

Development of Enhanced Ultraviolet Resistance Hybrid Cotton Fabric using Functionalized Biocarbon derived from Teff (*Eragrostis tef*) Hay

KIBEBE SAHILE^{1,✉}, S. VENKATESA PRABHU^{1,2,*} and ABEBE WORKU^{3,✉}

¹Department of Chemical Engineering, College of Biological and Chemical Engineering, Addis Ababa Science and Technology University, Addis Ababa 16417, Ethiopia

²Centre of Excellence, Biotechnology and Bioprocess, Addis Ababa Science and Technology University, Addis Ababa 16417, Ethiopia

³Department of Environmental Engineering, College of Biological and Chemical Engineering, Center of Excellence for Sustainable Energy, Addis Ababa Science and Technology University, Addis Ababa, 16417, Ethiopia

*Corresponding author: E-mail: venkatesa.prabhu@aastu.edu.et

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Due to the depletion of ozone atmosphere, there is a progressive increase in impact of ultraviolet (UV) radiation on human skin. The continual exposure of UV radiation results in serious adverse effects, such as photodermatitis, abnormal skin aging, erythema and skin cancer. Present study aimed to develop an improved UV resisting cotton fabric using the biocarbon derived from the abundantly available biomass residue of teff (*Eragrostis tef*) hay. The biocarbon had its surface functionalized, and then it was deposited onto the cotton using polyaniline as a grafting medium. *In situ* polymerization method was adopted for preparing the enhanced UV shielding fabric. The required functionalized biocarbon and polyaniline were optimized for attaining the maximum possible UV protection factor of the cotton. From the results, the UV protecting ability of the developed cotton fabric was found to be improved by 21 times higher as compared to ultraviolet protection factor (UPF = 66.3) than the original cotton (UPF = 3.1). In addition, the developed cotton fabric was observed to be exhibited excellent tensile strength. The morphological and structural studies were investigated using scanning electron microscopy and Fourier transform infrared spectroscopy for pure cotton fabric and hybrid material coated by biocarbon. Experimentation results revealed that the biocarbon obtained from teff hay could be a used for developing the enhanced UV protecting textile material. Hence, the acquired material can perform for better ultraviolet protection.

Keywords: Teff (*Eragrostis tef*) Hay, Biocarbon, Cotton fabric, Ultraviolet resistance.

INTRODUCTION

Cotton fabric has been widely used in the clothing industry. In addition to its different industrial applications, the wearable business is increasingly getting attention within society due to its several desirable properties [1]. However, due to its low resistant to ultraviolet (UV) ray, sensitive to microorganisms and hygroscopic properties curb the cotton fabrics in number of industrial application areas as a functional material [2]. Hence, development of improved UV protective cotton fabric-based material seems to be more essential toward the fabric can be used in extensive areas like outdoor clothing, sports, automotive and food packaging industries so forth. Even though the cotton fabric has more industrial importance, its poor resistance to UV radiation hinders its potential to high end appli-

cation areas [3]. In this context, there are most widely studied techniques, such as impregnating organic dyes, application of hybrid polymers and coating of nanomaterials are observed to be more effective on improving the functional property with respect to UV protection.

As aforementioned, among the three most familiar methods, the use of organic-inorganic hybrid material plays a great role for modifying functional properties of cotton fabrics, in specific to UV shielding purpose [4]. In such a way, in addition to improved UV protection tendency, applications of hybrid materials on the cotton fabrics has a major role to enhance the different functional behaviours of the fabric, such as flame retardancy, microbial resistance and hydrophobicity [5]. With this perspective, researches are still being carried out to improve the UV protection property for the cotton fabrics using different

novel materials. So far, number of carbon derivatives have been employed for developing a hybrid UV blocking materials. In this context, graphene oxide, carbon nanotubes and graphene are subsequently used carbon derivatives for developing UV blocking properties on the cotton fabrics [6]. However, the use of carbon nanotubes and graphene is observed to be difficult in pragmatic approach due to high cost and complications associate during synthesis. In addition, they require harsh reaction condition for developing the hybrid materials [7]. Hence, the use of these materials limits their industrial applications for developing economically eminent UV blocking material with cotton fabrics. Keeping this view, still there is a research room on the thematic area for apparel research to develop UV protection clothing using easier synthesis method with low cost through a green approach [8].

The studies showed that the carbon derived from crop residues (biocarbon) can be coated toward the hybridization of cotton fabric, which results in improved UV-shielding property for cotton fabrics [9]. Woody based plant materials obtained from forests and non-woody plant materials such as sugarcane bagasse, rice husk, recycled fibers, straws, byproducts from agro-industrial wastes and are found to be best for the preparation of biocarbon [10]. Keeping this in view, this study has been framed to focus to use one of the under-utilized Ethiopian agriculture byproducts, teff hay/straw, was selected as precursor for biocarbon production [11]. Teff is known as most common indig-enous crop of Ethiopia and Eretria. After cultivation, seeds were undertaken to post-harvest process and a significant volume of teff hay is found as its agricultural residue [12]. Teff straw, which is the dry stalks part of teff hay and composed

with cellulose (37.1%), lignin (17.85%), hemi-cellulose (28.99%) and extractives (8.55%). Such a considerable amount of cellulosic and hemi-cellulosic contents make Teff straw can be a promising feedstock for biocarbon production [13].

In order to develop the fabrics having functionally enhanced property, one of the well-known techniques called hybridization by coating with the use of polyaniline and polyurethane. A bio-based activated carbon obtained from teff hay will be obtained as graphite having sp^2 hybridized structure that can effectively absorb UV rays. Considering the low cost, easy synthesizing method and stability of aniline, it is being widely used in industries for polymeric hybridization process. As a result of the high conductivity, optical properties and high stability of polyaniline combined with nanoparticles is getting a great attention in functionalization of materials [14].

Hence, in the present work, teff hay derived activated carbon with polyaniline has been used with varying weight percentages *via in situ* polymerization on cotton fabric. Fig. 1 depicts the mechanism of functionalized carbon using polyaniline onto cotton fabrics. So far, teff hay derived biocarbon has not been studied for the developing a UV shielding fabric material [15]. In understanding from this study, it is anticipated that biocarbon prepared using the waste agro-material, teff hay, may results in improved UV-blocking stuff [16]. In addition, the biocarbon-polyaniline coated fabric has been examined for UV-blocking, mechanical and thermal properties.

EXPERIMENTAL

Plain woven cotton bleached fabric (weight/unit area: 105 g/m²; yarn count: 38 × 38 Tex) was purchased from Bahir Dar

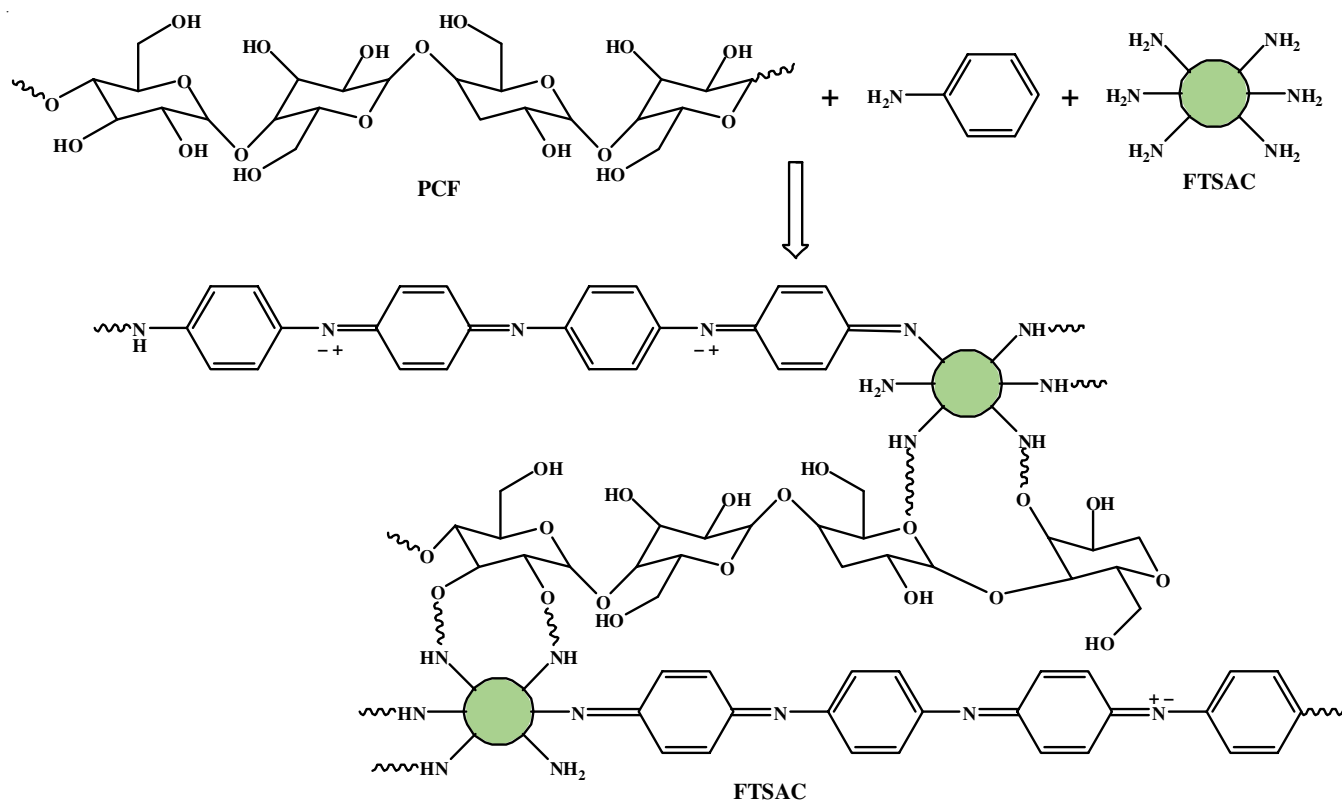


Fig. 1. Coating mechanism of functionalized carbon FTSAC using polyaniline onto cotton fabrics

Textile S.Co. (Ethiopia). The chemicals, aniline, ammonium persulfate, hydrochloric acid, acetic acid and sodium hydroxide were obtained as analytical pure grade from reputed chemicals (Merck), Addis Ababa, Ethiopia. Tef hay was collected from the farmers of teff crop field near Addis Ababa, Ethiopia. Double distilled water was used in all the processes.

Preparation of biocarbon from teff hay: Tef hay was used as precursor for preparing the biocarbon [17]. The biocarbon was prepared according to the method reported in the literature. In such a way, teff hay was washed and purified using distilled water and subjected to dry for moisture removal [18]. The carbonization of the teff hay was undertaken at 500 °C with a nitrogen environment (flow rate: 5 L/min for 1 h). Further, it was digested using NaOH (20%) with weight ratio of 1:4. Subsequently, the carbonized sample was dried at 100 °C for 12 h, followed by the sample was carbonized at 500 °C for 30 min. Aging it was carbonized at 800 °C for 60 min for completing the graphitization process. Further, the carbonized material was subjected to neutralize using HCl (0.1 M) solution followed by washing with clean water until to attain a neutral pH (7.0). The resulted activated biocarbon was allowed to dry at 120 °C in vacuum chamber for 20 h. Further, it was undertaken to surface functionalization [19].

Surface functionalization of teff hay derived activated biocarbon: The carbonized teff hay sample was undertaken to surface functionalization with the amine group for attaining good compatibility with aniline [20]. In this context, the surface of the carbonized activated biocarbon particles was introduced by functional groups, like acid and hydroxyl under elevated temperature (300 °C) for 3 h. Later, the biocarbon was well-disseminated in ethanol solution using a sonication for 30 min [21]. After that, premeditated quantity of 3-aminopropyl-triethoxy silane (3-APTES) was added to the mixture having an adjusted pH of 4.5 using acetic acid [22]. The resulted product was known as surface functionalized biocarbon (FTHBC), which was separated after centrifugation. In order to remove unreacted remaining silane, it was washed 4-5 times using ethanol followed by hexane and vacuum dried for 60 °C for 20 h.

Preparation of FTHBC coated cotton fabric via polyaniline grafting: This study was aimed to prepare cotton fabrics with various combinations of aniline monomer and FTHBC (wt.%) on cotton fabric and to determine its UV-blocking property. The selected combinations of FTHBC and polyaniline are presented in Table-1. In order to prepare the FTHBC coated cotton fabric, predetermined FTHBC and cotton sample were mixed with HCl (1 M) using sonication for 45 min [23]. Then, as per the required precalculated amount, ammonium persulfate (APS) was dissolved in slowly. During the addition of APS, the temperature was controlled to keep below 10 °C for favouring the polymerization reaction of aniline. Using sonication, the mixture was well-dispersed for another 3 h [24]. Then, the hybrid cotton material, coated by FTHBC with polyaniline grafting was obtained. To remove the excess oxidant and unreacted monomers, resulted hybrid cotton material (HCM) was washed with HCl (1 M) solution [25]. Then, the HCM was subjected to wash using clean water and allowed to dry for overnight at 50 °C. The acquired fabrics of HCM was observed

TABLE-1
SELECTED COMBINATIONS OF WEIGHT RATIOS FOR FTHBC AND POLYANILINE

Sample code	FTHBC (wt% based on the fabric)	Aniline (wt% based on the fabric)
Untreated pure fabric (control)	0	0
FC ₀ PA ₃₅	0	35
FC ₁ PA ₃₅	1	35
FC ₂ PA ₃₅	2	35
FC ₃ PA ₃₅	3	35
FC ₄ PA ₃₅	4	35
FC ₃ PA ₀	3	0
FC ₃ PA ₁₀	3	10
FC ₃ PA ₁₅	3	15
FC ₃ PA ₂₀	3	20
FC ₃ PA ₂₅	3	25
FC ₃ PA ₃₅	3	30

as dark green colour due to polyaniline grafting network. Further, the coated samples were coded (Table-1) based on the weight% used for FTHBC and aniline, respectively. To examine the influence of polyaniline alone on UV protecting behaviour, the cotton fabric was coated with 35% (by weight with respect to cotton) polyaniline [26]. For studying the impact of functional property of FTHBC, the cotton was coated with 3 wt.% of FTHBC alone, without polyaniline [27].

Characterization of developed cotton material

UV-visible analysis: Since the objective of the study was to develop a high UV shielding property material, UV-visible spectrophotometer (UV 2450, Shimadzu Model) was used to investigate the UV-Visible transmittance spectra [28]. In this regard, the developed samples of HCM (4.5 cm length, 1 cm breadth) were subjected to investigate for recording the spectrum [29]. The potential of UV protecting tendency for developed HCM samples was evaluated by calculating ultraviolet protection factor (UPF) using eqn. 1 [30]:

$$\text{Ultraviolet protection factor (UPF)} = \frac{\int_{280}^{400} S_{\lambda} E_{\lambda} d_{\lambda}}{\int_{280}^{400} S_{\lambda} T_{\lambda} E_{\lambda} d_{\lambda}} \quad (1)$$

where S_{λ} is known as solar UV spectral irradiance, E_{λ} is known as effectiveness of relative erythral spectral, T_{λ} refers to the transmittance spectral of the given specimen, d_{λ} refers to the increment of wavelength (nm) and λ is the wavelength (nm).

Thermal and SEM analysis: In order to ascertain the thermal stability, TGA was investigated using a thermogravimetric analyzer (Bioevopeak Co., Ltd. TGA-1550, China). In this context, experiments were executed under an air atmosphere using a rate of heating of 10 °C min⁻¹ and the generated thermograms were recorded [31]. To observe the surface morphology, high resolution scanning electron microscope (Thermo Fisher Scientific, Axia ChemiSEM) was used, Horiba equipment attached with this was executed for analyzing the elemental composition of developed samples at 15 kV [32].

Tensile strength, air permeability and stiffness testing: A universal testing machine (HUDA Technology, Model: HUD-B612-S, China) was used test the tensile characteristics of pure and developed UV shielding cotton fabrics. The experiment

was executed with constant rate of extension. The tensile tests were investigated the procedures adopting to ASTM standard D5035-11. In this regard, the length of gauge was set to 75 mm and the speed of the clamp was set as 300 mm/min [33]. Air permeability of the pure and modified hybrid fabrics was investigated using an air permeability testing instrument (GESTER, Model:GT-C27B, Italy) according to the standard BS 3424. In this context, an examination was carried out using fabric contains 5 cm² area [34]. The air flow rate was determined while keeping a constant pressure drop (1 cm head of water). Since the bending length of any textile fabric is a crucial property, it was measured with a stiffness testing instrument (Taber[®] Fabric Stiffness Tester-Model 112) was used. It was carried out with respect to the B.S 3356:1961 standard using a developed specimen [35].

RESULTS AND DISCUSSION

UV protection studies: The results obtained from UV-visible transmittance spectra on pure cotton and FTHBC coated hybrid materials are illustrated in Fig. 2. Using eqn. 1, UPF values were calculated for all the HCM samples and presented in Table-2. As seen Fig. 2, the pure cotton exhibited very poor UV protecting (98% of transmittance) behaviour with UPF 3.7. In present study, towards improvization for UV shielding characteristic of pristine cotton, FTHBC has been coated to develop HCM with the use of polyaniline. Using various weight ratios of polyaniline and FTHBC, different HCMs were developed [36]. In this regard, the hybrid coating cotton materials were prepared based on the gradual increment of FTHBC (0, 1, 2, 3 and 4% by wt. of cotton) by kept concentration of aniline was constant (35% by wt. of cotton). In this approach, while testing the UV shielding property of each HCM, the sample contained FTHBC-3% and polyaniline-35% which was coded as FC₃PA₃₅, exhibited highest UV protection. It had UPF value of 57.9. However, further increment in FTHBC to 4%, with 35% of polyaniline (FC₄PA₃₅), showed a reverse trend, which had the UPF value only 51.4. Such a decreased value of UV shielding behaviour was occurred due to the indecorous distribution and aggregation of FTHBC over the cotton surface [37]. With this approach, it was apparent that the requirement appropriate wt.% for FTHBC to coat on the cotton can be 3%. Consequently, additional optimization has been needed to determine the optimal wt.% with respect to aniline. Hence, wt.% of aniline was varied (10, 15, 20, 20, 25 and 30%) by keeping the concentration of FTHBC as 3 wt.%. In this approach, an HCM coated with 25 wt.% of aniline and 3 wt.% of FTHBC (coded as FC₃PA₂₅) had showed a highest value of UPF (64.1). While comparing these UPF results, the developed HCM, FC₃PA₂₅, had showed 17.32 times higher UV shielding property than that of pure cotton. However, the cotton coated without FTHBC and only with polyaniline (FC₀PA₃₅) had exhibited 4.5 times improved value of UPF [38]. On other hand, the 7.6-time higher value of UPF was found in the HCM (FC₃PA₀) which prepared using FTHBC only. The results cleared that the UV shielding potential has been significantly enhanced for the hybrid material, FC₃PA₂₅ due to the synergistic effect made by the appropriate contribution of polyaniline and FTHBC. In general, activated carbon-

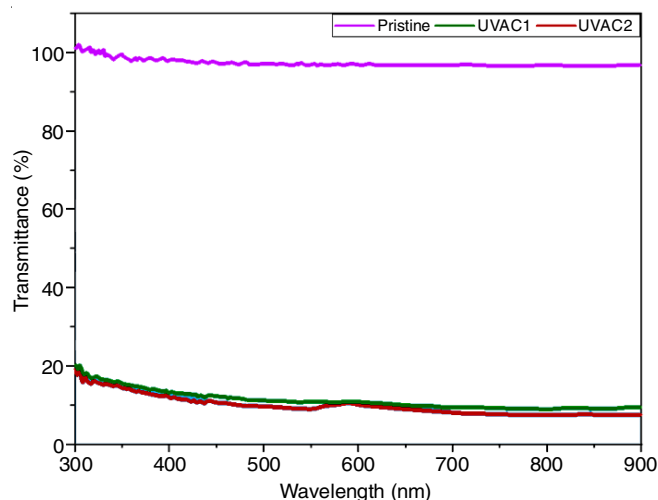


Fig. 2. Spectra observed from UV-visible transmittance for pure cotton, FC₃PA₃₅ and FC₃PA₂₅

TABLE-2
UPF VALUE FOR DEVELOPED HYBRID
COTTON MATERIALS AND PRISTINE COTTON

Sample code	UV-protection factor	Sample code	UV-protection factor
Untreated pure fabric (control)	3.7	FC ₃ PA ₀	8.4
FC ₀ PA ₃₅	14.2	FC ₃ PA ₁₀	19.5
FC ₁ PA ₃₅	17.1	FC ₃ PA ₁₅	37.9
FC ₂ PA ₃₅	39.6	FC ₃ PA ₂₀	51.1
FC ₃ PA ₃₅	57.9	FC ₃ PA ₂₅	64.1
FC ₄ PA ₃₅	51.4	FC ₃ PA ₃₀	59.7

aceous materials exhibit nano form that possesses high surface area. Such ability can effectively absorb the photons that provides an enhanced UV rays protection. It has been established that the energy stored in C=C bonds is about 335 kJ, which appears to be the same as the energy of UV photons. Hence, the huge number of C=C bonds present in the biocarbon probably lead to protect the cellulose structure of the cotton fabrics against UV rays. Furthermore, polyaniline has benzenoid and quinoid rings that also possess very good UV absorption character and thereby increase the behaviour of HCM towards improved UV shielding [39].

Spectral analysis on activated biocarbon and functionalized biocarbon: For achieving the better compatibility, the activated biocarbon surfaces have been subjected to modify using amine terminated silane [40]. After modification for surface functionalization, the biocarbon was examined by FT-IR studies. The results obtained by FTIR investigations on biocarbon and FTHBC have been illustrated in Fig. 3a. From the FT-IR results, the peak occurred at 1580 cm⁻¹ attributed to Si-O-Si due to the silane. The peaks have been observed at 3422 and 3332 cm⁻¹ attributed to the symmetric and the asymmetric stretching due to the -CH₂-aliphatic group present in 3-aminopropyltriethoxy silane. Since amino groups have advantageous interfacial qualities, their presence can facilitate polyaniline development, allowing for more precise regulation of polymer chain adhesion and deposition. During polymerization process, NH₂-groups were converted to NH₂⁺ radicals by addition (APS) oxidant.

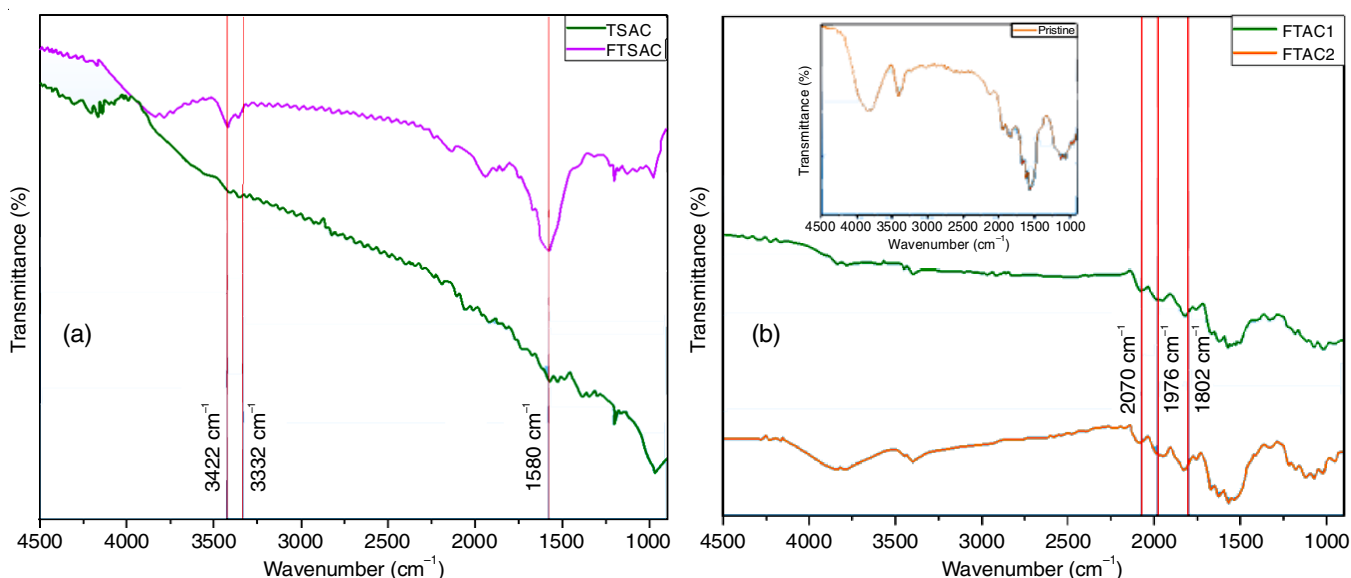


Fig. 3. FT-IR spectra for prepared biocarbon (TSAC) and functionalized biocarbon (FTSAC) (a) and functionalized biocarbon coated polyaniline grafted fabrics (b) and untreated pure cotton (inset picture)

The FTIR spectra for untreated pure cotton (inset picture) and developed the HCM coated by FTHBC have been presented in Fig. 3b. The untreated pure cotton had showed a number of peaks within the wavenumber range 1600 to 1100 cm⁻¹. Such peaks were attributed to C=O and C-O stretching vibrations due to cellulose molecules. A peak occurred at 2963 cm⁻¹, which confirmed the -COOH group. The FTIR spectra for FC₃PA₃₅ and FC₃PA₂₅ are presented in Fig. 3b, where the peaks were observed at 2070 and 1976 cm⁻¹ which confirmed the C=N stretching vibration because of benzenoid and quinoid rings of polyaniline, respectively. Furthermore, the peak observed at 1802 cm⁻¹ was occurred due to the presence of C-N stretching [41].

Morphological studies: Fig. 4 illustrates the morphological observations of the FTHBC sample and prepared hybrid cotton material. In the present study, since a significant UPF was found in the material coded as FC₃PA₂₅, SEM morphological studies were carried out on this material.

The presence of FTHBC particles on the cotton fabrics exhibited a rough surface on the developed hybrid material.

original and smooth surface was found to be absence in the HCM, which indicated the existence interfacial attachment between fabric and FTHBC. It was inferred that the intercalation of biocarbon particles among the weft and warp of the cotton fibers were promoted for UV protecting character. In addition, the SEM observations exhibited a contradict morphology on the cotton surface structures due to the existence of polymeric network attained by hybrids [42]. Hence, such coatings of FTHBC and polyaniline grafting on the cotton surface provide an enhanced functional UV protecting behaviour. Fig. 5 shows the EDX results obtained on pure untreated cotton and prepared hybrid cotton fabric (FC₃PA₂₅). Here, the carbon content found to be increased in the hybrid cotton fabric due to the added polyaniline on the cotton fabric. Also, the EDX results on elemental analysis showed the existence of nitrogen (7.5%), that confirmed polyaniline amendment on the hybrid fabric.

Thermal studies: In order to understand the thermal behaviour of developed cotton fabric coated with FTHBC, thermogravimetric analysis (TGA) has been performed for

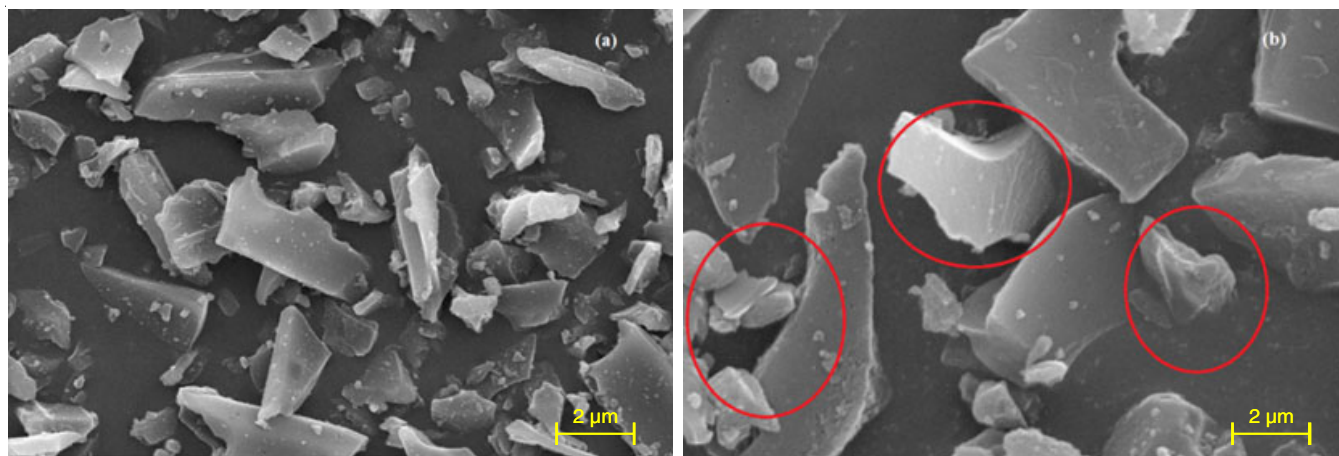


Fig. 4. Morphological inference of the FTHBC samples and hybrid cotton fabric (FC₃PA₂₅)

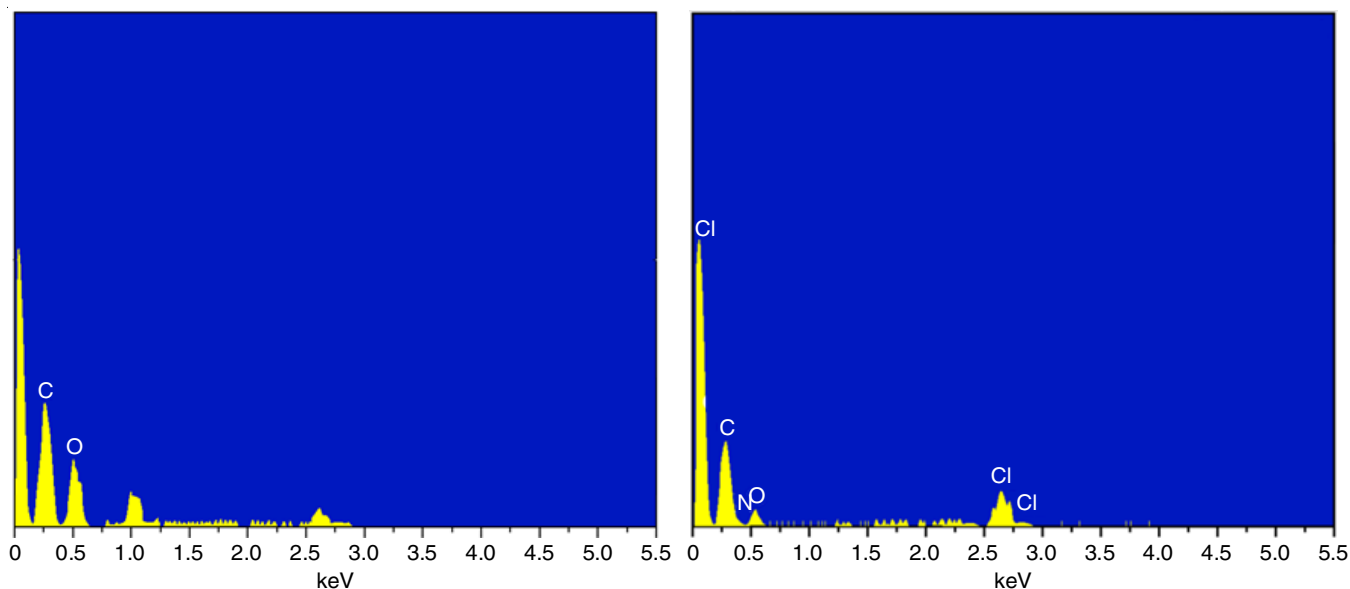


Fig. 5. EDX results obtained on pure untreated cotton and prepared hybrid cotton fabric (FC_3PA_{25})

FC_3PA_{35} and FC_3PA_{25} . Fig. 6 presents the thermal degradation behaviour of those hybrid materials with comparing to the pristine cotton. In general, cotton fabrics has been degraded in four stages [43]. In such a way, till near $100^\circ C$, the materials started to degraded because of the moisture removal process. In second stage, during $300-400^\circ C$, a sharp weight loss was observed because of char formation and volatilization. During the second stage, aliphatic to aromatic char has been formed as residue and notable degradation was also found because of the carbonization, which occurred at $400-600^\circ C$. During this course of degradation, cellulose fabrics were getting to degrade

and combustion gases are releases. The stage associated beyond $600^\circ C$, which is known as fourth stage, where the complete oxidization or decomposition of carbonaceous compounds was found. The same kind of patterns were observed for FC_3PA_{35} and FC_3PA_{25} . However, some improvement in thermal stability was found in hybrid cotton materials which can be a desiring factor with respect to end-use purpose.

Selected basic properties of untreated pure cotton and FC_3PA_{25} : It was apparent that the cotton fabric showed enhanced UV protecting character while grafting and coating with appropriate amount of functionalized biocarbon and polyaniline [44]. However, the basic properties of developed materials should not be significantly altered by such grafting modifications. In this context, selective properties, like tensile strength, elongation and stiffness have been ascertained for FC_3PA_{25} and pure cotton are presented in Table-3.

The obtained results were seeming to be interesting in checking with the properties of elongation at break and tensile strength. In present study, the HCMs have attained an increased value of elongation and tensile strength, which might be the protection given by grafting of FTHBC particles in the woven structure and functionality of silane on the surface of cotton material. The aforementioned reasons can reduce the textile deformation and increase the flexibility which are the desirable properties. However, the bending length for the developed HCM was determined to be lower than pure untreated cotton. Such results were obtained because of the hybrid particles were in nano-form and they have penetrated and deposited interstices of the cotton fabrics. Air permeability test showed that the hybrid fabric materials had decreased value of air permeability, while

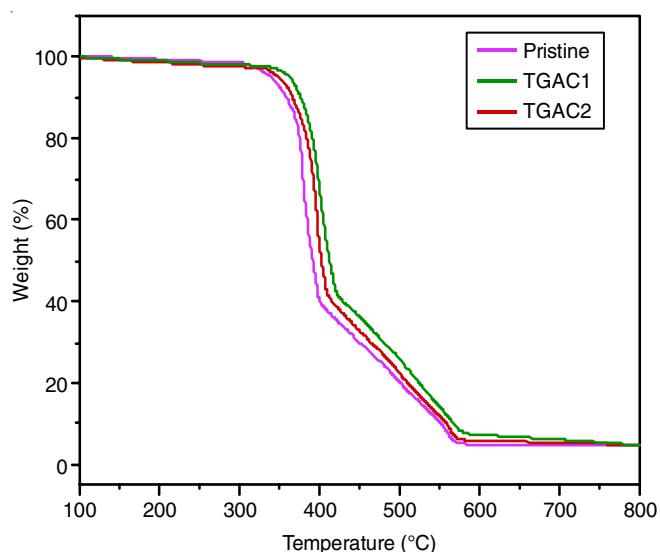


Fig. 6. Thermogravimetric profiles of pristine cotton, FC_3PA_{35} and FC_3PA_{25}

TABLE-3
SELECTIVE PROPERTIES OF THE TREATED AND PRISTINE COTTON SAMPLES

Sample	Tensile strength (lbf)		Elongation at break (%)		Bending length (cm)		Air permeability (cc/s/cm ²)
	Weft	Warp	Weft	Warp	Weft	Warp	
Untreated pure cotton	68.1	74.2	28.6	26.1	1.97	2.06	286.1
FC_3PA_{25}	77.22	78.92	32.17	30.98	1.74	1.95	111.1

comparing to the untreated pure fabric. Such outcome was attained due to the occurrence of inorganic-organic hybrid particles on the surface and interior molecules of the cotton materials.

Conclusion

The present study aimed to develop hybrid fabric cotton material (HCM) with enhanced UV shielding property grafted by low-cost biocarbon prepared from teff hay which is a common agro-waste in Ethiopia. In this regard, biocarbon was prepared followed by functionalized was carried out (FTHBC). The HCM was developed using the *in situ* polymerization method by grafting the cotton fabric with polyaniline and FTHBC. The results showed that the use of appropriate amount of polyaniline and FTHBC could be effectively resulted an HCM with improved UV shielding property. The UPF value was determined for the developed HCM, which indicated that strong UV protecting ability can be provided using and polyaniline and biocarbon to the cotton fabrics. Furthermore, the HCM had been found with enhanced tensile strength, hence, it was well-apparent that the coating with such hybrid particles can ameliorate the UV protection without altering the fabric's basic property adversely. Hence, environmental friendly and low-cost hybrid material with enhanced UV protective property with great advantages of functional behaviour can be more helpful for high-end application textiles such as UV protecting materials.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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