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Effect of ethylene acrylic acid co-polymer coating on bending and thermal properties of cotton fabrics

Gurumurthy B. Ramaiah [a,*], Seblework Mekonnen [a], Eshetu Solomon [a], Pradeep Kumar [b], M.P. Dhananjay Kumar [b], G.M. Nagesh [b]

[a] *Federal TVET Institute/University, Addis Ababa, Ethiopia*
[b] *VFSTR (Deemed to be University), Guntur 522213, India*

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ABSTRACT

Bleached cotton fabric and a water-based polymer coating solution made from ethylene acrylic acid co-polymer which is multifunctional polymer material is used in this experimental study to increase the bending and thermal properties of cotton fabrics. Roller coating and Exhaust methods is used to apply ethylene acrylic acid co-polymer solution on the cotton fabric surface. The coated fabric surface is then cured and dried at a temperature of 60–90 °C using a laboratory oven. The developed fabric samples are then taken for testing for its bending and thermal properties using a fabric stiffness tester and DSC thermal analyzer. The chemical composition and its chemical group finger print analysis is also ascertained using FTIR spectra. The fabric stiffness results of coated fabrics show increased bending properties and the thermal property results from DSC and plotted graphs prove its robustness and resistance to heat. However, Roller coating method seems to be more efficient than exhaust method with respect to the application method employed for coating ethylene acrylic acid co-polymer.

1. Introduction

A water-based polymer coating solution, namely ethylene acrylic acid co-polymer which is multifunctional in nature is coated on bleached cotton fabric with an objective to enhance its bending and thermal properties in this study. The bleached cotton fabric is coated with ethylene acrylic acid by padding and exhaust bath process. In this experimental study, small soft rollers are used for padding the cotton fabrics using ethylene acrylic acid solution. Ethylene acrylic acid is multifunctional co-polymer and is mainly used for textile fabric coatings and many other applications which are of medicinal value, including packaging substrates. This liquid forms a thin film and binds uniformly to the substrate when applied on the surface externally. The liquid also finds many applications in cosmetics and commercial polymer thin film products. The liquid is colorless, with no foul odor and hence is used in many textiles and non-woven textiles. The solution has excellent sealing, adhesion properties coupled with its flexibility and special hardness [1].

Coating formulation and its application on fabrics is a complicated process. Many chemicals and polymer additives are used for coating, since adhesion and durability properties are also important for a coated fabrics. The quality of coating is also determined by the coating method and machinery used for coating. There are many techniques of coating that are used in finishing process. Some of them are done using knife-over-roller method, particularly used for polymer coatings that are solvent based, dip-coating for variety of water-based polymers and fabric treatments [11,12,18]. The chemical formulations are based on the functional properties that is desired and the level of ethylene acrylic acid is also determined keeping the health hazard parameters that ethylene acrylic acid can cause to human skin [13,14]. Higher stiffness is a desirable property in some of the cotton fabrics. Bending of fabrics is a major contributory function on the fabric stiffness behavior. However, the stiffness of the fabrics can be improved if the bending property is minimized. This mechanical property contributes to the comfort and drapability of textile fabrics. Hence, achieving stiffness in fabrics, using fabrics with low stiffness levels, is of great importance when it comes to designing

* Corresponding author.
E-mail address: voguru1@gmail.com (G.B. Ramaiah).

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a fabric for soft feel usages. In many cases, increased stiffness is desirable to improve its draping characteristics. One way to achieve the desired level of stiffness in cotton fabrics is to pad the fabric using ethylene acrylic acid liquid. The padding is then followed by curing the fabric and drying, which would result in better stiffness property [2].

In this research the application of ethylene acrylic acid solution is coated on cotton fabrics using roller coating and exhaust method. Pad-dry-cure and exhaust method of application is primarily used in this study for comparing the thermal and Mechanical properties of the coated fabric. The applied ethylene acrylic acid solution reacts with the surface layer of cotton fabric, resulting in formation of a thin layer and renders the fabric a stiffer feel, finally resulting in improved bending stiffness and thermal properties [3,4]. The ethylene acrylic acid co-polymer is made by treating Acrylic acid and ethylene through polymerization technique involving catalytic reaction. In this study the cotton fabrics is coated with ethylene acrylic acid by roller coating and exhaust method. The coated fabric surface characteristic features and the variations in flexural and thermal properties is measured, observed and discussed to assess the effect of ethylene acrylic acid co-polymer when applied on cotton fabrics [5–7,16,17]. The ethylene acrylic acid coated fabric is tested for its thermal behavior and fabric stiffness properties using DSC, TGA and cantilever stiffness tester. The roller coating method has higher thermal resistance values and stiffness values as compared to exhaust method of application.

2. Experimental details

2.1. Materials and methods

2.1.1. Ethylene acrylic acid

The ccommercially available ethylene acrylic acid, which is sold in the form of beads or in solution form (Fig. 1). They are made using acrylic acid, being polymerized with ethylene compound. Fig. 1 shows the characteristic appearance of ethylene acrylic acid co-polymer beads [8,9]. The liquid form of ethylene acrylic acid has an acrylic content of 20% and is melt spun having melt index of 300 g/10 min. The compound is found to have a density of 0.91–0.94 g/cm^3.

2.2. Synthesis and coating of ethylene acrylic acid co-polymer

Suspension or emulsion polymerization method in the presence of free-radical catalyst is used for making ethylene acrylic acid co-polymer. The monomer makes to 5–20% of the co-polymer, which is the acrylic acid compound. It is able to produce ethylene acrylic acid beads of micron size ranging approximately from 10 to 1000 μm by using suspension polymerization technique. Dibenzoyl peroxide is added as an initiator during polymerization to make the monomer water-soluble.

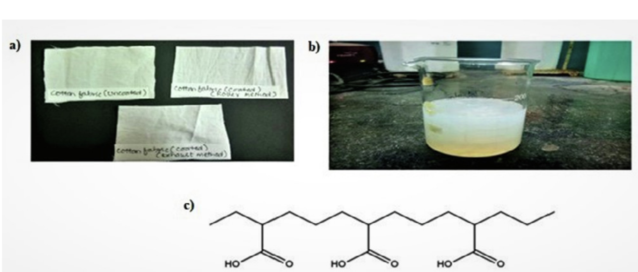

Fig. 1. a) Ethylene acrylic acid copolymer coated fabric b) Ethylene acrylic acid co-polymer solution c) Ethylene acrylic acid chemical structure.

The result of this polymerization is a heterogeneous polymer solution (Fig. 1b, c). Stabilizers like PVA, Cellulose, Gelatin, etc. are also added during polymerization process so that they help in formation of beads, which are separated after filtering and water washing. Fig. 1a shows the samples of cotton fabrics used in this study. The pretreated bleached cotton fabrics which are impurity free is taken for coating [10].

2.3. Methodology

Fig. 2 shows the steps followed in this experimental study. Firstly, the cotton fabric which is bleached and free from external impurities and process contamination like stains, fabric defects, etc., is washed and dried using a washing machine with addition of 3% of standard detergent on weight of the sample. The ethylene acrylic acid co-polymer is taken in a small beaker and stirred well so that the prepared solution does not clot on the surface of the fabric during coating process. The exhaust bath is set and the calculated amount of ethylene acrylic acid solution is added to the finish bath. However, in case of roller coating process the solution is used directly by padded on the surface of the fabric using soft small soft rollers, then dried, and cured in an oven. The finished coated fabrics are then taken for observing and testing surface morphology analysis using Scanning Electron Microscopy, Fabric bending tester and DSC thermal property analyzer [15].

2.4. Fabric construction parameters

The cotton fabric sample construction particulars before and after treatment with ethylene acrylic acid liquid by using exhaust coating and roller coating methods is displayed in Table 1. From Table 1 it can be observed that there is no change in the count of the warp and weft yarn. However, ends per inch and picks per inch of the coated cotton fabric samples show about 23% increase in the thread density in warp and weft directions.

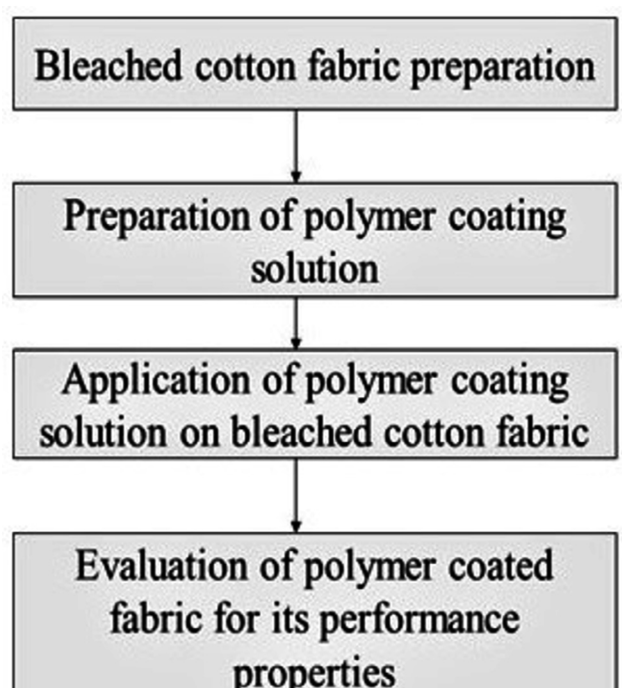

Fig. 2. Methodology followed in this study.

Table 1
Fabric construction particulars.

Test Sample	Warp count (Ne)	Weft count (Ne)	EPI	PPI
Uncoated fabric	50	50	143	110
Exhaust method coating	50	50	150	143
Roller method coating	50	50	150	143

2.5. Cotton fabric preparation

Fabric samples which were bleached and made from 100% cotton is used for coating with ethylene acrylic acid co-polymer by roller coating and exhaust coating process. The samples of 20″ × 20″ is washed in soap solution and followed by drying for 5 h. The samples were carefully prepared after thorough checking of surface stains and impurities on the cotton fabrics. The sample shape was rectangular and contained no surface additives or finishes. In case of pad-dry-cure process, the sample size was slightly bigger with dimension of 30″ x 20″.

2.6. Application of ethylene acrylic acid solution by exhaust method

The recipe used for application of ethylene acrylic acid co-polymer on bleached cotton fabrics is discussed below.

a. Material to Liquor Ratio(MLR) : 1: 40
b. Quantity of Ethylene acrylic acid used : 20gpl
c. Temperature Ranges : Start temperature – 30 °C, End temperature – 90 °C
d. Time Duration : 1 h 30 min
e. Exhaust method finish cycle (Fig. 4): Absorption – Penetration – Increasing the temperature to 90 °C. Cold-water cooling, washing and neutralizing.
f. Roller coating process finish cycle: Pad- Dry-Cure operations

The bleached cotton fabrics is treated with ethylene acrylic acid co-polymer in a set-up bath containing 20 g per liter of ethylene acrylic acid at a start temperature of 30 °C. The temperature then is raised to 90 °C and the sample is worked for a period of one hour. The samples are then cooled and quenched in a running tap water and finally neutralized with cold-water wash. The finished samples are then dried and cured in an oven at 60 °C (Fig. 3). The cotton fabrics are then taken for conditioning before subjecting them to surface scanning, bending tests and thermal analysis.

2.7. Roller method of finish application

The bleached cotton fabric samples are placed on a flat surface and stretched well and pinned at its four corners. Care is taken to make sure the edges is secured using cellophane tape to prevent the formation of creases on the surface of fabric. A similar recipe used for exhaust method of application is also used for roller coating process. 5 times to and fro, strokes is repeated for padding the solution of the surface of the fabric. The padded cotton fabric is allowed to dry for 15 min in open and then cured in a hot air oven at 60 °C for 30 min.

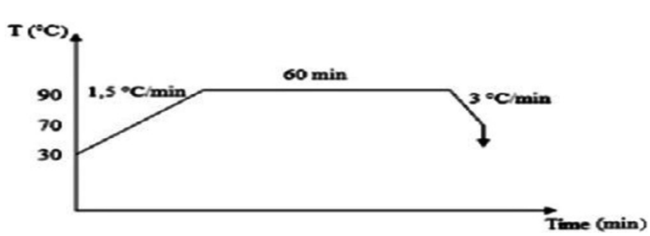

Fig 3. Finish Cycle Temperature- Time diagram.

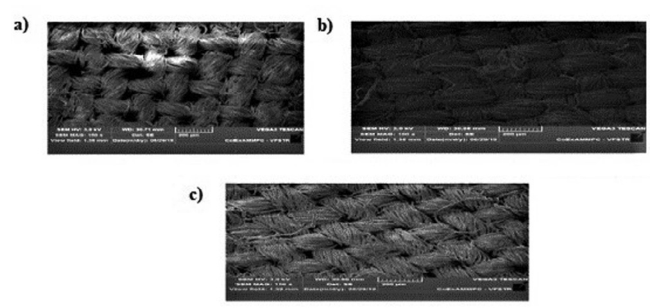

Fig. 4. a) SEM micrograph of bleached cotton fabric b) SEM micrograph of exhaust method coated cotton fabric c) SEM micrograph of roller method coated cotton fabric.

2.8. Drying and curing of coated cotton fabrics

After application of ethylene acrylic acid co-polymer on bleached cotton fabric, the coated fabric is then subjected to drying and curing. In this process of drying and curing the cross linking of ethylene acrylic acid co-polymer takes places with the surface molecules of bleached cotton fabric. The cross-linking forms strong bonds on the fabric surface and hence ensuring good fastness of the finished fabric. Laboratory hot air oven is used to dry and cure the coated fabrics.

2.9. Coating parameters calculations

The coating parameters play an important role in the application of ethylene acrylic acid co-polymer solution on bleached cotton fabrics. These coating parameters indicates how much quantity (percentage) of ethylene acrylic acid co-polymer is taken up by bleached cotton fabric during exhaust and roller coating process. The percentage coatedand percentage increase in thickness (mm) is calculated using the formula as shown in (Eqs. (1) and (2)). The bleached cotton fabrics coating data is shown in Table 2

$$\%Coated = \frac{Coated\ fabric\ weight - Uncoated\ fabric\ weight}{Coated\ fabric\ weight} \times 100 \tag{1}$$

$$\%\ Increase\ in\ Thickness(mm) = \frac{Coated\ fabric\ thickness(mm) - Uncoated\ fabric\ thickness(mm)}{Coated\ fabric\ thickness(mm)} \tag{2}$$

Table 2
Bleached cotton fabrics coating data.

Test sample	Weight (gsm)	% coating
Uncoated fabric	104	
Exhaust method coating	108.9	3.81
Roller method coating	136.9	30.5

2.10. Change in thickness of coated fabrics

Change in thickness of coated fabrics is measured as per ASTM D 1777 standards. The thickness of the coated and uncoated fabrics is reported in Table 3. The digital thickness measuring apparatus used for measuring the thickness of the samples.

From the data of change in thickness (Table 3), the change in thickness is prominent in roller coating process. However, in the exhaust coating process the change in thickness was reported to be at 10%. In case of roller coating method, the fabrics showed about 50% increase in the thickness underlining the fact that the roller coating method is more effective and the fabric has sufficient area which can absorb and facilitate the penetration of the coating solution and forming cross-linking bonds with the substrate.

2.11. Scanning electron microscopy of coated fabrics

Scanning electron microscope of TES SCAN VEGA – 3 make was used for surface image capturing of the coated and uncoated fabrics. The scanning electron microscopic tests were conducted under different levels of resolution and magnification. The samples were well conditioned and tested for its surface features as per the standard operating procedure laid out for scanning electron microscopes. Fig. 4 shows the scanning electron micrographs of the cotton coated and uncoated bleached cotton fabrics.

2.12. Measurement of thermal property of coated fabrics

In order to ascertain the stability of the coated bleached cotton fabrics using ethylene acrylic acid, the samples was subjected to thermal analysis. DSC (Differential Scanning Calorimetry) and TGA (Thermo Gravimetric Analysis tests is carried out on both the uncoated and coated fabrics. Based on the results obtained from these test, suitable inferences is drawn in the results and discussions section.

2.13. Differential scanning calorimetry (DSC)

In the Differential Scanning calorimetry experiment the coated and uncoated bleached cotton, fabric samples was tested for the heat flow rate with a reference standard. The equipment was used was Hitachi HTG, Japan, Exstar DSC7020. The Instrument readings and graphs plotted from the instrument output is discussed in the results and discussion section. The DSC data also gives information on various thermal parameters like heat of fusion, crystallization temperature, melting temperature, etc.

2.14. Thermogravimetric analyzer (TGA) and Differential thermal analyzer (DTA)

The rate of change in mass and the amount of mass loss relationship is plotted from the data obtained from Thermogravimetric analyzer. The coated and uncoated cotton samples was also subjected to standard tests on a Thermogravimetric Analyzer (TGA). The change in temperature of the specimens with reference to heating and cooling is one test that indicates the thermal behavior of the samples when heat is applied and withdrawn. The Differential Thermal Analyzer gives the relevant data that can be used to infer relations on application of heat and relative sample behavior. STA7200, Hitachi HTG, Japan, TGA and DTA analyzers is used to record the thermal data of coated and uncoated bleached cotton fabrics. The TGA data give information on the thermal stability behavior of the samples.

Table 3
Change in thickness data of cotton fabrics.

Test sample	Thickness (mm)	% Increase
Uncoated fabric	0.213	
Exhaust method coating	0.235	10.32
Roller method coating	0.321	50.7

2.15. FTIR tests for coated fabrics

The coated and uncoated fabrics was also subjected to identify and analyse the functional groups that are present. Cary 630 FTIR spectrophotometer was used to record and interpret the peaks, which correspond to functional groups present in the coated and uncoated samples.

2.16. Fabric stiffness property measurement

ASTM standard D1388 is used to measure the bending property of both the coated and uncoated fabrics. The fabric stiffness tester is used to measure the bending length of the samples. The test apparatus works on cantilever principle and makes use of a rectangular strip sample of dimension $6'' \times 1''$. The strips are mounted on a horizontal platform and pushed in such a way that the fabric bends on its own weight. The end on the mirror coincides with the bending length. This is recorded as bending length L. The flexural rigidity of the sample is calculated using standard equation (Eq. (3)) as given below

$$\textit{Flexural rigidity}\,(G) = 3.39\,W_1c^3\,\mathrm{mg/cm}\;\textit{or}\;W_2c^3 \times 10^3\,\mathrm{mg/cm} \quad (3)$$

where

W_1 = Cloth weight in ounces per square yd
W_2 = Cloth weight in gms per sq cm

3. Results and discussion

3.1. Scanning electron micrograph observations

In the micrographs (Fig. 4) obtained from scanning electron microscopy, the woven patterns are visible only in uncoated material. The presence of ethylene acrylic acid co-polymer masks the surface and make the patterns of woven fabric appear hazy, which is due to the deposition of ethylene acrylic acid co-polymer on the surface of bleached cotton fabric. The visibility of woven patterns is less in roller-coated samples due to higher absorption and fixation of ethylene acrylic acid co-polymer.

3.2. Thermal property analysis

All the samples used in this study show endothermic behaviour when subjected to heat. This behaviour is studied using differential scanning calorimetry data for both the methods of application of ethylene acrylic acid (Fig. 5). The endothermic curve shows melting peak around 90 °C. The onset of endothermic process maybe due to the evaporation of water molecules present in the cotton fabrics and ethylene acrylic acid coated material. Similar peaks are observed in all the samples. Further the curve shows exothermic peak at around 400 °C and decomposes. The extrapolated curve at this point is used to drop a tangent to obtain the point of maximum slope (Rise/fall) helping in interpreting the calculating the amount of heat evolved. The peak length of the fabrics coated using rollers is found to be very different as compared to other fabrics. The roller coated fabric curve proves to show slightly better thermal stability as compared to exhaust method of coating.

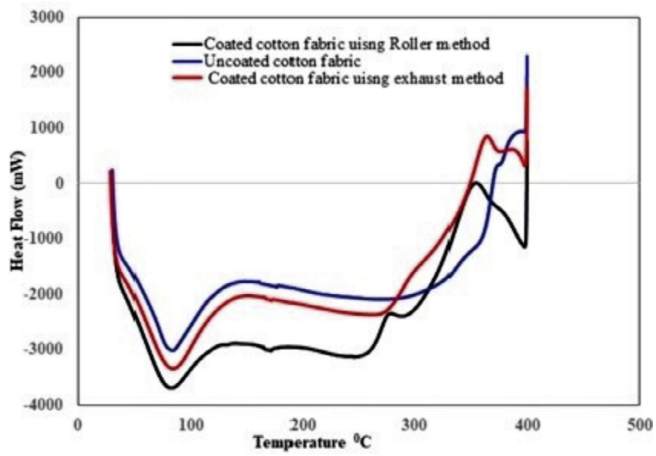

Fig. 5. DSC Graphs of samples.

This differential thermal behaviour of coated and uncoated fabrics may be accounted due to change in the molecular structure and thermal transitions happening in both coated and uncoated fabrics. In Figs. 6 and 7, the samples derivative thermogravimentry plots (DTG & TGA) show well-defined peaks. The peaks clearly indicate the thermal decomposition stages. Higher thermal decomposition temperatures is reported for coated woven fabrics. All the DTG plots of different samples how different behaviour. The DTG graphs of samples show the peaks at decomposition temperatures starting at 300 °C.

Here again the fabric coated using roller methods shows lower peak indicated lower weight loss as compared to exhaust method and uncoated fabric whose peaks are seen overlapping. However, in the TGA plot, the weight loss % is rapid after 250 °C.

In the TGA curve (Fig. 7) we also can see the weight loss % is relatively higher for uncoated fabric as compared to coated fabrics. This transition may also be absorbed at 300 °C. However, the weight loss % suddenly drops to 15% when the heating temperature reaches 380–400 °C. All the 3 samples seems to be stable till 350 °C till the onset of the slope of DTG. The coated fabric curves drop early as compared to uncoated fabrics which may be due to melting of ethylene acrylic acid co-polymer. In Fig. 8, the differential thermal analysis plots (DTA) show transition peaks at 230 °C and 425 °C. This indicates the various events that are associated with thermal behaviour of the coated and uncoated fabrics.

At 425 °C, the coated fabric transition events are prominently observed. The peak at 300 °C indicates the stable behaviour of roller coated fabric. The peaks indicate the onset of structural

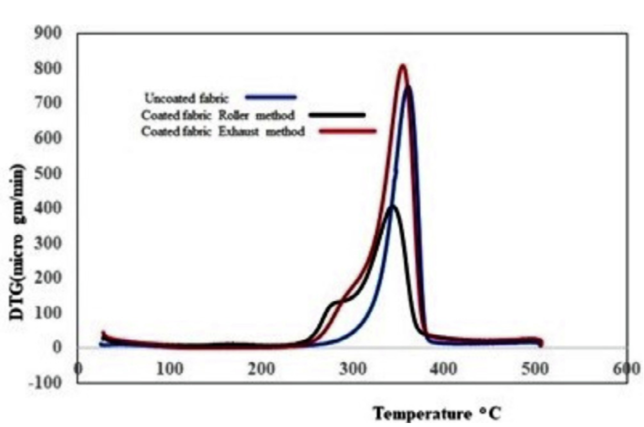

Fig. 6. DTG Graphs of samples.

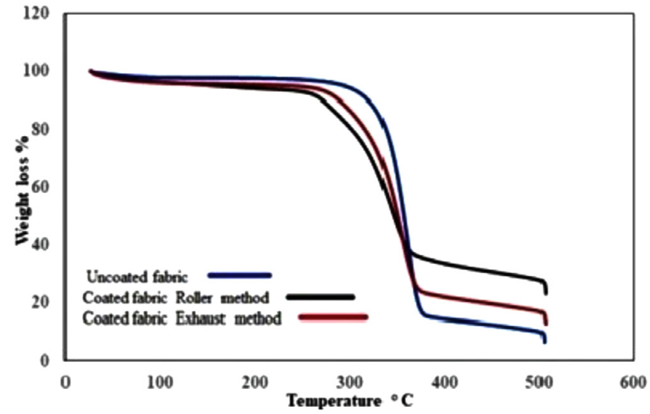

Fig. 7. TGA – Graphs of samples.

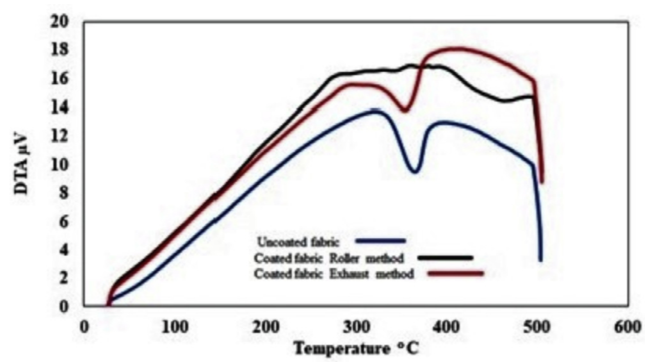

Fig. 8. Differential thermal analysis plots (DTA).

changes at molecular level and breaking down of cellulose molecules present in cotton materials and finally decomposing by evolving heat and turning into ashes. The transitions happening at this stage may be accounted to changes in the materials molecular structure.

3.3. FTIR test results and interpretations

The peaks in the FTIR spectra of the uncoated fabrics (Table 4 and Fig. 9) are at 3335.179 cm^{-1} associated with -C–H- stretching, at 2897.378 cm^{-1} with $–CH_2–$ symmetric stretching, C–H-bending at 1313.762 cm^{-1}, C-O stretching at 1025.377 cm^{-1}. However, the spectra assignment for coated fabrics (Fig. 9) is more confined to OH group stretching at 3580 cm^{-1}, CH bending at 1458.61 cm^{-1} , and C-O- stretching at 1157.395 cm^{-1} respectively. The fingerprint shows traces of cellulose molecules and functional groups present in ethylene and acrylic acid. The presence of aromatic groups in the samples is ruled out.

3.4. Changes in mechanical properties of coated fabrics

From Table 5 and Fig. 10, coated fabrics using ethylene acrylic acid show a clear increase in the flexural rigidity values. This increase indicates that as the deposition of ethylene acrylic acid compound increases, the fabrics become more rigid and hence are stiffer. The flexural rigidity of roller coated fabric showed higher values as compared to uncoated and coated fabrics. Due to the increased absorbency of ethylene acrylic acid during roller coating process, the weight (GSM) of coated fabrics was reported at higher scale as compared to uncoated and exhaust coated fabrics.

Table 4
FTIR spectra data of uncoated and coated fabrics.

Uncoated sample		Roller method		Exhaust method	
Wavenumber cm^{-1}	Transmittance %	Wavenumber cm^{-1}	Transmittance %	Wavenumber cm^{-1}	Transmittance %
4000	94	4000	95	4000	94
3335.179	78	3583.43	82.5	3400	77
2897.378	85	1458.611	85	1425.597	85
1425.827	85	1364.255	82.5	1364.176	82.5
1363.829	82.5	1313.394	80	1313.96	80
1313.762	80	1157.395	77.5	1157.884	75
1159.013	78	1101.745	70	1102.727	70
1101.88	70	1052.704	62.5	1052.172	58
1052.426	58	1026.339	60	1025.602	54

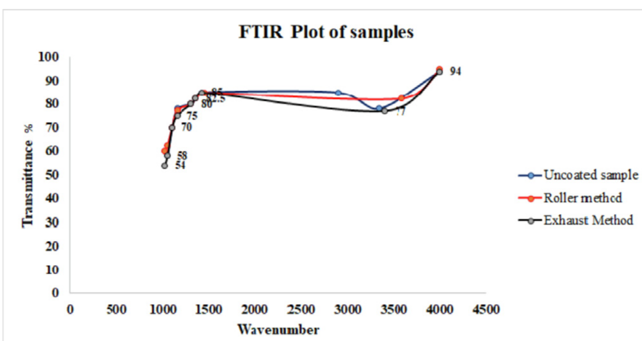

Fig. 9. FTIR plot of samples used in this study.

Table 5
Flexural rigidity values of test samples.

Test Sample	Flexural rigidity 'G' mg/cm
Uncoated fabric	650.73
Exhaust method coating	730.81
Roller method coating	1174.37

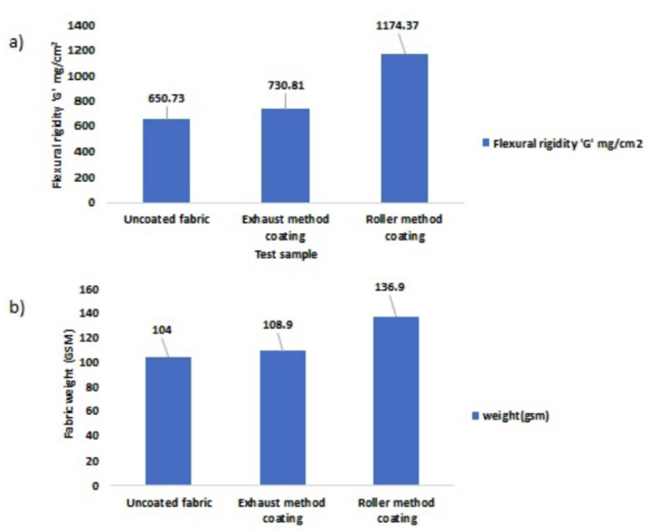

Fig. 10. a) Comparative change in Flexural rigidity b) Comparative change in GSM.

4. Health hazards of ethylene acrylic acid co-polymer

Ethylene Acrylic acid is used in many Textile and leather finishing applications. They are also used in fabric coatings and as a chemical intermediate. However, strong levels of this acrylic acid co-polymer is found to be irritant to eyes, mucous membranes and skin. The hazards caused by ethylene acrylic acid to developmental and reproductive organs is not reported by medical experts after carrying out intensive research on humans and mices. The substance is also found to have no carcinogenic effect on humans. Ethylene acrylic acid co-polymer is proven to be safe when used in making cosmetic products and other applications at low concentration levels. Even though the monomers used are toxic and certain levels of formulations are approved by medical experts to ensure safety levels and prevent hazards to human beings. Many of the cosmetic review panels have issued caution notes for companies to carefully use the formulations and avoid any type of irritation caused to human skin. The monomers like acrylic acid when used in high levels is found to be toxic when it is absorbed into the skin. In the present study care was taken to ensure safety in the lab when applied on cotton fabrics. The concentration levels of acrylic acid copolymer used for coating on cotton fabric is 20 gpl only. More studies and research is required to be carried out on the effect of ethylene acrylic acid coated fabric on human skin. This can be taken as a separate project in consultation with medical and pharmacy experts.

5. Conclusion

The application of ethylene acrylic acid on cotton fabrics produces a stiffer feel and increased flexural rigidity values of the fabrics. This also effects the thermal and other mechanical properties of the coated cotton fabrics. The coated fabrics are thermally stable and possess good surface features. The fabrics coated using pad-dry-cure process like the roller method of coating results in a better-finished fabric and increased mechanical and thermal properties.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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