this stretch increases and is temporarily set in the fabric. The fabric then remains in a state of dimensional instability [31]. It is clear from the table that, in the warp and weft directions, the shrinkage percentage may have increased due to increasing acid concentration and processing time. In this regard, samples 4 and 6 are the same.

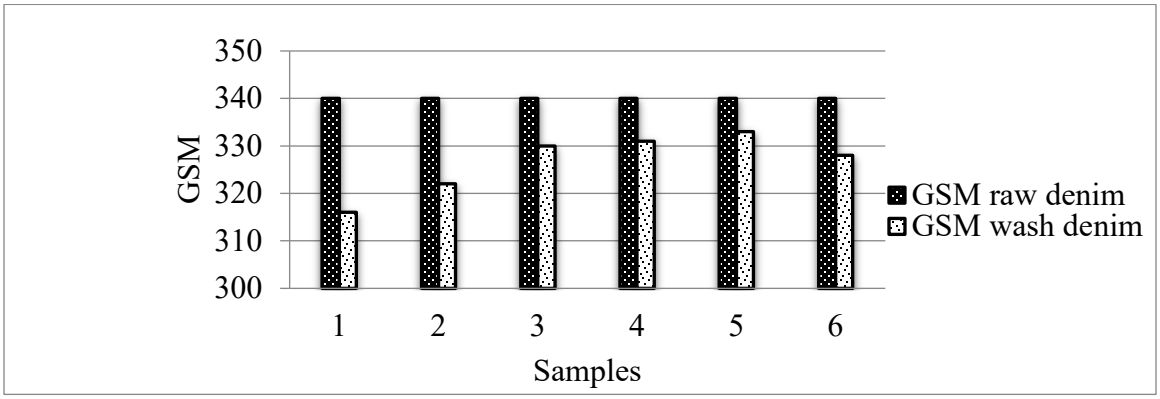


Figure 3: Graphical representations of GSM

3.4 Changes on GSM of different denim samples

Figure 3. shows the comparison of GSM values of the different concentrated washed samples. It is clear that GSM increases with the increase in acid concentration. This may be because of shrinkage due to washing. As a result of this shrinkage, EPI and PPI also increased, which increased the GSM of the fabric. However, samples 4 and 6 remained almost similar.

3.5 Comparison of the effects of washing on EPI and PPI:

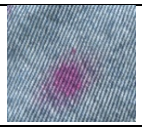
EPI and PPI are important factors for GSM and the cover factor. Due to higher PPI, the cover factor increased and the warp crimp also increased. At a higher crimp, the warp tension also went up, probably breaking the warp yarns. Thread density is also related to it [32]. In this study, with increasing acid concentration, shrinkage increased, so an increase in EPI and PPI was also found with the increase in acid concentration.

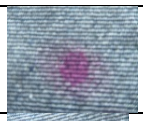
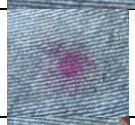
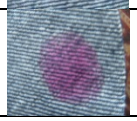
Table 4: EPI & PPI of washed samples

Sample no.	EPI		PPI	
	Before Wash	After Wash	Before Wash	After Wash
1	83	87	55	61
2		84		59
3		86		58
4		85		58
5		89		56
6		85		58

After washing was performed, the thorough wetting of the denim tended to bring the fabric back to its more stable dimensions, resulting in a contraction of the yarns. Generally, this effect is greater in the warp direction than in the weft and is known as relaxation shrinkage [31]. EPI and PPI increased more than untreated denim garments due to this relaxation shrinkage. However, samples 4 and 6 showed the same result.

Table 5: Absorbency of washed samples

Sample no.	Absorbency time (sec)	Absorbency spot
1	14	
2	12	
3	10	

4	7	
5	5	
6	6	

3.6 Effect of washing on absorbency of the samples:

The passage of a liquid through yarns and fabrics may be caused by external forces or by capillary forces. It occurs when fibers with capillary spaces in between them are wetted by a liquid. The resultant capillary forces draw the liquid into the capillary spaces. The interaction between the forces of cohesion (within the liquid) and the forces of adhesion (between the liquid and the fibers) determines whether or not wetting takes place, as well as the spread of the liquid over the surface of the solid [33]. It can be seen from the table that the absorption time gradually decreases due to increasing acid concentration. Maybe, after increasing acid concentration, the forces of cohesion dropped more than the forces of adhesion, causing absorption to move upward gradually. Nevertheless, samples 4 and 6 gave the same result.

Table 6: Comparison of CMC value of different samples

Sample	Test			
	L	C	H	CMC
Original	21.241	4.445	283.472	
1	19.076	8.880	281.648	5.78
2	19.667	8.478	281.351	5.19
3	21.593	9.653	279.878	4.62
4	22.179	8.516	280.342	4.54
5	19.084	9.061	282.170	5.38
6	19.430	9.239	281.544	5.48

3.7 Comparison of CMC value of different samples

The CMC value of naturally faded denim fabric treated with acid exhibits a higher color difference. This is because acid reacts with indigo dye and removes dye from the sample. Figure 4. shows that, after increasing acid concentration, the CMC value (1–4) decreased, but samples 5 and 6 show similar results.

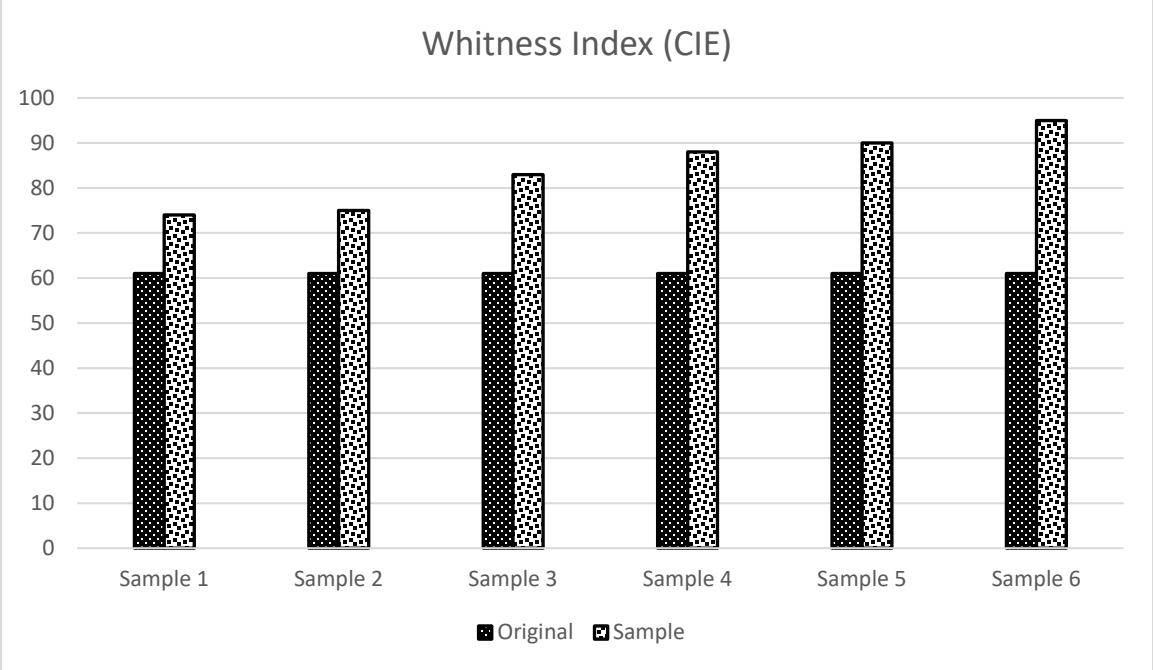


Figure 4: Whiteness index (CIE) value of different samples

3.8 Whiteness test

During chemical fading, whiteness increased more in the faded sample than in the unfaded sample [22]. The above graph indicates that, after both acid and bleach washes, the whiteness value increased more than in the original sample, but the whiteness value of the bleach-washed sample was higher than that of the acid-washed sample.

Table 7: K/S Value of treated samples

Sample no.	K/S value
1	15.7
2	17.3
3	18.1
4	18.5
5	19.5
6	18.7

3.9 K/S value test:

The K/s value is linearly related to the concentration of colorant in the medium as well as to the reflectance percentage. The darker the shade percentage, the lower the reflectance value. This shows a lower K/s value [34]. From the table, it can be gathered that the K/S value of the acid-washed samples (1, 2, 3, 4) is lower than the bleached-washed samples. But the K/S value of acid-washed sample (5) is higher than the K/S value of bleach-washed samples. Here, the dye molecule stability of sample 4 is comparatively better than other samples. For this reason, after increasing acid concentration, more dyes are removed, creating more fading and a higher reflectance value, so K/S value increases.

3.10 Comparison of fastness of the samples:

It is a quality assurance system of resistance to fading of dyed fabric when rubbed against staining. Fixation of color and depth of shade are related to it. Each color has its own fastness property to rubbing. It mainly depends on the chemical structure and fabric construction.

Table 8: Rubbing fastness grades of the samples

Sample no.	Dry	Wet
1	3/4	3
2	3	3
3	3	2
4	2/3	2
5	2	2
6	3	2

By comparing the contrast between the treated & untreated white rubbing with grey scale, evaluate the rate at 1–5. From the above graph shows that rubbing fastness between acid & bleach washed sample are almost same rating. But in case of acid wash (sample 1-5) rubbing fastness properties decreased with the increase of concentration of phosphoric acid. Maybe the higher concentration of phosphoric acid causes more dye instability in the fabric.

Table 9: Washing fastness among the samples

Multi-fiber/ Sample no.	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
1	¾	4	2/3	4	4/5	4/5
2	3	4	2/3	3	4	4/5
3	2/3	4	2/3	3	4/5	4/5
4	2	4	2/3	3	4/5	4
5	2	4	2/3	3	4	4

Sample→	1	2	3	4	5	6
Fabric	4/5	4	4/5	5	4/5	4/5

3.11 Washing fastness of treated samples:

Phosphoric acid ($KMno_4$ + H_3Po_4 + H_2O) solution applied on the garment, which oxidizes the cellulose and color, is partially removed according to the intensity of the solution. Chemicals are used in acid-washing jeans. The process strips off the color of the top layer, leaving the white fabric exposed. The above table shows that the wash fastness rating of acid-washed samples (1, 3, 5) tends to be similar to that of bleach-washed samples. But the wash fastness rating of sample 4 shows a better result than bleach-washed samples.

3.12 Comparison of perspiration of the samples:

Continuous contact with human perspiration has an impact on the fastness of some washed fabric. When the fabric is subjected to alkaline or acidic perspiration, the tone and depth of the dyed shade is affected. This test is intended to determine the resistance of color of the washed fabric to the action of alkaline perspiration.

Table 10: Perspiration of different washed samples

Multi-fiber/ Sample no.	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
1	5	4	4	4/5	4/5	4
2	5	5	4/5	5	4/5	4/5
3	4/5	4/5	4/5	4/5	5	4/5
4	4/5	4/5	4	4/5	4/5	4
5	5	4/5	4	4	5	4
6	5	5	4/5	4/5	4/5	5

Sample→	1	2	3	4	5	6
Fabric	4/5	4	4/5	4/5	4/5	4/5

The above table shows that perspiration fastness of acid- and bleach-washed samples gives an almost similar rating, except for sample 2.

4. Conclusion

It has been found that a change in acid concentration has a noticeable effect on the physical and mechanical properties of denim garments. The following observations were made:

- Strength and elongation at the break moved upward in the warp direction, while both moved down toward the weft direction. Higher shrinkage led to higher yarn density, helping to increase strength as well as elongation at the break to the warp direction.
- Weight loss happened due to acid concentration including the rubbing action between the pumice stone, washing machine, and denim garments.
- Shrinkage made the fabric more compact with higher EPI and PPI.
- In case of high EPI and PPI after treatment, the GSM of the treated garments showed an upward trend.
- Acid concentration destroyed the fabric's absorbency property. It seemed that the fabric absorbed liquid within a short time after increasing acid concentration.

- CMC value dropped because highly concentrated acid removed more dye, consequently increasing the whiteness index and the K/S value.
- In case of color fastness, a comparatively better result was shown by rubbing, wash, and perspiration fastness.

To conclude, there are obvious reasons why acid wash is popularly used. Acid-washed garments exhibit a significant difference in almost all properties compared to bleach-washed garments. The sample, tested with 2% phosphoric acid, looked almost the same as bleach-washed samples. Hence, the desired fading effects of bleach wash can be achieved by using 2% phosphoric acid in case of acid washing of denim fabric, and the degree of weight loss, strength, shrinkage, GSM, K/S value, and wash fastness are significantly better than in bleach wash. Moreover, EPI and PPI, rubbing fastness, and perspiration fastness between acid- and bleach-washed samples showed almost the same consequence. Thus, the same fading effect of bleach wash can be achieved by using 2% phosphoric acid in case of acid wash of denim fabric.

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