Cold-Pad-Batch Bio-Pretreatment of Cotton Woven Fabrics: A Case Report on Industrial Trials

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Abstract
In spite of technologically proved bioscouring using pectinase that substitutes for conventional alkaline scouring, the batch-wise bioscouring in a single bath is sometimes not welcome to certain companies where continuous or cold-pad-batch alkaline pretreatment for cotton woven fabrics used to be done with pad steamer or pad-batch equipments because of low productivity of single batch-wise process as well as the company's existing unique facility. To increase the applicability of enzymatic bioscouring, two industrial trials of bio-pretreatment by cold-pad-batch method were carried out in two different sites using their own pad-batch facilities. Simultaneous enzymatic desizing and scouring for cotton and cotton-blend woven fabrics were carried out using amylase of low-temperature type and alkaline pectinase with 100% cotton woven fabric of 200 yard length and cotton/nylon/polyurethane woven fabric of 1000 yard length by the cold-pad-batch method. Padding solution was prepared by mixing amylase, alkaline pectinase and wetting agent where pH is adjusted to 8.5 using sodium carbonate. Absorbance of treated fabric was good enough for dyeing and uniform when absorbance at five locations across the length of 1000 yards were measured. Results were compared with those of each company's conventional cold-pad-batch chemical pretreatment. When conventional bleaching is done after simultaneous desizing and scouring in cold-pad-batch method using two enzymes, absorbance and whiteness is as excellent as the company's conventional method.

Keywords: Cold-pad-batch; Pretreatment; Bio-pretreatment; Desizing; Scouring; Bioscouring; Enzymatic; Alkaline; Cotton; Cotton blend

Introduction
The noncellulosic constituents of cotton fiber are known to be waxes, pectins, hemicellulose, proteins, and mineral matters ranging from 4 to 12% of the dry fiber weight and to be located mostly in the cuticle, in the primary cell wall, and in the lumen. Natural cotton wax of 0.4 to 1.0% serves as a protective barrier to water penetration comprising the cuticle on the outer surface of the fiber while pectins of 0.7-1.2% are present as a poly-D-galacturonic acid in the form of insoluble salts of Ca, Mg, and Fe in the primary cell wall underlying the waxy cuticle [1,2].
reproducible high quality results in dyeing of cotton fabrics, pretreatment process is required to reduce impurities sufficiently to improve cotton fabric wettability (Figure 1). Besides natural impurities such as wax and pectins of which removal is essential [3], removal of secondary added impurities are also important. In the case of cotton knitted fabrics, the added chemicals are those lubricants, for example, paraffin waxes used in spinning the yarn and on the knitting machine that comprise about 0.5 - 2 % of fabric weight [4,5] and are removed in scouring process together with natural cotton wax. For cotton woven fabrics, a separate enzymatic desizing process has been carried out traditionally to remove starch-based sizes added on warp yarns about 15% of the yarn weight in weaving [6] while synthetic sizes applied to man-made fibers are generally water soluble and can be removed in scouring process [2].

The conventional alkaline pretreatment process has been replaced with environment friendly approach using different enzymes. For examples, amylases for removing starch-based sizing and cellulases for denim-washing and biopolishing were introduced in the 1960s and in the 1980s, respectively while pectinase for cotton scouring was launched in the 2000s [3,6-10]. Because pectin acts as glue in the primary cell wall of cotton fibers, the different components present in the primary cell wall layer can be removed easily in a subsequent washing procedure after enzymatic destabilization of the pectin structure [8]. According to (Figure 2(a)), neutralization can be omitted in bioscouring and effluent from alkaline scouring is darker than from bioscouring. In addition to lower loss in weight (Figure 2(b)) and lower pH of scoured fabrics (Figure 2(c)), other beneficial properties from bioscouring using pectinase have been reported to be softer handle, reduction of chemical load to environment and energy consumption as well as lower pilling tendency due to lower damage to fiber (Figure 2(d)) when compared to alkaline scoured fabrics [3,10-13]. Combined bioscouring and biopolishing as well as combined desizing and bioscouring in one step have been also proposed [10].

Cold-pad-batch equipment for pretreatment and dyeing consists of a pad mangle, a means of winding fabric onto a roll and washing chambers with advantages of much reduced water consumption, relatively low cost of equipment, higher productivity over a single batch wise equipment such as a jet dyeing machine [14,15]. However, textile dyeing companies that are equipped with cold-pad-batch machines and run their conventional cold-pad-batch alkaline pretreatment are faced with the difficulty to introduce the new technology in spite of knowing that batch wise enzymatic scouring is successfully implemented. The aim of this case study is to try enzymatic cold-pad-batch bio-pretreatment using the equipment of industrial sites and compared its performance with the company’s conventional cold-pad-batch alkaline chemical pretreatment.

Case Report

Materials

Two kinds of cotton woven fabrics were used and characteristics of each fabric were presented in Table 1. Chemicals such as NaOH (50%), sodium carbonate and hydrogen peroxide (35%) were all industrial grade. Company-specific wetting agent

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Table 1: Characteristics of fabrics used.

| Fabrics      | Fiber Composition | Weave Type | Yarn Number | Fabric Weight (g/m²) | Amounts Treated / Company Name |
|--------------|-------------------|------------|-------------|----------------------|-------------------------------|
| Industrial Trial No1 | 100% Cotton | Plain weave | Ne=20 | 145 | 200 yards / K Textile Co., Ltd |
| Industrial Trial No2 | 60% Cotton, 40% Nylon & PU | Satin weave | Warp: Ne=40 Weft: Nylon 140 D / PU 40 D | 223 | 1000 yards / CW Textile Co., Ltd |

Industrial trials of Cold-Pad-Batch pretreatment

The first CPB bio-pretreatment was tried with 100% cotton woven fabric of 200 yards in one company where the company’s CPB alkaline pretreatment was carried out according to (Figure 3(a)). The second CPB bio-pretreatment was run with cotton-blend woven fabric of 1000 yards in another company according to the process schematized in (Figure 3(b)). For comparisons, each company’s conventional pretreatment was carried out with the same cotton fabric as used in bio-pretreatment. (Figure 3(c)) shows the overall view of the cold-pad-batch process done in two companies.

The CPB bio-pretreatment was carried by padding, batching and washing with hot water in a four-box washer. The details for CPB bio-pretreatment were as followings:
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- Padding solution: Aquazyme 3g/L, Scourzyme 6g/L, Wetting agent 3g/L, pH 8.5
- Padding at 30-35 °C
- Pick-up rate: 85%
- Batching for 24h at room temperature
- Hot washing using a continuous four-box washer:
  1st box (95 °C, emulsifier 2 g/L), 2nd box (95 °C), 3rd box (80 °C), 4th box(60 °C)

The first box contained emulsifying agent to wash out enzymatically degraded pectins and other impurities that came out together. Sodium carbonate (Na₂CO₃) was used for pH adjustment of enzyme padding solution. Because the temperature of padding and batching was about room temperature, amylase of low-temperature type was chosen because it was proved to be more effective than amylase of high-temperature type and importance of removal of sizing agent for enzymatic scouring performance was reported [16].

The second company’s conventional simultaneous alkaline desizing, scouring and bleaching carried out as followings:
- Padding solution: Oxidative desizing agent 1g/L, wetting agent 2g/L, scouring agent 10g/L, NaOH (50%) 100g/L, H₂O₂ (35%) 70g/L, H₂O₂ stabilizer 10g/L
- Pick-up rate: 85%
- Batching for 24h at room temperature

Measurements

Treated fabrics were conditioned and subjected to water drop test. A round fabric sample with a diameter of 10cm was cut and mounted on the test plate of Gravimetric Absorbency Testing System (GATS, M/K Systems, Inc. USA) where absorbed weight per gram of fabric sample was recorded every 0.4sec for 10sec. Additional water absorbency test was carried with all fabric samples according to the AATCC water drop test (test method TS-018) and time for absorbing a water drop was measured. Colored aqueous solution of 1% direct blue dye was additionally used for drop test on cotton-blend fabric. Color measurements of treated fabrics were taken with Macbeth Color Eye 3000 using D65 daylight as the illuminant. Whiteness, L-value and b-value were measured.

Results & Discussion

Performance of pretreatment is evaluated mainly by water absorbance of treated fabrics. When absorbance data were compared in (Figure 4(a)), 100% cotton woven fabrics obtained by two different CPB pretreatments resulted in the almost same absorbance between the simultaneous enzymatic desizing and scouring and the enzymatic desizing followed by alkaline scouring. Both new CPB bio-pretreatment and the company’s conventional alkaline pretreatment were tried with cotton fabric in industrial scale of 200 yards. With the company’s CPB equipment according to Industrial Trial No.1 presented in (Figure 4(b)) where both pretreatments contained CPB bleaching at the end.

When the simultaneous enzymatic desizing and scouring by CPB was tried in the other company with cotton-blend fabrics, absorbance data by GATS revealed that cotton-blend fabric after CPB bio-pretreatment absorbed less water than after the second company’s conventional pretreatment in which the simultaneous alkaline desizing, scouring and bleaching by CPB was carried out (Figure 4(b). However, the drop test on cotton blend fabric obtained by CPB bio-pretreatment was less than 3 seconds that is regarded to satisfy the absorbency standard for drop test that AATCC (American Association of Textile Chemists and Colorists) recommends to be in the range of 1 to 5sec. All other fabrics were wetted in less than 2 seconds by drop tests.
The cotton-blend fabric used in industrial trial No.2 consisted of the warp yarn of 100% cotton and the weft yarn of 60% nylon and 40% polyurethane. Therefore, CPB bio-pretreatment seemed to remove less impurities on the weft yarn than the company’s CPB chemical pretreatment while CPB bio-pretreatment worked very well on 100% cotton warp yarn as evidenced by drop test results (Figure 5(a)). In this industrial trial No.2 where the treated cotton blend fabric was 1000 yards in length, absorbance data measured in the spot of 100, 450, 680, 900 and 1000 yards proved the homogeneous treatment by CPB bio-pretreatment across the long length (Figure 5(b)).

![Diagram of yarn types and treatment methods](image)

**Figure 5:** Absorbance measurement on cotton/nylon/polyurethane woven fabric (Industrial Trial No.2) by (a) drop test and (b) gravimetric absorbency testing system (GATS) showing the homogeneous results across the 1000 yards of fabric pretreated by simultaneous enzymatic cold-pad-batch desizing and scouring (arrow bar: n=5).

Table 2 shows the results of color measurements of cotton fabrics treated by enzymatic CPB pretreatment and each company’s conventional chemical CPB pretreatment in industrial scale. Whiteness, lightness and b-values were focused among color measurement parameters. Bigger values in whiteness and L represent lighter and whiter while bigger b values mean yellowness of fabric. When values of final fabrics bio-pretreated were compared to those pretreated conventionally, subsequent dyeing of bio-pretreated fabrics is expected to give as reproducible results as those pretreated conventionally regardless of pale to deep shade dyeing both in industrial trial No.1 and No.2.

### Table 2: Evaluation of whiteness, lightness and b-value of cold-pad-batch pretreated fabrics.

| Fabrics pretreated by | Enzymatic CPB pretreatment | Company’s Chemical CPB pretreatment |
|----------------------|----------------------------|-----------------------------------|
|                      | Whiteness | L       | b       |
| **Industrial Trial No.1** |           |         |         |
| Greige               |           |         |         |
| Simultaneous enzymatic desizing & scouring | 8.6 | 85.5 | 11.7 |
| Enzymatic desizing & alkaline scouring in two steps | | | |
| H₂O₂-bleaching      | 67.8      | 63.7    | 93.7    | 89.4 | 2  | 2.4 |
| **Industrial Trial No.2** |           |         |         |
| Greige               |           |         |         |
| Simultaneous enzymatic desizing & scouring | | | |
| Simultaneous alkaline desizing, scouring & bleaching | 50.7 | 72.8 | 89.8 | 91.9 | 5  | 1.6 |
| Mercerized           | 51.6      | 71.6    | 89.6    | 91.6 | 5  | 1.7 |
| H₂O₂-bleaching      | 123.1     | 126.9   | 92.8    | 92.9 | -8.8 | -9.5 |

Table 2 shows the results of color measurements of cotton fabrics treated by enzymatic CPB pretreatment and each company’s conventional chemical CPB pretreatment in industrial scale. Whiteness, lightness and b-values were focused among color measurement parameters. Bigger values in whiteness and L represent lighter and whiter while bigger b values mean yellowness of fabric. When values of final fabrics bio-pretreated were compared to those pretreated conventionally, subsequent dyeing of bio-pretreated fabrics is expected to give as reproducible results as those pretreated conventionally regardless of pale to deep shade dyeing both in industrial trial No.1 and No.2.

### Conclusion

Industrial trials on bio-pretreatment of 100% cotton and cotton-blend woven fabrics by cold-pad-batch methods using the combination of amylase of low temperature type for desizing and alkaline pectinase for scouring gave the satisfactory results in water absorption and whiteness of pretreated cotton fabrics as well as the homogeneity of water absorption across the 1000 yards of fabric length. These results allow textile dyeing companies to use their existing CPB facilities for the introduction of new enzymatic pretreatment process substituting for a conventional strong alkaline process while maintaining their production capacity.

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Conflict of Interest
The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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