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Optimize the Amount of Phosphoric Acid (H_3PO_4) to Obtain Maximum Fading and Mechanical Properties of the Denim Fabric during Acid Wash

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ABSTRACT

Indigo dyed denim garment washing has recently become an essential method for creating stylish products of leisure wear. To achieve different fading effects and an aesthetic look, several techniques are used. In this research, acid wash was performed in the presence of phosphoric acid (H_3PO_4) (0.5-2.5gm/l) to obtain better fading effect as well as physical properties and draw a conclusion of optimize acid amount. Owing to the change in acid concentration, variations in the fabric's physical and mechanical properties like strength, weight loss, GSM, EPI, PPI, dimensional stability and absorbency were observed. In addition, color abrasion values such as Color Measurement Committee (CMC), Color strength (K/S), yellowness, whiteness values as well as color fastness properties of the treated fabrics were also assessed evaluated. According to the results of the experiments, 2gm/l phosphoric acid was the most successful in stripping dyes from denim fabric, resulting in an excellent acid washing action with the least amount of fiber hydrolysis.

Keywords: denim washing, acid wash, phosphoric acid, physical properties, fading effect

1. Introduction

Denim garments have been seen as a revival of interest among all the age groups because of their worn-out look, modified appearance, and comfort (Maryan & Montazer, 2013). 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(Grieve, Biermann, & Schaub, 2006; Özkan & Kaplangiray, 2017). 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(M. D. Hossain, Rashid, Kafi, & Sarker, 2014; Muhammad Abdur Rashid, Hossain, Islam, & Nakib-Ul-Hasan, 2013; Muhammad Abdur Rashid, Hossain, Kafi, & Yesmin, 2014). Of these, washing plays a crucial part during denim garments production due to the multiple effects that consumers look for on their jeans (Bhattacharjee, Dhar, Islam, & Rashid, 2019; Kan, 2015). Without washing treatments, denim garments are uncomfortable to wear because of the strong, stiff, and hard wearing woven fabric (Hackett, 2015). Moreover, industrial washing makes the fabric soft including removing dust, dirt, and hazardous materials from the garments(M. J. Hossain, Hoque, & Rashid, 2020; Mondal & Khan, 2014; Muhammad Abdur Rashid, Hoque, & Hossain, 2020; Muhammad Abdur Rashid, Miah, Siddique, & Hannan, 2020). Usually, for the washing of denim garments, bleaching, enzymatic, acid, stone, normal wash etc. are applied(Md Saiful Hoque, Hossain, Imtiaz, Das, & Rashid, 2018; Md. Saiful Hoque, Rashid, Chowdhury, Chakraborty, & Haque, 2018; Kan, 2015; Muhammad Abdur Rashid, Hoque, et al., 2020). Among various types of washing, acid wash is one of the best techniques to improve the faded looks of the denim fabrics as well as decreasing the water consumption (Arjun, Hiranmayee, & Farheen, 2013). 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. During acid washing, potassium permanganate (KMnO4) is mostly applied to fade of denim garments. Under acidic conditions, potassium permanganate acts as an oxidant, producing active oxygen [O], which can fade denim garments. (Aleboyeh, Olya, & Aleboyeh, 2009; M. J. Hossain et al., 2020). Equation (1) is used to illustrate the reaction:

$$2MnO_4^- + 6H^+ \rightarrow 2Mn^{2+} + 3H_2O + 5[O] \quad (1)$$

Compared to sodium hypochlorite and hydrogen peroxide, the fading of denim garments by potassium permanganate combined with phosphoric acid causes less physical damage. (Choudhury, 2006). The concentration of phosphoric acid (H3PO4) plays an important role in KMnO4 oxidation, which further affects the fading performance of denim garments (M. J. Hossain et al., 2020; Yao & Wei, 2013). Phosphoric acid (H3PO4), combined with potassium permanganate (KMnO4), acts as a color-discharging agent on the surface of denim fabric (Aleboyeh et al., 2009; M. J. Hossain et al., 2020). Jiming Yao et al. examined the impact of potassium permanganate on the color of denim garments. According to their findings, the brightness of denim fabric was significantly improved by increasing the duration of treatment, KMnO4 and H3PO4 content (Yao & Wei, 2013). X. R. Xu et al. investigated the impacts of potassium permanganate on the color removal of dyes and textile effluent, and discovered profound impacts on the color removal efficiency (Xu, Li, Wang, & Gu, 2005). LI Jin-peng et al. examined the de-coloration of five kinds of azo dyes-azo blue, alizarie red, acidic chrome blue K, eriochrome black Y and arsenazo by potassium permanganate and they also showed that the de-coloration was rapid and highly efficient with de-coloration rate which is over 90% in 30 minutes(LI, LI, GAO, WEI, & JIA, 2003). S.M. Mortazavi et al. researched removal of natural color from cotton fibers by potassium permanganate and they had been identified noteworthy impacts on the removal of color(Mortazavi, Ziaie, & Khayamian, 2008).

In this investigation, the fading of indigo colored denim garments was manipulated chemically by changing the concentration of phosphoric acid with potassium permanganate and optimizing the phosphoric acid concentration. However, no previous research has looked into the impacts of H3PO4 fading on color shade and physical properties. Therefore, the desired faded appearance as well as optimized physical and mechanical properties of the denim fabric can be attained.

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 specification of $\frac{84 \times 55}{10 \times 12}$ x56″ 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) (0.5- 2.5gm/l) were used.

2.2 Desizing and washing

In a prototype stone washing machine (NISHO, Model-NH-WH-25, Singapore), the denim fabrics were desized with detergent (1 gm/l), Cellzyme (2 gm/l), wetting agent (1 gm/l), sequestering agent (2.5 gm/l), and a material : liquor ratio of 1:12. The treatment was carried out at 60^0C for 30 minutes and dropped out the liquor. After that hot wash was performed at 70°C for 10 minutes. Further cold wash was carried out two times for three minutes each. Subsequently, the samples were put into a hydro-extractor (Model-NH-EX-10, Singapore) to remove excess water for three minutes. After that, the samples were left in a tumble dryer (Mel-HX-30, England) for 25 minutes.

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) (4gm/l) and phosphoric acid (0.5gm/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. Then, the treated pumice stones were unloaded from the machine. Neutralization was done using sodium meta-bi-sulfate (4gm/l) and detergent (2gm/l) at 60^0C for five minutes with a liquor ratio of 1:20. 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 gm/l, 1.5 gm/l, 2 gm/l, and 2.5 gm/l) from the above recipe.

2.3 Characterizing the denim fabric

The treated denim fabrics were conditioned for 24 hours in 65 % RH at 20°C before being tested according to ASTM D1776. Tensile strength and elongation of the samples were evaluated by a strength tester (Testo Metric M250-3CT, England) using the ASTM D 503 standard grab test method. According to ASTM D 3776, the weight change (%) in fabric/GSM was estimated. The change in fabric length before and after washing the garment was used to compute dimensional change/shrinkage (%) according to AATCC test methods 135 and 150, ASTM D 2724, and BS4931A counting glass was used to determine the ends per inch (EPI) and picks per inch (PPI). For the absorbency test, a droplet of 1 % direct red dye (Congo red) solution was placed on the fabric using a pipette. Then the shape of the absorption area on the fabric was noted. In a D65-10 degree illuminant, the CIE whiteness index, color variations, and yellowness values of the samples were measured using a spectrophotometer (data color 650, USA). The Kubelka–Munk equation (2) was used to calculate the color intensity of the samples:

$$\frac{k}{s} = \frac{(1-R)^2}{2R} \qquad (2)$$

Where, K/S is the color strength and R signifies the reflectance value of the sample. The AATCC crock-meter method was used for testing the rubbing fastness test. Moreover, ISO 105-C06:2010 was used to assess color fastness to washing. The AATCC 15:2002 method was used for testing color fastness to perspiration.

3. Results and discussion

3.1 Effect of washing on tensile strength and elongation of the samples

The tensile strength of a garment is an important consideration. It has to do with the fiber, yarn, fabric qualities, and the finishing method used for fabric treatment. This is a crucial performance characteristic, particularly for denim materials, which are often employed.

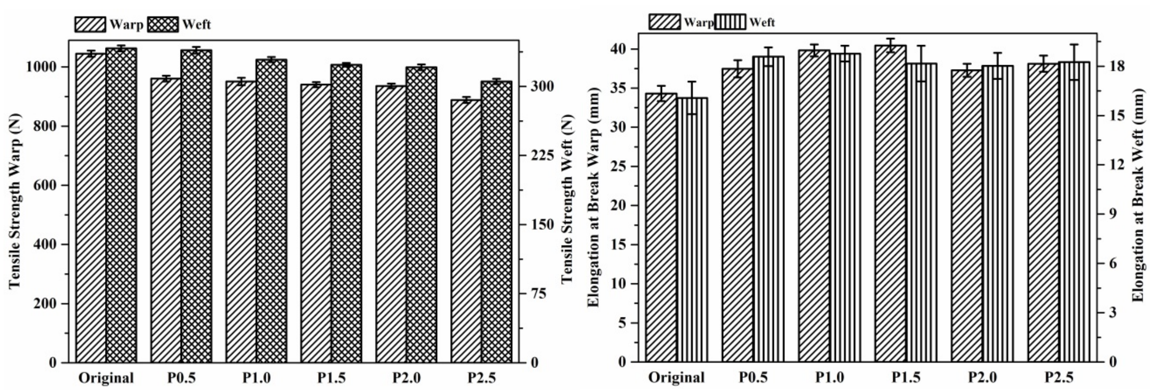


Figure 1. (a) Tensile strength (warp and weft) (b) Elongation properties of washed samples

The data of differently washed for denim fabric is revealed in Figure 1(a). After washing, the tensile strength of samples (warp and weft) tends to be weaker than the original. According to the Figure 1(a), the fabric tensile strength (warp and weft) tends to be weaker corresponding to the increment of the amount of phosphoric acid (H_3PO_4). Furthermore, the strength loss of the treated samples was almost identical during using the amount of phosphoric acid (0.5-2gm/l), after that, rapidly decreased in case of 2.5gm/l of phosphoric acid. The possible explanation could be the potassium permanganate is being more oxidative under 2.5gm/l acidic conditions generates more active oxygen [O] that could make denim decoloration as well as fiber hydrolyzed. Figure 1(b) depicts the elongation at break of treated denim fabric. Figure 1(b) also shows that the treated denim sample has a higher percentage of elongation at break (warp and weft) than the original. The washed sample had a larger elongation at break as it was totally relaxed and processed with a higher yarn crimp percentage. The absence of a sizing element from the original denim fabric could be another factor for the washed sample's higher elongation to break.

3.2 Finding physical specification changed of the treated samples

Table 1 compares the physical attributes of faded denim samples to those of the original. The shrinkage of denim garment after washing is deeply connected to the change in its structural properties. This is also correlated to the dimensions of the wash sample. Cotton fabrics are frequently exposed to severe stress while weaving process, particularly in the warp direction. Due to continuous finishing processes like calendaring and compacting, this pressure increases and is temporarily fixed in the fabric. The fabric then stays in a dimensionally unstable state. The shrinkage % in the warp and weft directions may have increased with the increasing of acid content, as shown in Table 1. When the denim fabric was washed extensively in various processes, it tended to return to its more stable dimensions, causing the yarns to shrink. In most cases, the rate is significantly higher in the warp direction than the weft direction. This is referred to as "relaxation shrinkage" (Nayak et al., 2016). According to Table 1, EPI (ends per inch) stayed unchanged, whereas PPI decreased due to relaxation shrinkage for different wet washed samples. In this investigation, shrinkage was observed when the acid content was raised and an increase in PPI was also observed as the acid content was increased.

Table 1. The physical attributes of a denim fabric that has been washed

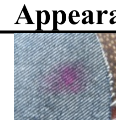
Sample Description	Shrinkage (%)	Fabric weight (GSM)	EPI & PPI (ends/ inch)	Weight Loss (%)
Original	0	340	84×54	0
P0.5	L=1.01 W=0.75	336	84×55	2.58
P1.0	L=1.25 W=0.80	333	84×56	2.70
P1.5	L=1.45 W=0.89	330	84×56	3.04
P2.0	L=1.60 W=0.92	331	84×56	3.12
P2.5	L=1.73 W=1.21	330	84×58	3.71

The weight loss of treated samples throughout the washing process is a significant impact. Moreover, it is related to profit and the washed fabric's quality, durability, comfortability, and other properties. Table 1 demonstrates that when the acid content increased, the fabrics gradually lost weight. It could be attributed to the application of as much acid as necessary to remove the pigment molecules, as well as the pumice stone's rubbing effect hydrolyzing the hairy fiber and some thread. It was observed that the weight loss was more in case of 2.5gm/l acid.

3.3 Washing effect on sample absorbency

External forces or capillary forces can allow liquid to move through yarns and fabrics. It happens when a liquid wets fibers with capillary gaps between them. The liquid is drawn into the capillary gaps by the capillary forces that arise. The interaction of cohesion forces (within the liquid) and adhesion forces (between the liquid and the fibers) controls whether or not wetting occurs, as well as the spread of the liquid over the solid's surface (Petrulyte & Baltakyte, 2009). As can be observed in table 2, the absorption time is shorter as the acid content increases. Maybe, after increasing acid concentration, the forces of cohesion dropped more than the forces of adhesion, causing absorption to move upward gradually.

Table 2. Absorption performance of treated samples

Sample Description	Absorbency Time (sec)	Visual Appearance
P0.5	14	
P1.0	12	
P1.5	10	
P2.0	7	
P2.5	5	

3.4 Fading properties of treated denim fabrics

Table 3 shows the results of treated denim fabric samples compared to original samples in terms of L, C, H, and CMC values obtained using a spectrophotometer. The parameters are illuminated by D65 and evaluated by 10º angles.

Table 3. Spectrophotometer data of treated samples

Sample Description	Test Results			
	L	C	H	CMC
Original	21.241	4.445	283.472	0
P0.5	19.076	8.880	281.648	5.18
P1.0	19.667	8.478	281.351	5.23
P1.5	21.593	9.653	279.878	5.32
P2.0	22.179	8.516	280.342	5.54
P2.5	19.084	9.061	282.170	5.38

The CMC values of original to acid wash in consist of varied H_3PO_4 conc. treated fabrics were 5.18; 5.23; 5.32; 5.54 and 5.38 correspondingly. The value of CMC was increased; this means that the shade deviations of original to treated samples increased when the H_3PO_4 concentration was raised, however the value of 2gm/l indicated the highest value. The allowable explanation could be the creating more active oxygen [O] of potassium permanganate at 2gm/l of H_3PO_4 that would be released more indigo dye from the fiber by oxidation reaction.

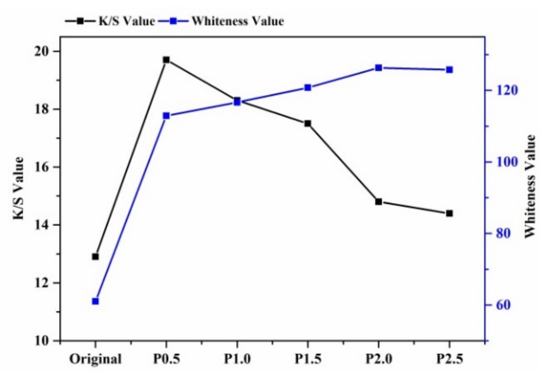


Figure 2. K/S and Whiteness index value of faded samples

3.5 K/S and Whiteness values of the treated samples

The K/S value is proportional to the colorant concentration in the fabric. The K/S and whiteness index values of the treated samples are shown in Figure 2. From the results, it is noted that the K/S values were decreased with increasing the conc. of H_3PO_4. Generally speaking, the higher K/S value, the higher the color yield will be, and it can conclude that a paler shade was obtained after different treatment due to enhance removing dye molecules by active oxygen [O] of the potassium permanganate. In case of whiteness index, it could be also increased with increasing the conc. of H_3PO_4.

Table 4. Rubbing fastness grades of the samples

Sample Description	Dry	Wet
P0.5	3/4	3
P1.0	3	3
P1.5	3	2
P2.0	2/3	2
P2.5	2	2

3.6 Rubbing 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. By comparing the contrast between the treated and untreated white rubbing cloth with grey scale, evaluate the rate at 1–5. From the Table 4 shows that the rubbing fastness properties of the treated samples are decreased slightly with the increase of concentration of phosphoric acid. It might be the higher concentration of phosphoric acid because of more dye instability in the fabric due to the active oxygen [O] of the potassium permanganate.

Table 5. Wash fastness performance of the treated samples

Sample Description	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
P0.5	3/4	4	2/3	4	4/5	4/5
P1.0	3	4	2/3	3	4	4/5
P1.5	2/3	4	2/3	3	4/5	4/5
P2.0	2	4	2/3	3	4/5	4
P2.5	2	4	2/3	3	4	4

3.7 Wash 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. 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 tends to be practically identical

Table 6. Perspiration fastness performance of the treated samples

Sample Description	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
P0.5	5	4	4	4/5	4/5	4
P1.0	5	5	4/5	5	4/5	4/5
P1.5	4/5	4/5	4/5	4/5	5	4/5
P2.0	4/5	4/5	4	4/5	4/5	4
P2.5	5	4/5	4	4	5	4

3.8 Comparison of perspiration fastness of the samples

The fastness of certain treated fabrics is affected by prolonged contact with human perspiration. The brightness and intensity of the colored shade are modified when the fabric is exposed to alkaline or acidic perspiration. The purpose of this test is to see how resistant the color of the washed fabric is to the action of alkaline perspiration. The perspiration fastness of acid washed samples is practically identical, as shown in Table 6.

4. Conclusions

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

- Strength loss of the treated fabrics was practically identical when using 0.5-2gm/l of phosphoric acid, but rapidly dropped when using 2.5gm/l of phosphoric acid due to the generation of more active oxygen [O] of potassium permanganate that could make denim de-coloration as well as fiber hydrolization. Additionally, the treated denim exhibits a larger percent of elongation at break (warp and weft) than the original.
- Weight loss occurred due to the acid concentration including the rubbing action between the pumice stone, washing machine, and denim garments.
- The percentage of shrinkage increased as the acid content rises. Owing to relaxation shrinkage, EPI (ends per inch)

stayed consistent, whereas PPI (picks per inch) altered for treated samples.

- As the acid content rises, the absorption time decreases significantly.
- CMC value as well as K/S value dropped because highly concentrated acid removed more dye, consequently increasing the whiteness index value.
- Rubbing, wash, and perspiration fastness all produced nearly identical results in terms of color fastness.

It was found that 2gm/l phosphoric acid was the most successful at removing color from denim fabric, resulting in an excellent acid washing effect with the least amount of fiber hydrolysis.

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