Study on Physical Properties of Butane Tetra Carboxylic Acid (Btca) & Beta Cyclodextrin (Cd) Treated Lyocell Fabric

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Abstract:- The physical properties of Lyocell fabric was studied by treatments of polycarboxylic acid (pca) & beta cyclodextrin with different concentration .The surface morphology, tensile, tear strength, whiteness, pilling tendency, flammability, Abrasion resistance and Crease recovery are assessed for each of the samples and compared with original sample and normal Dimethyl di-hydroxy ethylene urea treated sample. The fibrillation of Lyocell fabric is an normal structural changes in case of any wet treatment and the same was analysed and the fibrillation behavior was studied among different concentration with different resins; it shows that the fibrillation property is minimized when the treatment is takes place with both kind of resins and Both treatments/ significantly improve the crease recovery properties of the fabric. And the tensile and tear strength of the Polycarboxylic acid treated sample shows less impact than normal resin used for anticrease finishing. There are no appreciable changes in the flammability of Dimethyl di-hydroxy ethylene urea and there is little improvement in Polycarboxylic acid treated fabric. The pilling tendency and abrasion resistance of the cvclodextrin treated sample shows more impact than Dimethyl di-hydroxy ethylene urea.

Key-words: Lyocell, Cross linking, Beta Cyclodextrin, 1,2,3,4 Butane tetra carboxylic acid

1. INTRODUCTION

The countries like India where summer is prevailing for about 6-9 months, people like to wear dress materials made of 100% lyocell. To date, only the NMMO (N-methyl-morpholine-N-oxide) process is used for better production of Lyocell fabric (1, 2) offers a commercially viable alternative. The structure and properties of Lyocell fibers are considerably different from other and man-made cellulose fibers [3, 4, and 5]. A special feature of Lyocell fiber is their ability to fibrillate in the swollen state. The process involves primary fibrillation by means of controlled mechanical action, which leads to the appearance of fibrils on the fiber surface. The fibrillation process is followed by an enzymatic treatment, which removes the fibrils and cleans the fiber surface [6] Functional finishes can change the properties of lyocell fibre and improve its behavior towards eliminating the above problems. It is a routine practice to apply one finish at a time. Polyfunctional finishes is the latest attractive terminology among the textile processors which mean that more than one functional finish can be applied by a single application process.

Accessible hydroxyl groups of cellulose take an active part in crosslinking reactions which can be used to gain/introduce mechanical properties modification(7); Polycarboxylic acids appear as very promising non-formaldehyde crosslinking agents to replace the traditional, mostly formaldehyde based, compounds [8–12]. Among these acids 1, 2, 3, 4, butane tetra carboxylic acid (BTCA) has shown the best results.

In this work, polycarboxylic acids (PCAs) are used to produce functional finishes. The most successful acid in crosslinking of cellulose to impart durable press finish is 1, 2, 3, 4 -Butane Tetra-Carboxylic Acid (BTCA) in combination with citric acid. There are several other PCAs which are tried less by the research workers are subjected to trial in single and in combination to produce more than a functional finish in a single stroke of application. Traditional finishes like soft finish is also combined with poly carboxylic acid finish. The functionalities imparted on the material have been tested and reported.

Polycarboxylic acids are nowadays increasingly put under research to produce functional finishes like Durable Press finishes, they are successfully applied as alternative chemicals to formaldehyde based cross linking agents (as formaldehyde is considered to be harmful to both ecology and to human being).

However, the health risk associated with formaldehyde emission has caused increasing concern worldwide. There is a constant search for alternative chemicals which are formaldehyde-free to produce crosslinking in the cellulosic chains. It is found that polycarboxylic acids along with catalysts are very effective in producing this effect. Polycarboxylic acid can react with cellulose to form cross linking by the formation of esters (esterification)

CDs are a series of cyclic oligosaccharides of 1,4linkedD-glucopyranose units [13]. The CD molecules have truncated cylindrical cavity with a depth of а approximately 7.0 Å and internal diameters varying from 4.5 to 8.5 Å, depending on the number of structural units in the macro cycle (six, seven or eight units for the first three members of the homologue series known as alpha, beta and cyclodextrin (13) . CDs possess hydroxyl gamma functionalities on the two rims and hydrocarbon and ether moieties in the inner cavity. Thus, CDs have both hydrophobic (inner) and hydrophilic (external) faces. Linear components used to form inclusion complexes with CDs usually have two hydrophilic end groups and a hydrophobic middle part. Different inclusion complexes based on CDs and either homo- or copolymers have been prepared and investigated. Poly(ethylene oxide) [14], poly(propylene glycol) [15],poly(methyl vinyl ether) [16], polyamides [17], polyesters [18], cationic polymers [19-22], poly isobutene [23] or poly azo methane's [24-27] were considered as guest partners for CDs.

2. METHODOLOGY

Materials used for the study

The following construction used in Lyocell fabric. Warp - 40^{s} Weft - 40^{s}

Poly carboxylic Acids selected for the study: Maleic acid

Cyclodextrin: Beta cyclodextrin

The well scoured and bleached fabric samples were treated with BTCA and CD used individually and with combinations

3.1 Procedure

The below technique was used to apply the BTCA and CD. The receipe was optimized by treating with various concentrations and the results were tabulated and analysed for various tests.

Pad with an expression of 85%-Dry at 110°C for 5 minute-Cure at 150°C for 3 minute

The treated samples were studied for the following properties. The type of treatment has given below*.

- 1. Whiteness
- 2. Flammability
- 3. Crease recovery
- 4. Tensile Strength
- 5. Tearing Strength
- 6. Abrasion Resistance
- 7. Pilling Tendency

4. RESULT AND DISCUSSIONS

4.1 Effect Of Btca / Cd On Whiteness And Flammability

The whiteness index of the untreated and treated samples analysed by using computer colour matching software ; the flammability of the samples were tested by using 45° angle flammability tester for the flammability time. The whiteness index lowest value for the DMDHEU treated sample is due to the presence of amine group in the resin, the fabric turns to little bit yellow after treatment. The flammability of the treated samples with various level of concentration of BTCA &CD was not shown noticeable changes but the difference in DMDHEU treated sample shown different properties as shown in below Fig.I



Fig*. I: Whiteness & Flammability of BTCA ,DMDHEU and CD Treated & Untreated Fabric

4.1 Effect Of Btca / Cd On Crease Recovery Angle:

A rectangle specimen of prescribed dimension is folded in half and compressed under a load for a specified time. The load is then removed and the specimen used allowed to recover for the specified time. The amount recovery is expressed as the angle between the limbs of the fold which is called the crease recovery angle. Crease recovery angles were measured according to *AATCC Standard Test Method* 66 option 2, The wrinkle recovery angles were recorded as the added total of warp and

weft averages. BTCA treated for 8% along with β CD sample shows more CRA than other samples due to better crease recovery property by the cross linking agent and obviously the cyclodextrin also plays a vital role for the good recovery property.



Fig*. II: CRA of BTCA, DMDHEU and CD Treated & Untreated Fabric

4.2 Effect Of Btca / Cd On Tensile Strength:

The tensile strength of the fabric refers to its resistance to tensile force. The breaking strength of a fabric is used both for quality control and as a performance test. For industrial and other purposes were the fabrics is impact subjected to high tension, it is proper that breaking strength be measured. In case where the fabric isn't called upon to withstand large tensile force, breaking strength is often used to provide a central check on quality of a woven fabric. In this project, the tensile strength of untreated and BTCA treated fabrics was determined with a tensile strength tester according to ASTM *Test Method D 5035*. From this project, BTCA is affected tensile strength, when concentration of BTCA is increase; tensile strength of treated fabric is decrease, when compare to DMDHEU, BTCA shows less tensile strength loss due to the formation of more cross linking. Cyclodextrin not impact on the tensile testing behaviour.



Fig*. III: Tensile Strength of BTCA, DMDHEU and CD Treated & Untreated Fabric

4.3 Effect Of Btca / Cd On Tearing Strngth Test:

Tear resistance is one of the important properties of the fabric. Tear strength of a fabric refer to its resistance to tearing force. For all flat sheets like materials such as fabric, plastic films and papers, the breaking strength of the material in tension is far greater than its tear resistance. While it may be difficult to introduce a tear in any of these materials usually the tear propagated at a relatively low load. This project the tearing strength of untreated and CD / BTCA treated fabrics was determined with a Elmendorf tear strength tester according to ASTM Test Method D 1424.for this project, BTCA is affected 6-9 % of strength loss when we use 8% of BTCA, whereas DMDHEU treated fabric have strength loss is 15 to 20 %; whereas the CD is significantly affect the strength of the fabric.



Fig*. IV: Tearing strength of BTCA, DMDHEU and CD Treated & Untreated Fabric

4.4 Effect Of Btca / Cd On Abrasion Test:

Abrasion is the wearing a way of any part of a material by rubbing against another surface. Adequate abrasion resistance of textile materials is essential for customer acceptance and stratification. This Martindale apparatus is designed to give a controlled amount of abrasion between fabric surfaces at comparatively low pressures in continuously changing directions. The results of this test should not be used indiscriminately particulars not for comparing fabrics of widely different fiber composition or construction. This present study of abrasion resistance BTCA, CD treated fabrics was determined with a Martindale abrasion resistance tester according to ISO 12947-2- 1999 method. DHFHEU treated sample cause more abrasion than untreated sample and the due to less cross linking nature of CD, the sample cause less abrasion than BTCA treated sample.



Fig*. V: Abrasion resistance of BTCA, DMDHEU and CD Treated & Untreated Fabric

4.5 Effect Of Pilling Tendency

Visual evaluation of the samples revealed that the pilling of fabrics untreated lyocell was better than that of the one which underwent the various wet treatment process. Lyocell fabric laundered ten times as per the AATCC 135-2004 standard, and then observes the pilling grades. As per the ISO 12945-1 standard, the grade 5 represent the outstanding pilling resistance, as well as 1 represent the poor pilling resistance, in general the cross linking cause more pilling especially in case of DMDHEU treated sample than untreated but due to the peculiar nature of CD and BTCA such instance is not occur; however, the sample shows better pilling tendency for CD/BTCA treatment.





*Sample code

S.No	Sample	Sample code
1.	Untreated	А
2.	Treated with DMDHEU	A1
3.	Treated with BTCA 2 %	A2
4.	Treated with BTCA 4 %	A3
5.	Treated with BTCA 8 %	A4
6.	Treated with β CD 2 %	A5
7.	Treated with β CD 4 %	A6
8.	Treated with β CD 8 %	A7
9.	β CD 2 % + BTCA 2 %	A8
10.	β CD 4 % + BTCA 2 %	A9
11.	β CD 8 % + BTCA 2 %	A10



Fig.VII Untreated sample



Fig. VIII Treated sample cause fibrillation

CONCLUSION

The study conducted on the treatment of DMDHEU, BTCA and β CD on physical properties of lyocell fiber has derived the following conclusion.

Treating the lyocell with BTCA and beta cyclodextrin which could block the side chine formations, which in turn reduce the fibrillation tendency of the lyocell. Particularly when we treated the lyocell with 8% of BTCA and 2% CD will prove smooth surface. A Cyclodextrin treatment could crosslink the fiber and thereby prevents or inhibits the fibrillation of the lyocell along with BTCA. Compare to conventional DMDHEU, BTCA has numerous advantages in terms of strength loss, yellowing and other fabric property losses. However, the presence of CD causes more hydroxyl group availability on the surface of the fabric causing the more dyeability and absorbency.

REFERENCES:

- Bochek, A. M., Petropavlovsky, G. A., and Yakimansky, A. V., Solubility of Cellulose and its Derivatives, in "Cellulose and Cellulose Derivates: Physico-Chemical Aspects and Industrial Applications, J. F. Kennedy, G. O. Phillips, P. A. Williams, and L. Piculell, editors, Woodhead Publishing Limited, Abington, U. K., 1995, pp. 131-137.
- McCorsley, C. C., and Varga, J. K., Process for Making a Precursor of a Solution of Cellulose, U. S. patent 4142913,1979
- Colom, X., and Carrillo, F., Crystallinity Changes in Lyocell and Viscose-type Fibres by Caustic Treatment, Euro. Polym. J. 38 (11), 2225-2230 (2002).
- Lenz, J., Shurz, J., and Wrentschur, E., Properties and Structure of Solvent-spun and Viscose-type Fibres in the Swollen State, Colloid Polym. Sci. 271, 460-468 (1993).
- Lenz, J., Shurz, J., and Eichinger, D., Properties and Structure of Lyocell and Viscose-type Fibers in the Swollen State, Lez. Berichte 9, 19-25 (1994).
- Valldeperas, J., Carrillo, F., Lis, M. J., and Navarro, J. A., Kinetics of Enzymatic Hydrolysis of Lyocell Fibers, Textile Res. J. 70 (11), 981-984 (2000).
- Grancaric, A. M., Soljac □ic, I., and Katovic, D., "Textile Finishing II', Zagreb, Tekstilno tehnolos ki fakultet 1994
- Yang, C. Q. and Xu, L., Non-formaldehyde Durable Press Finishing of Cotton Fabrics by Combining Citric Acid with Polymers of Maleic Acid, Textil. Res. J., 68(5), 457–464 (1998).
- Welch, M., Formaldehyde-free Durable-press Finishes, Rev. Prog., Coloration, 22,32–41 (1992).
- Voncina, B., Durable Press Finishing of Cotton with Polycarboxylic Acid, Fibres Textiles East. Europe, 12, 69–71 (1996).
- 11. Welch, C. M., Formaldehyde-free DP Finishing with Polycarboxylic Acids, Amer. Dyestuff Reporter, 83, 19–26 (1994).
- Hyung, M. C., Non-phosphorus Catalysts for Formaldehyde free DP Finishing of Cotton with Polycarboxylic Acids, Textile. Res. J., 63(11), 650–657 (1993)
- 13. Szejtli, J. (2004). Present, Past, and Future of Cyclodextrin Research, Pure. Appl. Chem.,76(10): 1825–1832
- Harada, A. and Kamachi, M. (1990). Complex Formation between Poly(ethylene glycol) and -cyclodextrin,Macromolecules,23: 2821– 2823.
- Kamitori, S., Matsuzaka, O., Kondo, S., Muraoka, S., Okuyama, K., Noguchi, K., Okada, M. and Harada, A. (2000). A Novel Pseudo-Polyrotaxanes Structure Composed of Cyclodextrins and a Straight-Chain Polymer: Crystal Structures of Inclusion Complexes of -Cyclodextrin with Poly(trimethylene oxide) and Poly(propylene glycol),Macromolecules,33: 1500–1502
- Harada, A., Li, J. and Kamachi, M. (1993). Complex Formation between Poly(methyl vinyl ether) and gamma Cyclodextrin, Chem. Lett., 22(2): 237–240
- Wenz, G., Steinbrunn, M.B. and Landfester, K. (1997). Solid State Poly condensation within Cyclodextrin Channels Leading to Water soluble Polyamide Rotaxanes, Tetrahedron, 53: 15575–15592
- Kawaguchi, Y., Nishiyama, T., Okada, M., Kamachi, M. and Harada, A. (2000). Complex Formation of Poly(-caprolactone) with Cyclodextrins, Macromolecules, 33: 4472–4477
- Choi, H.S., Ooya, T., Lee, S.C., Sasaki, S., Kurisawa, M., Uyama, H. and Yui, N. (2004). pH Dependence of Polypseudorotaxane Formation between Cationic Linear Polyethylen amine and Cyclodextrin, Macromolecules, 37(18): 6705–6710.
- Wenz, G. and Keller, B. (1992). Threading Cyclodextrin Rings on Polymer Chains, Angew. Chem. Int. Ed., 31: 197–199.
- Wenz, G. and Keller, B. (1993). Synthesis of Polyrotaxanes or How to Thread Many Cyclodextrin Rings on a Polymer Chain, Polym. Prepr., 34: 62–63.
- Herrmann, W., Schneider, M. and Wenz, G. (1997). Photochemical Synthesis of Polyrotaxanes from Stilbene Polymers and Cyclodextrins ,Angew. Chem. Int. Ed., 36: 2511–2514
- Harada, A., Li, J., Suzuki, S. and Kamachi, M. (1993). Complex Formation between Polyisobutylene and Cyclodextrins: Inversion of Chain-length Selectivity between -cyclodextrin and -cyclodextrin, Macromolecules, 26: 5267–5268
- Simionescu, C.I., Grigoras, M., Farcas, A. and Stoleru, A. (1998). Synthesis and characterization of poly (azomethine)s with rotaxane architecture ,Macromol. Chem. Phys.,199: 1301–1306.

- 25. Farca, s, A. and Grigora, s, M. (2003). Synthesis and Characterization of a Fully Aromatic Polyazomethine with Rotaxane Architecture, Polym. Int., 52: 1315–1320.
- Farca, s, A. and Grigora, S. M. (2001). Synthesis and Characterization of a Fully Aromatic Polyazomethine with Main Chain Rotaxane Architecture, High. Perform. Polym., 13: 201–210.
- Farca, s, A., Grigora, s, M. and Simionescu, C.I. (2001). Poly(aromatic azomethines) Containing Noncovalently Bound Cyclodextrins in the Main-chains: Synthesis and Characterization, Rev. Roum. Chim., 46: 25–30

