

# Sustainable Coating of jute fabric for achieving moisture vapour permeable waterproof textile

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**Abstract-** Jute fabric was coated with coating formulation containing blend of solid natural rubber, polyvinyl alcohol and starch in complete absence of solvent employing a calendar coating technique to produce moisture vapour permeable waterproof jute fabric. Coating of jute with a formulation containing solid natural rubber, solid polyvinyl alcohol and tamarind kernel seed powder as starch, in the ratio of 10:8:5 in presence of a typical sulphur curing system, followed by vulcanization at 140°C for 3 h employing steam produced most balanced improvements in the properties of the coated fabric in respect of waterproofness assessed in terms of hydrostatic water column resisted by fabric, breathability as revealed by moisture vapour transmission rate, coating adhesion, tensile properties, abrasion resistance and flex endurance. Incorporation of polyvinyl alcohol makes the coating permeable to moisture vapour and such property is promoted further in presence of starch in the coating formulation. Examination of surface morphology of vulcanized coating by scanning electron microscopy revealed that poly vinyl alcohol crystals formed clusters of dimensions of only few microns in the rubber matrix. Such clusters of polyvinyl alcohol served as conduits for transportation of moisture vapour through the film of natural rubber, while acting as an effective barrier for liquid water droplets.

## INTRODUCTION

The horizon of breathable waterproof textiles is expanding rapidly to meet various demands of sophisticated lifestyle of mankind (Sen, 2001). Their ability to block rain and liquid water while allowing moisture vapour from enclosed environment and/or sweat leads to their use in rainwear, waterproof outdoor sports clothing, tents, construction industry and in many other applications. Moisture vapor permeable or breathable waterproof textile is characterized by its ability to block liquid water while allowing moisture vapor from sweat or other sources to pass through them (Sen 2007). The average size of water vapor is in the range of  $4 \times 10^{-5}$  micrometers and that of liquid water is 100 micrometers (Mukhpadhyay and Midha, 2008). Most such breathable waterproof textile contains polymeric coating or membrane with micropores having a size in between the two, thus allowing moisture vapour to pass through it while arresting liquid water. Typical waterproof fabrics are characterized by high order of water proofness, capable of resisting 90-130 cm of water column, when assessed employing a hydrostatic waterhead tester (BIS : 5915, Fung 2002) depending on one or both sides of the fabric being coated with waterproof coating. Among the different technologies reported for producing such moisture vapour permeable waterproof fabric, technologies based on micro-porous poly-tetra-flouro-ethylene (GORE-TEX), and micro-porous coatings based on polyurethane appear to be prospective in this respect (Horrocks, 2000). However, most of them suffer from one or more of the following disadvantages such as, use of complicated high-end technology, solvent-based process, high cost and non-biodegradability of the coating and/or lamination produced. Polyvinyl alcohol film is known for its high-water vapour permeation, water soluble salt permeability and high-water vapour selectivity from a physical mixture of water vapour and nitrogen (Chen, 2014 and Merkel, 2000). Polyvinyl alcohol is the simplest homo-polymer used for different biomedical applications such as, wound dressing, wound management, drug delivery system, artificial organ and contact lenses (Elbadawy, 2015). In such homogeneous films of polyvinyl alcohol, transfer of moisture is reported to take place by pure diffusion mechanism, which restricts the rate of absorption and size of the species that are absorbed. Dissolution of hydro-gel of polyvinyl alcohol in water is reported to be prevented, if they are cross linked or is bound in a highly cross-linked matrix. Hydro-philicity and moisture vapour transport property of such polyvinyl alcohol can be improved by incorporation of polysaccharide in the system that makes also the polyvinyl alcohol polymer system less stiff and more extensible in the presence of moisture. Present article reports studies on the role of polyvinyl alcohol in the blend of natural rubber, when such blend was made in complete absence of solvent and water in solid form and applied as coating polymer on jute fabric, in promoting moisture vapour permeability and waterproofness of the coated fabric

## EXPERIMENTAL

### Materials

Mill made plain weave jute fabric with 46 ends/dm, 54 picks/dm, 207 tex warp and 207 tex weft and having an average areal density of 260 g/m<sup>2</sup> were used for this study.

97% hydrolyzed polyvinyl alcohol obtained from Ms. Loba Chemical Private Ltd; India was used without any purification or treatment. ISNR-5 solid natural rubber obtained from Ms. N. Shashikant & Co, India was used. Commercial grade tamarind kernel seed powder as the starch was used in our study. All other chemicals including Paraphenylene diamine (antioxidant) from KK India Pvt, Zinc oxide form Vijaya Enterprise, mercapto-benzo-thio-urea (MBT) from N. Shashikant & Co., Stearic acid from

Alpha Chem., sulphur from Devanshi Corp., Paraffin wax, China clay from M M Industries, Spindle oil from Nexton Lubricants used in this study are of general-purpose reagent grade.

## Methods

### *Compounding of solid rubber*

Compounding of natural rubber was done in a two roll mixing mill in absence of any solvent by mixing thoroughly natural rubber (100PHR), PVA (0- 80PHR), starch (50PHR), china clay( 30 PHR), mercapto benzothium (MBT 1.5 PHR), Sulphur (2PHR),zinc oxide (5 PHR),stearic acid(1 PHR), paraphenyl diamine( 1.5 PHR),Parafin wax( 0.5 PHR) and spindle oil (2 PHR).

### *Application of solid rubber compound on jute*

Application of Homogeneous solid rubber compound and the coating formulations so prepared, as described above was done on one sided jute fabric employing a Calendar coating machine. The coated jute fabric was subsequently dried at 140°C for 3h in a steam heated autoclave and was considered for characterization.

### *Determination of waterproofness*

Waterproof-ness of the coated cotton fabrics were determined in a hydrostatic water-head tester following a method prescribed in ASTM D 3393-91(1997).

### *Determination of Waterproof-ness by Cone Test*

Waterproof-ness of selected coated fabrics were also assessed by Cone test following the test method prescribed in IS: 2789; 1964.

### *Determination of tensile strength,*

Determination of tensile strength and extensibility of the uncoated and coated fabrics was done in a Zwick 1445 CRT Universal Tensile Testing Machine according to a method prescribed in ASTM D5034-09(2013). The results obtained are based on an average of 10 tests performed in the warp direction of each sample.

### *Determination of tear strength*

Tear strength of the specified fabric samples were determined following ASTM D 1922(2015) employing an Elmendorf tear strength tester.

### *Determination of moisture vapour permeability*

Moisture vapor transmission rate of the coated sample was determined following a desiccant method in accordance with a method prescribed in ASTM D 7790-12. The results were expressed in terms of g of moisture vapour transported through an area of 1 m<sup>2</sup> of coated fabric over a period of 24 h.

### *Determination of moisture vapour transmission rate*

Moisture vapour transmission rate of selected coated samples was also performed employing a modulated infrared sensor in Systech7000, USA following a method prescribed in ASTM F124906. The results obtained are average of 5 such tests done for each sample.

### *Accelerated ageing Test*

For determination of accelerated ageing test, selected coated samples were placed in an oven and kept at a temperature of 90°C for 120 h following a method, prescribed in ASTM D 573-04(2015). After accelerated ageing, the test samples were examined for development of crack, softness, stiffness, tackiness, brittleness and loss of other rubber like properties of the coating.

### *Flexibility test*

Flexibility of the coated samples was determined following a flat-loop method in accordance with a method prescribed in ISO 5979:1982.

### *Flex endurance test*

Flex endurance test of selected coated samples were performed employing De Matia flexing equipment by subjecting the coated sample to repeated flexing following a method prescribed in IS 5914: 1970.

### *Scanning electron microscopy:*

Scanning electron micrographs of the selected coated samples were obtained employing a scanning electron microscope ZWISS EVO18, England in vacuum with a magnification ranging from 5000 to 50,000.

## RESULTS AND DISCUSSION

### *Effects of variation of blend composition of natural rubber solid & solid polyvinyl alcohol on waterproofness & moisture vapor permeability properties of fabric; coating thickness: 0.45 mm*

Table 1 Effect of variation of application dose of solid polyvinyl alcohol in the solid blend of natural rubber and PVA on waterproofness and moisture vapour permeability properties of coated jute; coating thickness: 0.45 mm

Natural rubber Solid ISNRV	PVA (Solid)	Starch (TKP)	Waterproof cone test (Residence time)	Waterproofness hydrostatic water head test (cm)	Moisture vapour transmission rate (g/m <sup>2</sup> / 24h)
100	0	50	86 h	110	0-2
100	10	50	86 h	110	46
100	20	50	86 h	110	108
100	40	50	86 h	105	926
100	60	50	86 h	97	1063

**100	80	50	86 h	93	1765
100	100	50	75 h	87	1878
100	120	50	72 h	80	1962

Data in table 1 show effects of variation of blend composition on water-proofness and water vapour permeability of the coated jute fabrics. Waterproofness of the coated sample was assessed following two different methods viz. cone test and hydrostatic water head test. Each coating composition as detailed in table 1 has 50 PHR of TKP included in it. Jute fabric coated with the blend of only solid natural rubber and TKP show appreciable waterproofness when assessed following a cone test and hydrostatic water head test with little or no moisture vapour permeability as evident from the data in table 1. With the introduction of poly vinyl alcohol in the coating formulation there was notable improvement in moisture vapour transmission rate of the coated cotton fabric. With increasing dose level of poly vinyl alcohol moisture vapour transmission rate followed a steady increasing trend over the entire range of different dose levels of poly vinyl alcohol considered in our study. Such increasing trend of moisture vapour transmission rate of the coated jute fabric observed with the use of increasing dose level of poly vinyl alcohol in the coating composition indicates that the poly vinyl alcohol has the potential to transport water vapour efficiently, even when bound in the cross-linked vulcanized matrix of natural rubber solid. Polyvinyl alcohol has been reported to have low water binding energy (Eurotex, 1994). As a result, it acts as a conduit allowing passage of water vapour selectively through it under concentration gradient by frequently breaking and effectively reforming hydrogen bond with the water molecules. Such conduits are too small in dimension to allow passage of water droplets and hence act a barrier for them. It also appears from the table 1 that only poly vinyl alcohol in the matrix of natural rubber solid in absence of TKP as starch, failed to transport moisture vapour through the coated film of jute. Presence of TKP in the matrix therefore facilitates the process of transfer of moisture vapour through the coated fabric.

Waterproofness of coated jute fabrics appears to remain high when assessed in terms of resistance offered to hydrostatic water head and also when assessed following cone test for increasing dose level of poly vinyl alcohol in the coating formulation up to about use of 80 PHR of poly vinyl alcohol. For use of poly vinyl alcohol beyond a dose level of 80 PHR, waterproofness suffers adversely when assessed following both the methods, as shown. Poly vinyl alcohol forms clusters of its monoclinic orthorhombic crystals in the cross-linked natural rubber solid which are of few microns and far less in size than that of liquid water drop i.e. 100 microns. When such clusters of poly vinyl alcohol are held in the cross-linked matrix of natural rubber solid, they can allow water vapour to pass through them, the average size of which is 0.004 micron. When poly vinyl alcohol is used at a level beyond 80 PHR in the blend of poly vinyl alcohol & natural rubber solid, such clusters of poly vinyl alcohol grow in size so much so, that their dimension becomes close to that of the minimum dimension of water molecule. In view of above fact, coating formulation containing more than 80 PHR of poly vinyl alcohol are capable of allowing absorption and transmission of liquid water through them, making such coating permeable to liquid water. The fall in waterproofness and swelling of the coating of the coated cotton fabric for use of poly vinyl alcohol beyond the dose level of 80 PHR, in the coating formulation may be understood and interpreted keeping, such fact in view.

#### *Effects of variation of blend composition of natural rubber solid and poly vinyl alcohol on the physical properties of cotton fabric.*

Table 2 Effect of variation of blend of Natural rubber solid & polyvinyl alcohol solid in coating composition on strength and coating adhesion of coated jute fabric; coating thickness 0.45 mm; coating composition contains 80 starch (TKP) in each application

Natural rubber Solid ISNRV	PVA (Solid)	Tensile strength (N/cm)	Tear strength (N)	Coating adhesion (N/5cm)
0	0	163	76.58	-
100	10	174	66.04	90.42
100	20	196	63.71	94.17
100	40	198	57.02	97.63
100	60	210	51.55	100.41
**100	80	215	48.55	102.70
100	100	221	43.14	103.62
100	120	234	40.11	104.33

Data in table 2 show the effect of variation of blend composition of coating, on physical properties of coated jute fabric. There is an enhancement of tensile strength of the jute fabric on coating with natural rubber solid; introduction of polyvinyl alcohol at a dose level of only 10 PHR in the coating composition caused further enhancement in the tensile property of the jute fabric. Tensile strength of the coated jute fabric however follows an increasing trend with use of increasing dose level of polyvinyl alcohol in the coating composition, as shown in table 2. Polyvinyl alcohol is known for its strong and tough film forming ability and, in this case, the strength of such film played its role, in addition to the contribution of cross-linked film of natural rubber solid in promoting tensile strength of the fabric.

Tear strength of the jute fabric follows a steady decreasing trend with the use of increasing dose level of polyvinyl alcohol in the coating formulation. Tear strength of the jute fabric appears to suffer adversely on coating, due to limitations imposed on

structural deformability of the woven jute fabric for coating. Inclusion of enhanced dose levels of polyvinyl alcohol in the coating formulation appears to impose an enhanced degree of such limitation on woven structure deformability, due to the enhanced tackiness of the coating formulation. Such coating formulation with enhanced tackiness when applied on jute fabric becomes responsible for holding the yarns in position, in a more efficient manner, without allowing them to slip as would otherwise required by the fabric structure for offering resistance against tearing.

Coating adhesion of the coated jute fabric follows an increasing trend with the use of increasing dose level of polyvinyl alcohol in the blend of natural rubber solid polyvinyl alcohol coating formulation. Such increasing trend in coating adhesion observed for use of increasing dose level of polyvinyl alcohol in the coating formulation, is due to the enhanced degree of tackiness of the coating formulation observed for use of enhanced dose level of polyvinyl alcohol.

***Effect of variation of blend composition of natural rubber solid and polyvinyl alcohol on, flex endurance, flexibility, accelerated ageing and abrasion resistance of the coated jute fabric***

**Table 3. Effect of variation of blend composition of coating on some mechanical properties of coated jute fabric; coating composition contains 80PHR starch (TKP) in each case**

Natural rubber Solid ISNRV	PVA (Solid)	Flex endurance (no of cycles)	Loop height (cm)	Accelerated ageing	Abrasion resistance weight loss (mg)
100	0	88,712	<b>2.9</b>	No appreciable change	53
100	10	62,214	<b>3.6</b>	-do-	41
100	20	50,043	<b>3.6</b>	-do-	30
100	40	48,943	<b>4.0</b>	-do-	21
100	60	42,544	<b>4.0</b>	-do-	0
**100	80	39,641	<b>4.5</b>	-do-	0
100	100	36,434	<b>4.5</b>	-do-	0
100	120	31,563	<b>4.8</b>	-do-	0

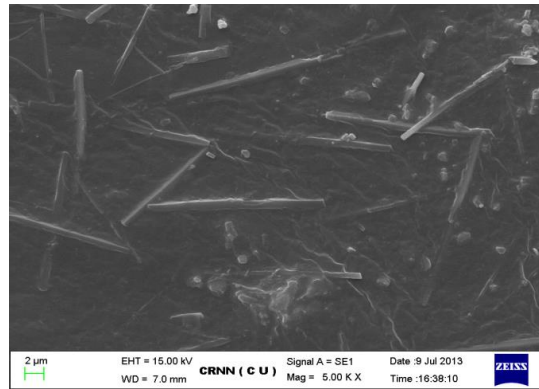
Flex endurance of the jute fabric coated with only natural rubber solid appears to be sufficiently high (88,712) as evident from the data in Table 3. Incorporation of stiff polymer of polyvinyl alcohol in the coating formulation to an extent of only 10 PHR, however, resulted in substantial lowering of the flex endurance of the coated jute fabric producing visible cracks and crevices on the surface of the coated jute fabric. With the use of increasing dose level of polyvinyl alcohol thereafter in the blend of natural rubber solid and polyvinyl alcohol, flex endurance appears to follow a decreasing trend although the rate of lowering of flex endurance is observed to be much less for increased presence polyvinyl alcohol in the blend composition in the range of dose levels of 10 to 120 PHR, as evident from the data in Table 3.

Flexibility as revealed in terms of loop height of the jute fabric coated with only natural rubber solid is somewhat lowered for incorporation polyvinyl alcohol in the coating formulation. Presence of polyvinyl alcohol in the blend of natural rubber solid and polyvinyl alcohol makes the coated jute fabric stiff, as evident from the data in table 3. However, flexibility of the coated jute fabric appears to remain unaltered for use of increasing dose level of polyvinyl alcohol in the blend for the entire range of polyvinyl alcohol used in the study (Table 3).

Accelerated ageing test of the jute fabrics coated with only natural rubber solid and blends of natural rubber solid and polyvinyl alcohol of varying blend compositions shows no appreciable change, when examined for development of softness, stiffness, tackiness, cracks and brittleness of the coated layer indicating that, the coated jute fabric developed is of practical significance and use. Abrasion resistance of the coated jute fabric follows an increasing trend with the use of increasing dose level of polyvinyl alcohol in the coating composition. Incorporation of smooth tenacious, tough and strong film of polyvinyl alcohol in the blend of natural rubber solid brings about the apparent change in the property of abrasion resistance of the deposited coating film of jute fabric.

***Scanning electron microscopy:***

Coating of blend of natural rubber and polyvinyl alcohol in absence of water or any other solvent on jute resulted in a coating layer on the fabric that shows distinct presence of clusters of polyvinyl alcohol having unseparated molecules of polyvinyl alcohol in natural rubber matrix, Figure 1a,b. Unlike blend of natural rubber latex and aqueous solution of polyvinyl alcohol, [Das et al., 2016] in this case in the solid mixture or blend used, many polyvinyl alcohol molecules remain in groups joined with each other by H-Bond, which appear to remain un-separated during mechanical shear force applied during mixing of only solid rubber in a two roll mixing mill. As a result, distinct presence of unbroken groups of polyvinyl alcohol appear to remain dispersed in the solid rubber matrix which are of the order of few micrometer only and are far less than the minimum size of liquid water. They being smaller than the minimum size of liquid water are incapable of transferring liquid water through them as exhibited by appreciable waterproofness of the coated jute fabric.



(a) Fig 1. Scanning electron micrograph jute fabric coated with blend of solid natural rubber and polyvinyl alcohol at 5000 x .

## CONCLUSION

The appropriate coating of jute fabric with blend of natural rubber solid, polyvinyl alcohol and in total absence of solvent and water following a calendar coating technique in presence of a typical sulphur curing system establishes a simple and sustainable route for achieving moderately performing moisture vapour permeable waterproof coated textile. The major property advantages that can be derived from such a coating system are i) moderate to good level of water vapour permeability, ii) appreciable waterproofness iii) high coating adhesion, iv) enhanced tensile strength, v) high flex endurance and vi) high abrasion resistance of the coated fabrics. Examination of surface morphology of such coating reveals that under optimum condition of coating exhibits clusters of polyvinyl alcohol of appropriate size held in the matrix of cross-linked natural rubber, that act as conduit for selective transfer of only moisture vapour through them while acting as barrier for liquid water droplet.

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