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PJBS

ISSN 1028-8880

**Pakistan
Journal of Biological Sciences**

ANSI*net*

Asian Network for Scientific Information
308 Lasani Town, Sargodha Road, Faisalabad - Pakistan

Effect of Washing on the Burning Behaviour of Phosphorylated and After-treated Jute

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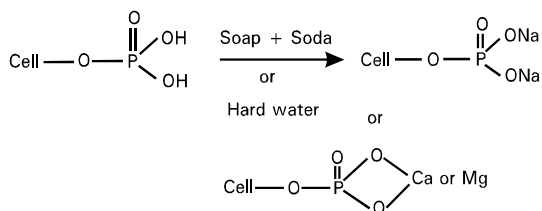
Abstract: Scoured and bleached jute fabrics were phosphorylated by treating with urea-phosphoric acid salt solution under an optimum condition of concentration, pH, liquor pick up and curing. The samples were rephosphorylated and again cured under same conditions and were treated with the solution of antimony salt and zirconium salt together, neutralized and finally subjected to washing treatment. The treated jute fabric was tested for flame retardancy by standard method and was found to be significantly flame retardant with good fastness to washing.

Key words: Flame retardancy, phosphorylation, curing, washing, ion-exchange, phosphorous content, after-glow, char length, flammability

Introduction

The flame retardant finishing of cellulosic fibres is of significance in diversified textile uses. According to the use in field and the use in textiles, quite different requirements need to be made of the good wash fastness of the finishing. Considering the wash resistant finishing of cellulosic fibres, finishes on the basis of phosphorus-nitrogen compounds got prior importance to the investigators (Berger, 1957; Liu, 1973; Lipson, 1975; Hussain 1980), amongst different types of non-resinous flame retardant finishes for cellulosic fibres, phosphorylation is reported to be of considerable importance as it imparts excellent flame resistant properties without significant influence on the handle of the fabric. But when attempts were taken to modify cellulose fibres by phosphorylation reaction to impart wash fast flame retardant property, it failed to a great extent as this finish became somewhat ineffective on washing with hard water or soap-soda (Einsele, 1978 and Einsele *et al.*, 1980).

The inefficiency of this finish was initially thought to be due to the ion-exchange property of the phosphorylated cellulosic fibres (Sharma, 1980; Pandya *et al.*, 1982; Horrocks, 1983; 1986). During phosphorylation of cellulosic fibres, only one OH group of phosphoric acid reacts with cellulose and the remaining two OH groups being free can take up alkali metal ions from the washing liquors to form an ion-exchange derivative of phosphorylated cellulose probably of the following type :



This ion-exchanged cellulose phosphate is converted to alkali metal phosphate during heating and does not release phosphorus at the pyrolytic temperature of the cellulose, which in turn impairs the flame retardancy mechanism and thus cellulose fibres burn (Sharma, 1980; Horrocks, 1983; 1986). However, another possibility which can also impair the flame retardance property of phosphorylated cellulose is the non stability of the phosphoric acid ester-bonds during soap-soda washing of the fabric. Metal oxides are known to reinforce the flame retardant property of cellulose in combination with phosphorus. The effectiveness of after treatment with metal oxide lies in the fact that these can form a complex with OH group of cellulose-phosphate and can reduce ion-exchange considerably. At the same time these complexes can

release phosphorus at the pyrolytic temperature of cellulose and thus impart flame retardant properties (Horrocks, 1983; 1986; Bras *et al.*, 1996; Bourbigot *et al.*, 1996).

It was revealed earlier (Sharma, 1980) that after-treatment with a combination of antimony and zirconium salts onto phosphorylated cellulose imparts better flame retardancy than single metal oxide. Though the washings were unable to make the finish almost completely ineffective, the finish was impaired to some extent, which resulted in after-glowing. This phenomenon led us to undertake the present study to evaluate the phenomenon critically and precisely using jute fabric as experimental sample. To understand in detail the burning behaviour of phosphorylated and antimony-zirconium salt after-treated jute after severe washing, it was felt necessary to investigate the effect of washings on phosphorus content, after-glow and char length of variously reacted jute fabrics. An attempt was made to examine whether the phosphorus elimination or the ion-exchange mechanism is responsible for the change in the after-glow and charring properties of the treated fabrics after washing treatment.

Materials and Methods

The experimental works were started since January, 1999 and were finished by the end of December, 2001. Most of the experimental works had been carried out in the Chemistry Laboratory, Bangladesh Jute Research Institute (BJRI), Dhaka. Some finishing works were done in the Textile Laboratory, ENSAIT, France in collaboration with Professor Serge Bourbigot in the year 2001.

Application of the urea-phosphoric acid salt solution to jute fabric:

Jute fabrics (carpet backing cloth) were pre-treated (scoured and bleached) by standard methods (Trotman, 1984). Urea-phosphoric acid salt was prepared and diluted in three different concentrations. The pre-treated jute fabric samples were padded with the respective salt solution having pH 7 and with a liquor pick up of 100%, then dried at 100° C for 5 minutes and subsequently cured at 150° C for 3 minutes. The cured samples were thoroughly washed in cold water and dried at room temperature.

Rephosphorylation: The phosphorylated samples were again padded with the salt solution of identical strength used in phosphorylation, cured, washed and again dried in the same manner as stated above.

After-treatment: The phosphorylated jute fabric samples were padded with a solution containing a combination of antimony oxide and zirconium oxy-chloride with a liquor pick up of 80%.

Hussain *et al.*: Burning behaviour of phosphorylated jute

The fabric was batched for 5 minutes and subsequently treated with 20% sodium carbonate solution to neutralize the acidity. The samples were then washed thoroughly in running cold water at room temperature.

Washing treatment: In a single washing treatment, the sample was boiled for 30 minutes in a solution containing 3g/l soap and 1.5g/l soda ash.

Estimation of phosphorus content: Phosphorus content in the treated sample was determined according to the method suggested earlier (Basch and Wassermann, 1970).

Testing of flame retardancy: The treated jute fabrics were tested for flame retardancy by measuring 'after-flame', 'after-glow' and 'char length' under vertical flammability test method (Anonymous, 1969).

Results and Discussion

During phosphorylation of jute fabrics to different levels of phosphorus content it was observed that the fabric showed excellent flame retardant properties. But simultaneously, it was found that when the samples were washed in hard water or with soap-soda, the fabrics failed in flame retardancy to some extent. This had been mainly attributed to the ion-exchange property of phosphorylated jute. However, it was thought worthwhile to examine the effect of different levels of phosphorus content in the fabrics on its ion-exchange behaviour. With the three phosphorylated samples studied having phosphorus content 3%(A), 2.65%(B) and 1.80%(C) respectively, the fabric loses phosphorus during the initial 1-3 washings after which the phosphorus content practically remains almost constant (Fig. 1). Fabric samples with 3 and 2.65% initial phosphorus, contain sufficient phosphorus even after a number of washings but the fabric burns along its entire length even after a single washing which indicates that whatever may be the initial phosphorus content in the fabric, during the subsequent washings, it picks up alkali metal ions and thus becomes somewhat ineffective against burning.

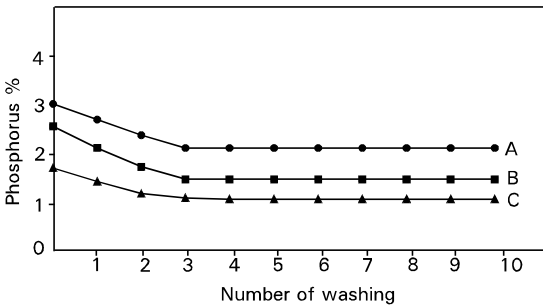


Fig. 1: Effect of washing on phosphorus. A, B, C presents phosphorylated samples

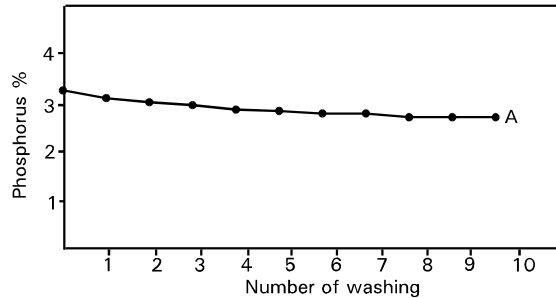


Fig. 2: Effect of washing on phosphorus (Rephosphorylated sample).

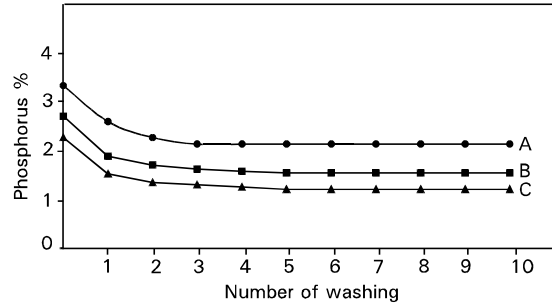


Fig. 3: Effect of washing on phosphorus (after treated samples)

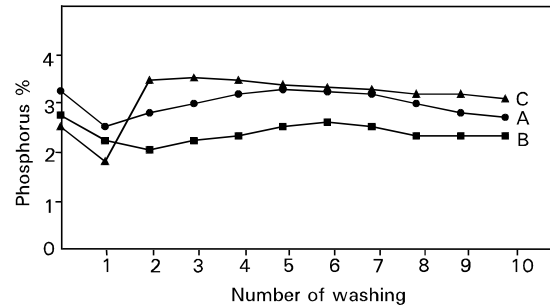


Fig. 4: Effect of washing on char length

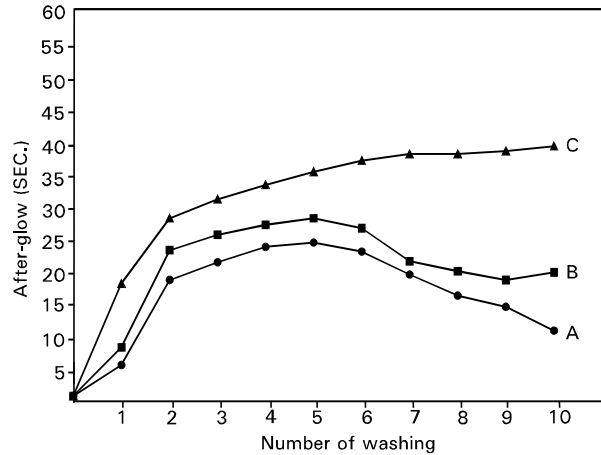


Fig. 5: Effect of washing on after-glow

These phosphorylated jute fabric samples when subjected to a rephosphorylation treatment, gave entirely different results. The rephosphorylated jute as such exhibited flame retardancy up to 5 washes and also lose phosphorus during the first 3 washing (Fig. 2), after which the phosphorus content remains almost constant. So, the ion-exchange phenomenon must have been suppressed by the blocking of the P-OH groups in phosphorylated jute to some extent. Increased add on of phosphorus seems to play little role in this respect. In spite of increase in phosphorus content of the sample, after first 3 washings, it comes to the level of phosphorylated jute (Fig. 2).

In subsequent washings, there is only marginal loss in phosphorus content while the sample retains flame retardancy up to 5 washings, after which the sample fails to exhibit flame retardancy because of regular ion build up at sites created by hydrolysis as also at some of the unreacted P-OH groups. Rephosphorylation, therefore, seems to be a very suitable method of overcoming the

ion-exchange tendency of phosphorylated jute to some extent. The after-treated fabric samples also lose phosphorus during the first few washings after which the phosphorus content remains practically constant (Fig. 3).

On the other hand, these samples show decrease in char length during the first few washes (Fig. 4). After these initial washings, the char length increases on more number of washings.

In fact, it is expected that as the phosphorus content of the samples decrease, the char length should increase. The initial decrease in char length may be attributed to reorientation and penetration of the metal oxides in the fibre structure. As these metal oxides, by themselves, impart flame retardancy to jute, the effective char length of the phosphorylated and after-treated sample is reduced. Also, during the soaping treatments, the sample loses some metal oxides, as also some phosphorus leading to increase in char length subsequently.

It was also observed that the phosphorylated and after-treated samples when washed in soap-soda, always exhibited after-glow, the duration of which being different with different samples (Fig. 5). But the after-glow of the samples observed was not of the same type as occurs under normal circumstances. In the experimental samples the glow appears only at the point of contact of the sample with the flame, the glow not proceeding along the length of the charred area. This is perhaps mainly due to the fusion and glowing of the sodium metal under high temperature of the flame. In the phosphorylated and after-treated samples, glowing may not always be eliminated. Here, although the experimental sample has been made ineffective to pick up Ca or Mg from the wash baths, it may still have the tendency to pick up Na. In the aforesaid conditions of treatments the cellulose ester molecules form a complex with one metal ion, leaving two separate free P-OH groups on two separate cellulose ester molecules, which cannot pick up Ca or Mg but can easily pick up Na. From the total observations it may be inferred that the ion-exchange property of the phosphorylated cellulosic fibres impairs flame retardancy mechanism by formation of Ca and Mg phosphates during washing with hard water or soap-soda and a reduction of this ion-exchange effect can be achieved markedly by an after-treatment of the phosphorylated cellulosic fibres with some metallic salts specially with antimony and zirconium salts.

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