



IN SITU GENERATION OF HYDROGEN PEROXIDE BY GLUCOSE OXIDASE IN DESIZED LIQUOR FOR COTTON BLEACHING

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ABSTRACT

Pretreatment of cotton are essential to make it suitable for finishing process where soul of bringing whiteness to the cotton fabrics after removal of sizing agents with lesser damage belongs to bleaching. In situ generation of hydrogen peroxide from glucose saves enormous amount of water and its eco-friendly. Glucose was estimated as 3800 mg/l in desized liquor and the maximal hydrogen peroxide generation was attained at pH 5 under 55°C using 1% glucose oxidase for 75 mins at 100 rpm. Bleaching was performed in desized liquor under optimized parameters. Various physical and functional properties were determined to ensure the quality of fabrics. Weight loss and wettability of enzymatically bleached fabrics were found to be 4.15% and 1 sec respectively. Strength of enzymatically bleached fabrics showed 43

and 36 lbs strength in warp and weft direction comparatively higher than conventionally bleached. Whiteness index of bleached fabrics was found to be 79.4 which slightly lower than conventional bleaching. Henceforth, the result evidenced that in situ generated hydrogen peroxide was efficiently performed an effective bleaching of cotton fabrics thus regarded as greener alternative.

KEYWORDS: Bleaching, in situ generation, hydrogen peroxide, desized liquor, glucose

INTRODUCTION

Cotton and its blended fabrics contain some inherent impurities such as oil, fat, wax and pectin within the fiber structure. In cotton wet processing, before finishing of fabrics, the impurities and yellowish color would be removed permanently using pretreatments (Eren, 2018). The yellowish coloration of fabric may be related to protoplasm residue of protein and flavones and even occurs due to dirt, dust, oils and greases of processing equipment (Brushwood, 2003). Therefore, the pretreatment processes for cotton are essential to make the textile substrate suitable for subsequent coloration or finishing processes (Imran *et al.*, 2015). Among various pretreatments, the soul of bringing whiteness to the textile fabrics with minimum or no damage to the fabrics belongs to bleaching despite desizing and scouring made absorbent (Hamzah *et al.*, 2015).

Each individual pretreatment process separately consumes water, steam, electricity and chemicals, causing chemical oxygen demand (COD) and biological oxygen demand (BOD) loads in the washing effluent (Imran *et al.*, 2015). Stringent rules and regulations related to environment, in addition to save water and energy, enzyme biotechnology has interestingly entered into textile area. The acceptable degree of desizing along with good absorbency and whiteness has remained a challenge in rapid enzymatic single bath process which is environmentally friendly and saves the enormous amount of water and energy (Öner and Sahinbaskan, 2011). In situ generation of hydrogen peroxide in desizing liquor contains glucose where acts as a substrate by glucose oxidase enzyme that aids in bleaching of cotton fabrics. Treatment bath of desizing exists with an enormous quantity of glucose molecules that could be used for hydrogen peroxide generation with the addition of a little amount of glucose if needed will ultimately be resulted in one bath – two-step pre-treatment. It is pertinent to mention that oxidase enzymes still have not been usually reported would show a noteworthy result in cotton pretreatments equal to highly mentioned hydrolytic enzymes.

The objective of our work was to compare the properties of enzymatically bleached by in-situ generated hydrogen peroxide using glucose oxidase in one bath - desizing/bleaching pretreatment with respect to conventionally bleached fabrics with the emphasis on their degree of whiteness. Based on the information said above, the ultimate goal of this study was designed as in-situ generation of hydrogen peroxide for cotton fabric bleaching.

MATERIALS AND METHODS

Materials needed

Plain weave 100% cotton fabric, a wetting agent was supplied by National Textile Corporation Private Limited, Coimbatore. Amylase enzyme was commercially obtained from Eco star textile chemical suppliers, Tiruppur. Glucose oxidase and D-glucose (Hi-Media) were purchased Jayam Scientific Company in Coimbatore, Tamilnadu, India.

Desizing

Fabric sample to be used were washed with running water in order to eliminate non-sticky impurities such as dust, dirt etc., applied sizing agent such as starch in the cotton fabrics was removed by desizing. Fabric samples were treated at 45°C with a liquor ratio of 40:1 using 0.1% amylase enzyme and 0.1% wetting agent at pH 7.4 for 1 hr. The fabrics were then washed several times with hot water at 90°C for 10 mins subsequently with cold water at 25°C in the liquor ratio of 20:1 and dried at 100°C (Sahinbasken and Kahraman, 2011). The Glucose molecule in desizing treatment bath was measured using dinitrosalicylic acid (DNSA) method and it has been stored for bleaching purposes. Now, desized fabrics were supposed to treat conventionally and enzymatic treatment respectively.

Estimation of glucose in desizing liquor

Reducing sugar formed after enzymatic hydrolysis of soluble starch in desizing liquor was measured using DNSA method. A volume of 2 ml of desizing liquor was added with 2 ml of dinitrosalicylic acid (DNS) reagent and heated for 5 mins in boiling water bath and absorbance was read at 540 nm to estimate reducing sugars released (Karnwal and Nigam, 2013).

Conventional bleaching of cotton fabrics

The desized cotton fabric was conventionally bleached with 35% hydrogen peroxide, 0.1% sodium silicate, 0.1% magnesium sulphate, and 1% wetting agents contained liquor solution at 90°C for 30 minutes. The fabrics were then washed several times with hot water subsequently with cold water and dried at 100°C.

Optimization of hydrogen peroxide production in desizing liquor

According to the methods described in Anis *et al.*, 2009, the physicochemical parameters such as pH, temperature, enzyme dosage, time was optimized under static and agitated

conditions. After incubation was over, hydrogen peroxide production was determined by potassium permanganate titration (AATCC-102) test method.

Effect of pH

Desizing liquor pH has been adjusted to 4, 5, 6, 7, 8, 9, 10 and 11 (acid the alkaline range) with addition of 1% glucose oxidase at 55°C for 1 hour under different agitated conditions (0, 25, 50, 75, 100 rpm).

Effect of temperature

Desizing liquor was subjected to different temperatures for hydrogen peroxide production such as 25, 35, 45, 55, 65, 75 and 85°C with the addition of 1% glucose oxidase at optimized pH for 1 hour under different agitated conditions (0, 25, 50, 75, 100 rpm).

Effect of time

Desizing liquor was subjected to a different time for hydrogen peroxide production such as 15, 25, 35, 45, 55, 65, 75 and 85 with the addition of 1% glucose oxidase under optimized pH and temperature and different agitated conditions (0, 25, 50, 75, 100 rpm).

Effect of enzyme dosage

Desizing liquor was subjected to different enzyme concentration for hydrogen peroxide production such as 0.25, 0.5, 0.75, 1, 1.25, 1.5, 1.75 and 2% with addition of 1% glucose oxidase under optimized pH and temperature and different agitated conditions (0, 25, 50, 75, 100 rpm).

In-situ generation of hydrogen peroxide for bleaching

Glucose oxidase enzyme oxidizes the glucose molecule existing in the desizing liquor thus hydrogen peroxide generates that aids in bleaching of the fabric sample. Desized fabrics were exposed for bleaching by in-situ generated hydrogen peroxide by glucose oxidase enzyme under optimized conditions. After bleaching, the enzyme bleached fabrics were taken out of liquor and put into quenching water bath at 100°C for 10 minutes to deactivate the enzymes. The fabrics were then washed several times with hot water at 90°C for 10 mins subsequently with cold water at 25°C and dried at 100°C.

Assessment of physical properties of bleached fabrics

Weight loss method

Weight loss reflects the degree of hydrolysis that has occurred during the treatment of fabrics. The sample was measured previously before treatment and subsequently measured after treatment. The following formula was used to calculate the weight loss in terms of percentage

$$\% \text{ Weight loss} = \frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \times 100$$

Evaluation of wettability (AATCC 39 - 1980)

According to an AATCC Test Method 39-1980, water absorbency was monitored by placing a drop of water on the fabric where the droplet took time to enter inside the fabric was noted regarded as wetting time. The rate of fabric absorbency is indirectly proportional to the wetting time. This test was done in triplicate and the average was calculated (The Resilient, 2007 & Hebeish *et al.*, 2009)

Tensile strength measurement using strip method

The tensile strength of the fabric samples was measured using tensile strength tester according to ASTM D5034-08. The sample strip size of 180 mm x 50 mm was precisely cut and the analysis was done at 50mm/min stretching rate. This test was done in triplicate and the average was calculated (Kenawy *et al.*, 2014).

The whiteness of bleached fabrics

The fabrics were measured for their whiteness index in 330 – 700 nm using Mac Beth 7000A spectrophotometer. This test was done in triplicate and the average was calculated.

RESULT AND DISCUSSION

Hydrogen peroxide was found to be an ideal choice for effective bleaching where it proved formerly as environmentally safe agent for cotton but requires a large amount of rinsing water after completion of the process. In order to achieve whiteness of woven cotton fabric, environment-friendly enzymatic bleaching by in situ generation of hydrogen peroxide using desizing liquor was designed in this study. As a first process, Sizing agents were removed using the alpha amylase enzyme which is currently employed for desizing in entire textile industries for grey woven fabric and now exposed for whitening. One bath – two-step

treatments had an advantage of lower contamination rather than scouring because removed impurities from cotton after scouring tended to redeposit while bleaching. DNSA result showed the presence of 3800 mg/l of glucose units in desizing liquor. Bushcle-Diller and Yang, 2001 showed the presence of 4100 mg/ml glucose units in desizing and scouring bath.

Conventional bleaching was performed by hydrogen peroxide and sodium silicate that combined together to give effective bleaching of cotton fabrics. Bleaching plays a key role in providing the degree of whiteness to the fabric. As per Wasif & Indi, 2010, Coloration and fabric finishing of the fabrics was done after bleaching is one of the key aspects for better quality enrichment would be based on the bleaching result quality. Conferring to the Ardon *et al.*, 1996 statement, the main objective of bleaching was to a removal of natural pigmented materials that are present in the fabrics eventually results in white appearance. The whiteness of the fabrics needs to be achieved without any losses in fabric strength. Peroxide residues were removed by a huge quantity of water while washing the fabrics.

Standardization of desizing liquor composition would ultimately result in sufficient production of hydrogen peroxide for bleaching under static and agitated conditions. Agitation is a promising factor for efficient and even supply of air into liquor medium thus enzyme activity increases. To evaluate the pH influence, glucose peroxidase was added after varying initial pHs were adjusted to liquor and generated hydrogen peroxide was estimated. Subsequently, the pH found on desizing liquor was about 4.4. Figure 1 showed that maximal production of hydrogen peroxide was attained at pH 5 was about 780 mg⁻¹/l under 100 rpm.

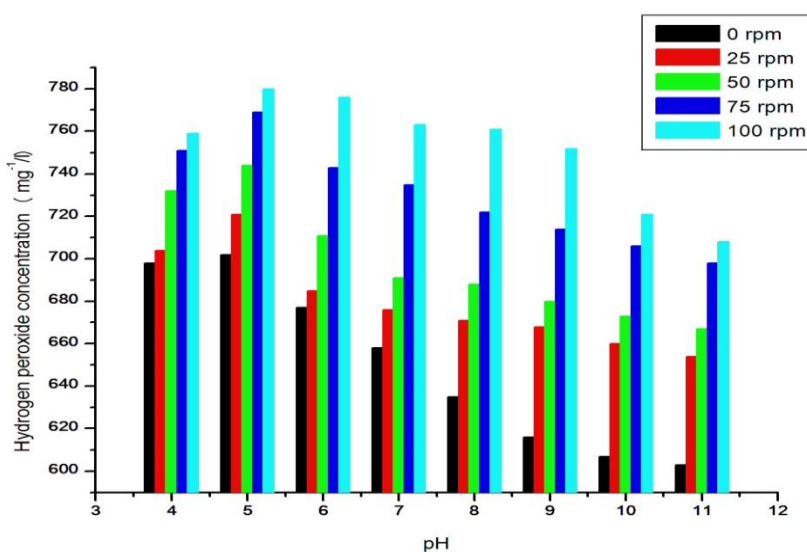


Figure 1: Effect of pH in hydrogen peroxide generation.

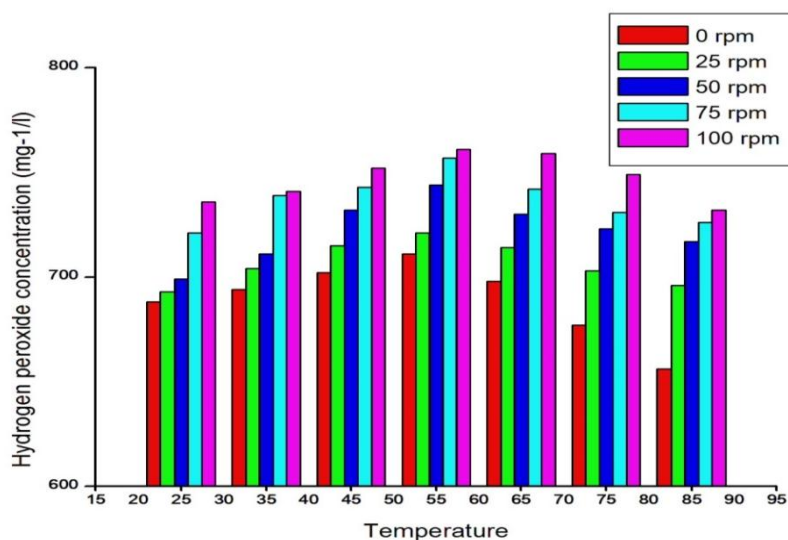


Figure 2: Effect of temperature in hydrogen peroxide generation.

Influence of temperature in peroxide production was performed from 25 – 85°C with an interval of 10°C after adding glucose peroxidase and generated hydrogen peroxide was estimated. Figure 2 shown that maximal production of hydrogen peroxide was 761 mg⁻¹/l that attained at 55°C under 100 rpm. Since time was considered as a small factor but it was a key role for little bit higher amount of product formation. Desizing liquor has been incubated at optimized pH and temperature along with glucose oxidase with varied time duration from 15 – 85 minutes with an interval of 10 minutes for generation of hydrogen peroxide was studied. Figure 3 has shown that optimal time required for peroxide generation was found to be 75 minutes was about 773 mg⁻¹/l under 100 rpm.

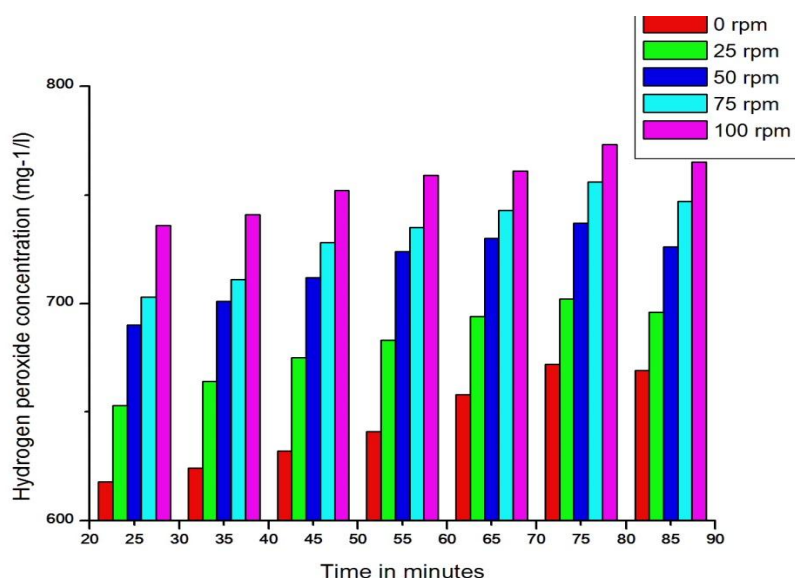


Figure 3: Effect of time in hydrogen peroxide generation.

Glucose oxidase solely plays a crucial factor for hydrogen peroxide from glucose molecule despite many physical factors. Of course, optimization of enzyme dosage must be studied along with different parameters. Desizing liquor was subjected with a varied dosage of glucose oxidase (1000 U/mg) from 0.25% - 2% with an interval of 0.25% under previously parameters had shown that maximal production was found at 1% concentration about 739 mg⁻¹/l under 100 rpm illustrated in the Figure 4. Investigation findings of Bushcle-Diller and Yang, 2001 had explored that maximum level of peroxide generation was reported at 0.01% enzyme dosage has 1million U/g at 55 °C for 45 minutes. Thus it significantly correlates with the result of optimization with a small difference in time but there was only a minute difference in hydrogen peroxide production.

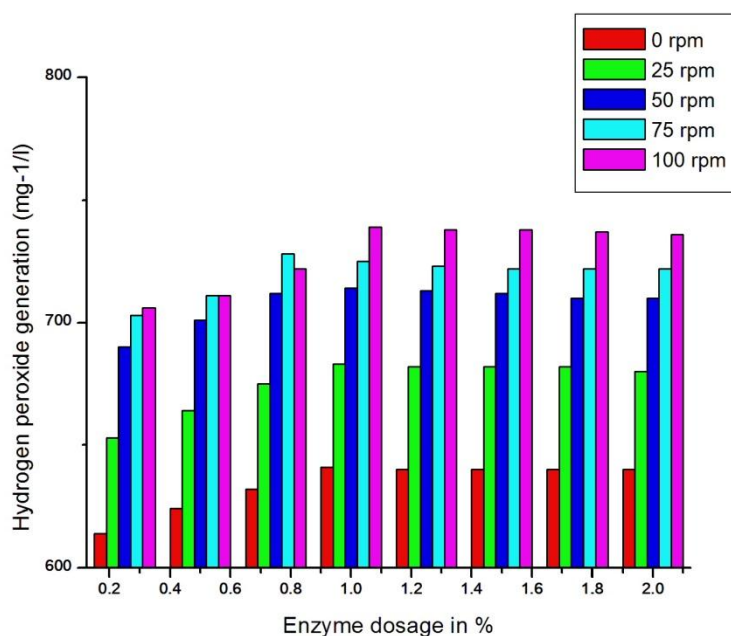


Figure 4: Effect of enzyme dosage in hydrogen peroxide generation.

Hydrogen peroxide was generated by oxidizing the glucose with the assistance of enzyme glucose oxidase publicized effective bleaching of the fabrics under optimized physicochemical parameters. Meanwhile, in-situ generated hydrogen peroxide bleaching technique was the trend back in upcoming years because of its signifying aspects at treatment time, usage of chemicals, reduced oxygen demand level in the textile effluent compared with previously reported ones. Moreover, it greatly enhances the dye utilization of bleached cotton fabric while dyeing.

To ensure the quality of cotton bleaching, analyzed various physical and functional properties such as weight loss, water absorbency, tensile strength and whiteness of the fabrics was relatively showed below. Weight loss is one of the most important factors that relate with strength of the fabrics. Higher weight loss would show weak strength, greater water absorbency and vice-versa. In this study, it has been noted that weight loss of the conventionally bleached fabrics had increased up to 5.14% whereas in enzymatic bleaching possessed minimum weight loss up to 4.15% of fabrics after desizing where lose 3.98% weight prior bleaching depicted in the figure 5. In spite of higher concentration of peroxide, it might damage the fabrics resulted in weight loss whereas in-situ generated peroxide in enzymatic bleaching caused no damage to the fabrics which stated by the author Uddin, 2010 in his findings.

It can be conferred from Figure 5 that wettability of conventionally and enzymatically bleached fabrics were observed as faster absorption of water molecule within 2 sec and 1sec, however, desized and grey fabrics required 6 sec and more than a minute respectively. Since sizing agents were removed thereby water molecule penetrates faster than grey fabrics. Moreover, bleached fabrics might cause some modification on the surface of the fabric, so that the water molecules could penetrate into the fibers thus increases water absorption.

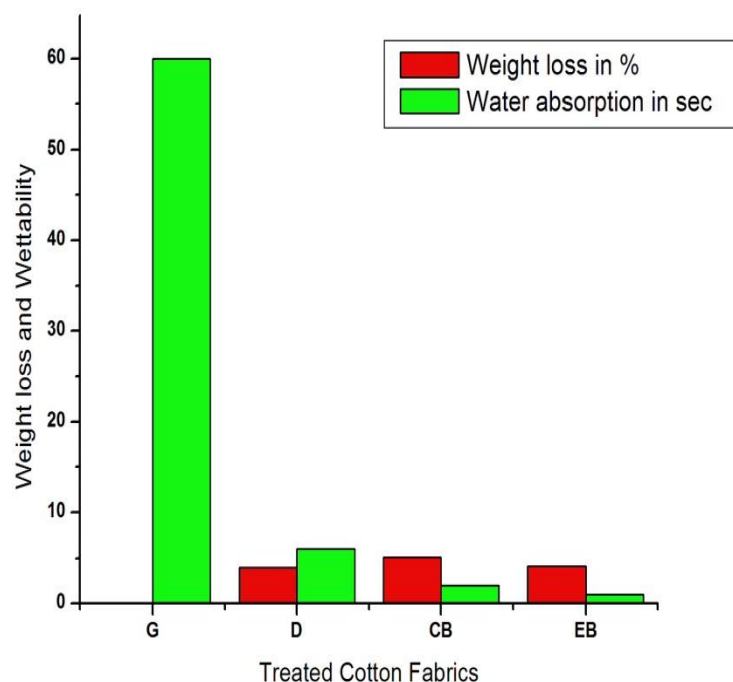


Figure 5: Weight loss and wettability of pretreated cotton fabrics.

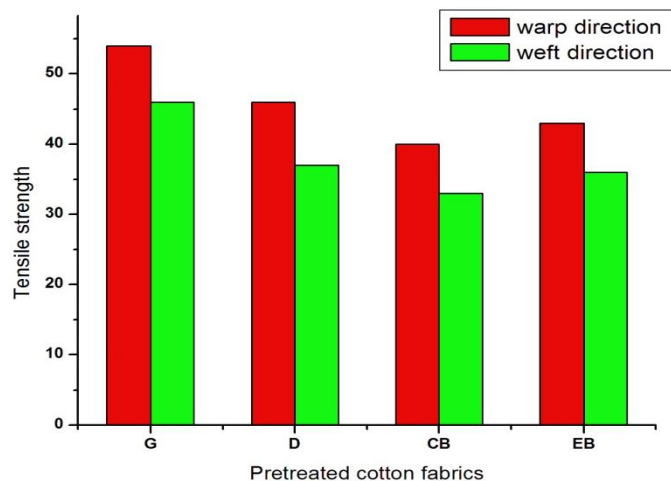


Figure 6: Tensile strength of pretreated cotton fabrics.

Strength of the fabrics would pay any attention to the researchers because it was directly proportional to the quality of the fabrics. The tensile strength of the grey, desized, conventionally and enzymatic was found to be 54, 46, 40, 43 in a warp direction and 46, 37, 33, 36 in weft direction respectively that depicted in the Figure 6. From the result, it has been shown that enzymatically bleached showed comparatively higher strength than the conventional process. In addition to that, it also conferred lower weight loss with a comparison of desized fabrics. In the case of conventional bleached fabrics, increased peroxide concentration could decrease the strength of fabric were clearly shown in the Figure 6 because of peroxide reacted with the fiber. Correspondingly, when fiber was oxidized thus strength lowered.

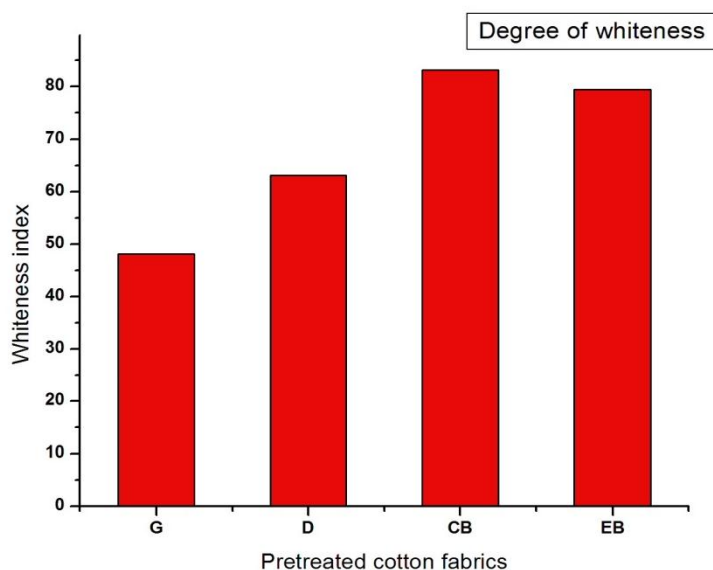


Figure 7: Whiteness index of pretreated cotton fabrics.

Degrees of whiteness were illustrated in Figure 7. In this Figure, the whiteness index of grey, desized, conventionally and enzymatically bleached fabrics was identified as 48.07, 63.1, 83.2 and 79.4 respectively. The degree of whiteness after a one-bath two-step treatment was slightly lower than those after bleaching with commercial peroxide. Even though whiteness was increased to some extent, the strength of the fabrics was very low in conventional bleached samples rather than enzyme treated. Sample, which was desized prior to bleaching, had the lowest degree of whiteness. Overall, it was suggested that bleaching with hydrogen peroxide either commercial or in-situ generated was effective even though the impurities were not removed from cotton fibres to a higher extent, said Abdul and Narendra, 2013.

CONCLUSION

A successful strategy of α -amylase desized liquor for in-situ generation of hydrogen peroxide using glucose oxidase was developed as one-bath two-step treatment. As we all well-known about ever-growing costs for water and energy, worldwide investigations were carried out to substitute conventional chemical textile processes by environment friendly and even economic attractive bioprocesses using enzymes. Hence, this present study has proved that this method would be regarded as a greener alternative thereby reduces the time and economic. In the future, this technical break-through have to be explored to open this environment-friendly concept in large-scale industrial purposes with the even more attractive application would ultimately reduce the cost of bleaching.

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