



# Structure and Properties of Thermally Stabilized and Ecologically Friendly Organic Cotton Fibers as a New Activated Carbon Fiber Precursor

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## Abstract

Organic cotton precursor yarn was impregnated in an aqueous solution consisting of diammonium hydrogen phosphate (DAP), boric acid (BA), and urea (U) mixtures before the thermal stabilization stage and then subjected to heat treatments in an air environment at 245 °C. The effect of chemical pretreatment on organic cotton yarn was examined using methods such as X-ray diffraction (XRD), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and infrared (IR) spectroscopy. The XRD analysis revealed a gradual decrease in the crystalline structure, attributed to the disruption of intermolecular hydrogen bonds. DSC and TGA measurements showed an improved thermal stability due to the formation of pre-graphitic structures with aromatic entities at higher temperatures. For samples chemically impregnated and then stabilized, the char yield values increased from 25% to 68% at 500 °C and 23% to 53% at 1000 °C. Analysis of IR spectra indicated a gradual reduction in both intermolecular and intramolecular hydrogen bonds associated with dehydration and dehydrogenation reactions. The IR spectra also confirmed a decrease in crystallinity with increasing oxidation time, which is consistent with the findings from X-ray diffraction. In addition, the IR spectra showed the presence of C=C bonds, indicating the formation of a crosslinked ladder-like structure. The results showed that DAP-BA-U integration increased the thermal stability of organic cotton fibers and the obtained samples were ready for the next stage, carbonization.

**Keywords** Organic cotton · Activated carbon fiber · Thermal stabilization · Diammonium hydrogen phosphate · Boric acid · Urea

## 1 Introduction

Recently, investigations have been conducted to purify polluted water, which poses significant threats to both human health and the environment [1, 2]. Developed countries are seeking methods to purify drinking water resources, such as rivers and lakes, for human consumption due to pollution [3, 4]. The development of advanced fiber-based structural materials, such as activated carbon fibers, has been prompted by escalating environmental pollution. These fibers are known for their favorable pore size distribution, high

surface area, and reactivity [4, 5]. Activated carbon fiber technology is highly favored for its efficiency, capacity, and scalability [3].

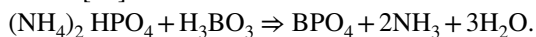
Activated carbon materials, in forms like granules, powder, and multifilament yarns, are preferred for their ease of processing in various industrial and medical applications [6]. The manufacturing of activated carbon fibers involves both chemical and physical activation procedures. Chemical activation, performed at temperatures between 400 and 500 °C, is favored for achieving a high activated carbon ratio compared to physical activation [7]. The physical activation process comprises two stages: Initially, the precursor materials undergo carbonization in an inert medium, followed by activation in a medium containing water vapor, pure oxygen, and carbon dioxide at temperatures ranging from 800 to 1100 °C. Following activation, micro and nano-sized pores form on the surface of activated carbon fibers, crucial for adsorption and desorption processes [8, 9].

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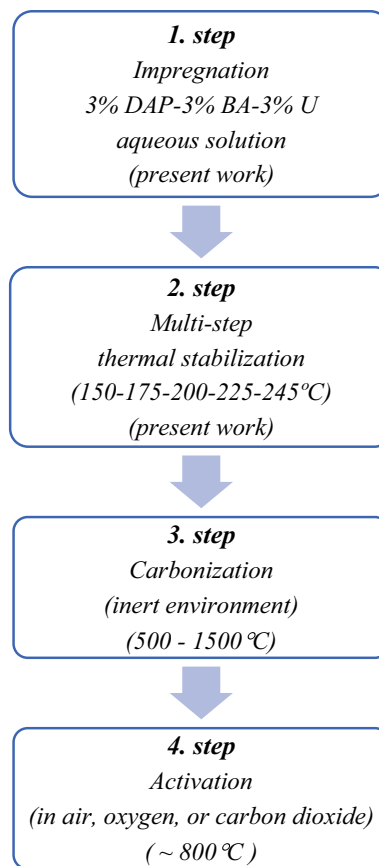
In the literature, many precursor materials have been used to produce carbon fiber and activated carbon [10–16, 31–35]. The industrial manufacturing of activated carbon fibers involves a sequential process encompassing the application of flame retardant agents, oxidation, carbonization, and activation. The oxidation step at temperatures between 200 and 300 °C is crucial for resisting high carbonization and activation temperatures, influencing the final material's physical and mechanical properties. To enhance char yield, pyrolysis of organic cotton is recommended in a reactive atmosphere. Post-carbonization and activation, the formation of micro and nano-sized pores on precursor fibers facilitates adsorption and desorption processes [7]. The environmentally friendly DAP-BA-U combination works as follows: Diammonium hydrogen phosphate (DAP) and boric acid are used as flame retardants, while urea is employed as a swelling agent for cellulosic-based materials. When urea is present, boron phosphate is formed, and it reacts with the hydroxyl groups present in cotton fibers [17]. This chemical reaction is anticipated to hinder the generation of tarry substances, specifically levoglucosan, during stabilization reactions [18].



The systematic exploration of DAP-BA-U's impact on enhancing the oxidative thermal stability of organic cotton yarns has not been addressed in prior research. For this reason, there was a need for a comprehensive investigation to conduct further studies on the potential chemical pretreatment with DAP-BA-U, aiming to improve the oxidative thermal stabilization of organic cotton yarns prior to the carbonization and activation stages. The objective of this work was to examine the effect of DAP-BA-U-impregnation on oxidative thermal stabilization of organic cotton yarns using a multi-step stabilization procedure at temperatures between 150 and 245 °C for various stabilization times.

## 2 Experimental

The organic cotton yarns were chemically impregnated by immersion in a 3% diammonium hydrogen phosphate, 3% boric acid, and 3% urea solution with a pH of 7.3 for at least 24 h at room temperature. Then, a multi-step thermal stabilization process was conducted utilizing five distinct holding times (40, 80, 120, 160, and 200 min) at temperatures spanning from 150 to 245 °C within an oven with air circulation. The heating and cooling rate was determined as 2 °C/min throughout the all process. The general flow chart for the production of cellulose-based activated carbon fiber is presented in Fig. 1. The present work focuses on the impregnation of DAP-BA-U into organic cotton fibers and their thermal stabilization, which are the first two steps in the provided general flow chart.



**Fig. 1** Flow chart illustrating the production process of activated carbon fibers derived from cellulose-based materials

XRD measurements were carried out using a Bruker AXS D8 X-ray diffractometer, with the  $2\theta$  angle ranging from 5 to 40°. The DSC experiments were conducted utilizing the Perkin Elmer Diamond DSC system. The samples, weighing approximately 5 mg on average, were analyzed. The heating rate applied during the DSC analysis was 10 °C/min, and the maximum temperature was set to 450 °C. Thermograms of experimental specimens were obtained using a Perkin Elmer TGA Diamond system. Specimens were put through to heating at a ratio of 10 °C/min until they reached a max temperature of 1000 °C. To ensure an inert atmosphere, a continuous flow of nitrogen gas was implemented at a ratio of 200 ml/min throughout the experimental procedures. From the TGA dataset, char yield (%) and weight loss (%) values were assessed for both untreated and heat-stabilized organic cotton fibers. IR spectroscopy experiments were conducted using the Perkin Elmer FT-IR Spectrum 400 spectrometer. The scan range spanned from 4000 to 450  $\text{cm}^{-1}$ , with a wavenumber resolution of 2  $\text{cm}^{-1}$ .

A peak resolution procedure is employed to assess the precise characteristics of the peak shape, considering the peak heights specified above the baseline for determining

the XRD-conversion index using Eq. (1). In this equation,  $I_0$  represents the intensity of the (002) reflection from untreated cotton yarn, and  $I$  is the intensity of the (002) reflection from heat-stabilized cotton yarn. The apparent crystallinity ( $X_c$ ) of the sample is defined by the relationship between the integrated intensity under the resolved peaks and the integrated intensity of the total area under the experimental trace. Where  $A_a$  is the integral area for the peak of amorphous and  $A_c$  is the integral area for the peak of crystalline. The apparent degree of crystallinity is evaluated for this investigation using Eq. (2). The d-spacing is calculated using Bragg's law in Eq. (3), and the crystal size for a given reflection is computed using the Scherrer formula in Eq. (4), where  $L_{hkl}$  is the average crystal length,  $\beta$  is the half-height width at half-full intensity in radians, and  $K$  is a Scherrer parameter. The DSC-stabilization index is determined using Eq. (5), where  $\Delta H_0$  is the decomposition enthalpy of original organic cotton sample,  $\Delta H$  is the decomposition enthalpy of DAP-BA-U-impregnated and heat-stabilized organic cotton sample, respectively. The equations mentioned above are listed below:

$$\text{XRD - conversion index (\%)} = \frac{I_0 - I}{I_0} \times 100\% \quad (1)$$

$$X_c = \frac{A_c}{A_a + A_c} \times 100\% \quad (2)$$

$$n \cdot \lambda = 2d \sin(\theta) \quad (3)$$

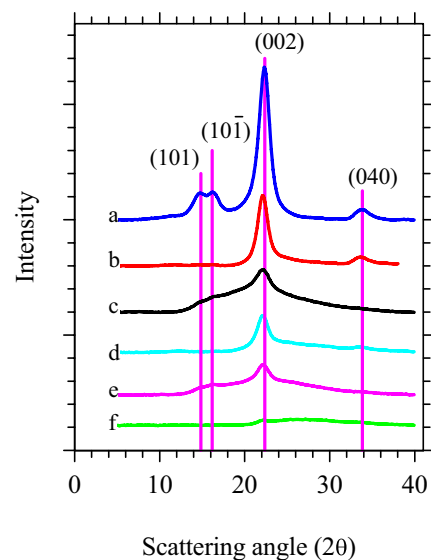
$$L_{hkl} = \frac{K\lambda}{\beta \cos \theta} \quad (4)$$

$$\text{DSC - stabilization index (\%)} = \frac{\Delta H_0 - \Delta H}{\Delta H_0} \times 100\% \quad (5)$$

### 3 Results and Discussion

#### 3.1 X-ray Diffraction

The historical roots of the knowledge about the crystalline structure of cellulose extend to studies on natural cellulose derived from ramie fibers [19]. The cellulose I structure in various plant sources like cotton consists of two chains per unit cell. Later studies [20, 21] have refined the crystal structures of cellulose I. Blackwell et al. [22] modified Meyer and Misch's unit cell [19], doubling a and b-axes and proposing a unit cell with eight chains. XRD reflections of untreated and DAP-BA-U-integrated and stabilized organic cotton yarns were analyzed. This analysis



**Fig. 2** X-ray diffraction traces of untreated and DAP-BA-U impregnated and then stabilized cotton yarns for various stabilization periods (a: untreated; b: 40 min; c: 80 min; d: 120 min; e: 160 min; and f: 200 min)

**Table 1** Analysis of the data obtained after peak fitting of the XRD traces of the samples

Stabilization time (min)	002 crystal size (nm)	Crystallinity fraction (%)	Amorphous fraction (%)	XRD-conversion index (%)
Untreated	6.08	56	44	0
40	5.85	48	52	70.8
80	5.74	27	63	86
120	5.19	19	81	86.5
160	4.54	18	82	90.5
200	3.19	2	98	98.8

revealed the four crystalline peaks corresponding to the cellulose I structure (Fig. 2). Peak fitting procedures were employed to accurately determine parameters such as crystallinity degree, crystal size (002), and XRD-conversion index, and the results are presented in Table 1. The XRD profiles of untreated organic cotton samples exhibited four well-defined crystalline peaks at approximately 14.8, 16.4, 22.4, and 34.8, corresponding to (101), (10 $\bar{1}$ ), (002), and (040) reflections. The intensity of the (002) peak was notably stronger than the other reflections. The impregnation of DAP-BA-U followed by oxidation at 245 °C resulted in a notable alteration of the crystal structure, leading to a progressive reduction in the crystal structure of the organic cotton yarns. The disappearance of specific crystal peaks like (101), (10 $\bar{1}$ ), and (040) due to the influence of increasing oxidation durations suggests the result of decrystallization processes. However, the (002) reflection

persisted throughout the stabilization process, although its intensity decreased significantly.

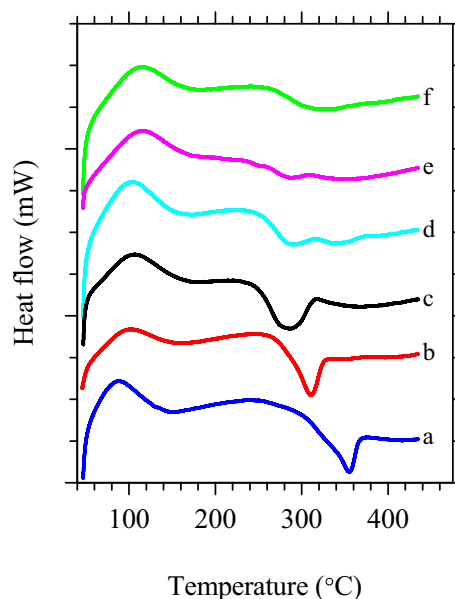
An approach was developed to assess the XRD-conversion index using the intensity of the (002) reflection, revealing growth in the index value up to 98.8% with an increasing oxidation period. The XRD-conversion index is a dependable technique for assessing the amount of cyclized and crosslinked structures formed during the stabilization stage. Therefore, it is believed that there is an improvement in the degree of aromatic structures, indicating the presence of crosslinked ladder-like structural types [10]. The values obtained using Eq. (1) are listed in Table 1. The experimental results indicated that the cotton fibers impregnated with DAP-BA-U and stabilized in an air atmosphere exhibited significant changes in their structural characteristics. Specifically, there was an observed disappearance of the cellulose I structure, accompanied by an increase in the disordered phase.

The reflections such as (101),  $(10^{-1})$ , and (040) disappeared due to ongoing stabilization reactions, while the (002) reflection peak remained slightly prominent. The broadening of the half-height widths of XRD traces was indicative of a gradual reduction in crystal dimensions, attributed to the collapsing structure as part of the continuous amorphization reactions. The progressive disappearance of peaks such as (101),  $(10^{-1})$ , and (040) was linked to the breakdown of the cellulose I structure during the amorphization process. The crystal size of the (002) peak exhibited a decline from 6.08 to 3.19 nm as the stabilization time increased. This decline reflected the ongoing decrystallization reactions.

### 3.2 Differential Scanning Calorimetry (DSC)

Thermograms obtained from DSC were utilized to examine the thermal alterations occurring during the stabilization processes of organic cotton fibers in the presence of DAP-BA-U impregnation, considering various stabilization times. The thermogram of the untreated organic cotton yarn (Fig. 3a) displayed a wide peak around 100 °C, attributed to water vapor. Subsequently, a stronger and wider peak with a peak temperature of 355 °C was identified, linked to decomposition reactions of cellulose and hemicellulose, leading to the formation of levoglucosan and its decomposition products [23]. As the oxidation period progressed, the intensity of this peak decreased while its width increased. Notably, the thermogram of untreated organic cotton fiber did not show a peak around 375 °C, which usually indicates the oxidation of levoglucosan-derived volatile products [24]. The details extracted from the DSC thermogram analysis are outlined in Table 2.

DAP-BA-U integration on organic cotton samples showed a noticeable effect (from Fig. 3b–f). The decomposition temperature of the organic cotton fiber oxidized for 40 min



**Fig. 3** DSC thermograms of untreated and DAP-BA-U impregnated and then stabilized cotton yarns for various stabilization periods (a: untreated; b: 40 min; c: 80 min; d: 120 min; e: 160 min; and f: 200 min)

was notably 45 °C lower than the original fiber. The 40 min oxidation of the sample at 245 °C resulted in a DSC thermogram shift to 310 °C, indicating depolymerization reactions affecting the remaining cellulose and hemicellulose. This shift is attributed to the catalyzed dehydration reactions facilitated by DAP-BA-U, acting as a flame retardant [17]. This lowered decomposition temperature resulted from chemical reactions involving the hydroxyl groups of cellulose rings, hemicellulose, lignin, and boron phosphate groups. The decomposition temperature of cellulose-hemicellulose-lignin significantly decreased as the oxidation time increased from 40 to 200 min. This shift in decomposition temperature was also associated with a reduction in crystallinity degree [25].

Equation (5) was used to evaluate DSC-stabilization index values based on the analysis of DSC thermograms. The results showed that the ladder-like molecular structure began to form even within the first 40 min of oxidation. After 200 min of oxidation, the DSC-stabilization index reached 94% (Table 2). While the untreated sample showed an exothermic peak at 355 °C, the thermogram of the organic cotton sample impregnated with DAP-BA-U and stabilized at 245 °C for 200 min showed an exothermic peak at 283 °C. This exotherm persisted between oxidation times of 40 and 200 min, associated with crosslinking reactions during decomposition and dehydration reactions. The  $\Delta H$  values of the peak gradually decreased over the stabilization times, which ranged from 40 to 200 min (Table 2), suggesting the almost complete conversion of the

**Table 2** Analysis of the data obtained from the DSC

Stabilization time (min)	Exotherm onset (°C)	Decomposition temperature (°C)	Exothermic heat ( $\Delta H$ ) (J/g)	Exothermic peak area (mJ)	DSC stabilization index (%)
Untreated	285	355	11.5	110	0
40	265	310	16.5	93	15.5
80	260	285	16.7	83	24.5
120	260	285	5.5	40	64.0
160	240	285	2.0	13	88.0
200	230	283	1.2	7	94.0

cellulose-hemicellulose-lignin structure into highly aromatized and crosslinked groups of chains.

### 3.3 Thermogravimetric Analysis (TGA)

TGA examines how materials react to changes in temperature, assessing their thermal stability and decomposition characteristics. In this work, the TGA technique was applied to characterize the oxidative thermal stability of original and DAP-BA-U pretreated organic cotton fibers stabilized in an air atmosphere. This method involves subjecting samples to controlled temperature increments during testing while continuously monitoring weight changes. Generally, higher decomposition temperatures indicate greater thermal stability [26]. The literature has demonstrated an increase in the decomposition peak position for soaked flax fibers as the heating rate rises from 5 to 150 °C, emphasizing the significance of heating rates in distinguishing results [27]. Liodakis et al. [28] outlined the temperature transitions identified in a standard TGA scan of lignocellulosic fibers, specifying hemicellulose within 200–280 °C, cellulose within 320–370 °C, and lignin within 370–550 °C.

TGA measurements on organic cotton fiber showed that 10% weight loss occurs at 300 °C and 20% at 320 °C. The TGA scan of untreated cotton fibers revealed rapid weight loss from 300 to 600 °C, persisting until the final scan temperature of 1000 °C. Accelerated pyrolysis without oxygen is not desired for making cotton-based ACFs because it can lower the char yield [7]. Figure 4A shows the TGA graphs of the original organic cotton and the yarns combined with DAP-BA-U and then stabilized. The all of graphs indicate weight loss at approximately 100 °C, which is caused by the removal of moisture from the structure. Beyond this point, original organic cotton exhibited negligible weight reduction up to 250 °C, succeeded by a swift decrease in weight between 250 and 350 °C, followed by gradual losses up to 1000 °C. In contrast, DAP-BA-U-integrated and stabilized organic cotton fibers demonstrated significant changes in thermal behavior, with lower weight loss and increased char yields with longer oxidation times. The untreated yarns experienced weight loss within a limited temperature range,

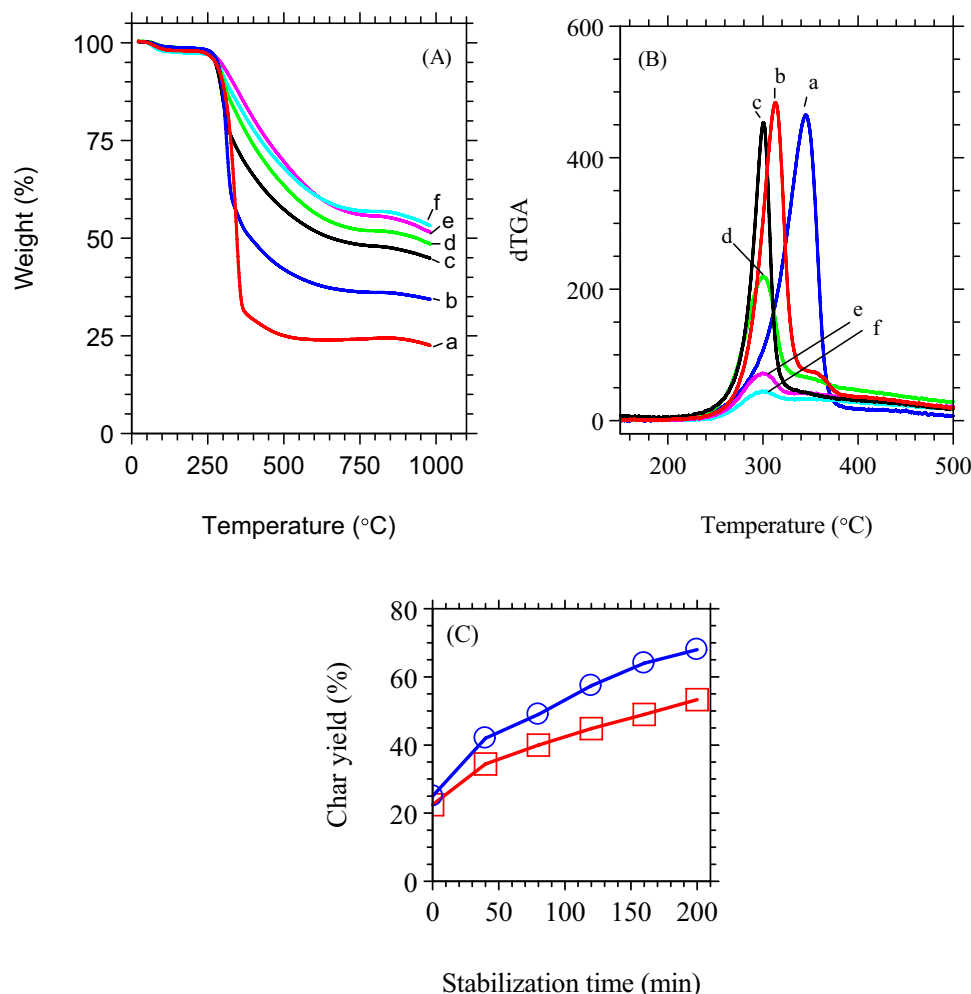
whereas the oxidized samples displayed weight reduction across a wider temperature span, attributed to heightened crosslinking and aromatization processes, resulting in the development of a ladder-like structure.

Figure 4 demonstrated a gradual reduction in decomposition enthalpy and an expansion of the half-height width of the peaks with increased stabilization time. This observation suggested a continuous reduction of decomposition enthalpy due to aromatization reactions as the stabilization time increased. Figure 4B, presenting dTGA thermograms, further confirmed the gradual decrease in peak intensity for samples stabilized at different durations (from 40 to 200 min). Figure 4C depicts the char yield values for original and DAP-BA-U-pretreated oxidized organic cotton fibers at two different temperatures. Results showed a continuous increase in char yield percentage with an extended stabilization period, consistent with previous studies [12, 29, 32]. The char yield values at 500 °C varied between 25 and 68%, and the highest char yield at 1000 °C was 53% for an oxidation time of 200 min. TGA analysis revealed that DAP-BA-U-impregnated and stabilized organic cotton fibers exhibited higher thermal stability than untreated fibers due to oxidation-based crosslinking reactions. The improved thermal stability observed was attributed to the more effective crosslinking and aromatization reactions catalyzed by DAP-BA-U. The results showed that DAP-BA-U strongly interacted with the hydroxyl groups in organic cotton fibers, resulting in both intra- and intermolecular crosslinking.

### 3.4 Infrared (IR) Spectroscopy

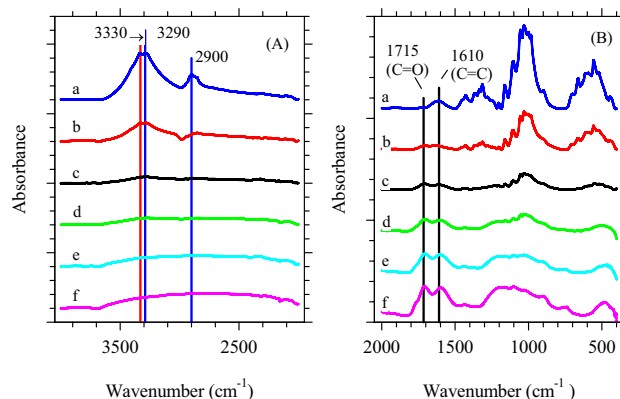
Infrared (IR) spectroscopy is a powerful method used in the analysis and characterization of the structure of polymers, based on the principle that molecules absorb certain infrared radiation frequencies, creating characteristic vibrations. IR spectroscopy was used in the present work to monitor and analyze the changes in structure occurring during heat treatment of organic cotton yarns pretreated with DAP-BA-U solution. It played a crucial role in investigating structural changes throughout the stabilization reactions, providing valuable insights into the molecular structure of cotton

**Fig. 4** **A** TGA and **B** dTGA thermograms of the untreated and DAP-BA-U impregnated and then stabilized organic cotton yarns for various stabilization periods (a: untreated; b: 40 min; c: 80 min; d: 120 min; e: 160 min; and f: 200 min) **C** Char-yield values for two different temperatures (O) 500 °C and (□) 1000 °C



fibers, particularly regarding hydrogen bonding networks among cellulose, hemicellulose, and lignin. The IR spectra of original, and DAP-BA-U-pretreated and then stabilized cotton yarns in the 4000–2000  $\text{cm}^{-1}$  range are depicted in Fig. 5A. The 4000–3000  $\text{cm}^{-1}$  range shows free OH groups (hydrogen-bonded) in cellulose I, while the CH stretch bands are in the 3000–2800  $\text{cm}^{-1}$  range. The hydroxyl groups of cellulose, hemicellulose, and lignin serve as reaction locations with DAP-BA-U functional groups, known for their use as flame retardants. During stabilization reactions, the interaction of DAP-BA with the known hydroxyl groups of cotton occurs in the presence of urea [17]. The components in the cost-effective and environmentally friendly DAP-BA-U solution participate in stabilization reactions and prevent the generation of tarry materials during heat treatments [18].

The intensity of the spectrum of the original cotton yarn weakened with increased oxidation time, indicating deterioration in both intermolecular and intramolecular hydrogen bonding. Specific bands attributed to intramolecular hydrogen-bonded hydroxyl bands experienced a reduction in intensity but did not disappear completely, suggesting



**Fig. 5** IR spectra between 4000 and 2000  $\text{cm}^{-1}$  (**A**), and 2000–450  $\text{cm}^{-1}$  (**B**) of the untreated and DAP-BA-U impregnated and then stabilized cotton yarns for various stabilization periods (a: untreated; b: 40 min; c: 80 min; d: 120 min; e: 160 min; and f: 200 min)

reactions mainly happened between hydroxyl bands of cellulose, hemicellulose, and lignin with DAP-BA-U (Fig. 5A). The gradual decline in crystallinity and the creation of an

amorphous phase with prolonged oxidation time are attributed to the disruption of hydrogen bonding networks induced by oxidation. The CH and CH<sub>2</sub> vibrations (2922, 2900, and

2860 cm<sup>-1</sup>) also exhibited reduced intensities, signifying that not all hydrogens participated in dehydration and dehydrogenation reactions (Fig. 5A).

IR spectra in the 2000–450 cm<sup>-1</sup> region of all samples are separately shown in Fig. 5B. The emergence and reinforcement of the carbon–carbon double bond vibration band at approximately 1610 cm<sup>-1</sup> in samples subjected to oxidation suggest the development of an aromatic structure during the thermal treatment (Fig. 5B). A quantitative analysis of the intensities of IR bands in the 3700–3000 cm<sup>-1</sup> region was performed using a peak-fitting procedure (Fig. 6), yielding a hydrogen-bonding index (HBI) based on intensity ratios. The analysis indicated a progressive decrease in intermolecular hydrogen bonding among hydroxyl bands in cotton yarns, denoted by HBI, with increasing heat treatment time, providing additional evidence of the disruption of hydrogen bonding networks (Fig. 7A, B). The carbonyl band, representing C=O stretching at 1730 cm<sup>-1</sup>, shifted to 1715 cm<sup>-1</sup>, suggesting a robust interaction with hydroxyl groups during the creation of hydrogen bonds. The increasing intensity of the carbonyl vibration with longer heat treatment time suggested the incorporation of oxygen-containing functional species during the oxidation reactions. The oxidation index, calculated as the A<sub>1715</sub>/A<sub>2900</sub> ratio in Fig. 7C, exhibited an

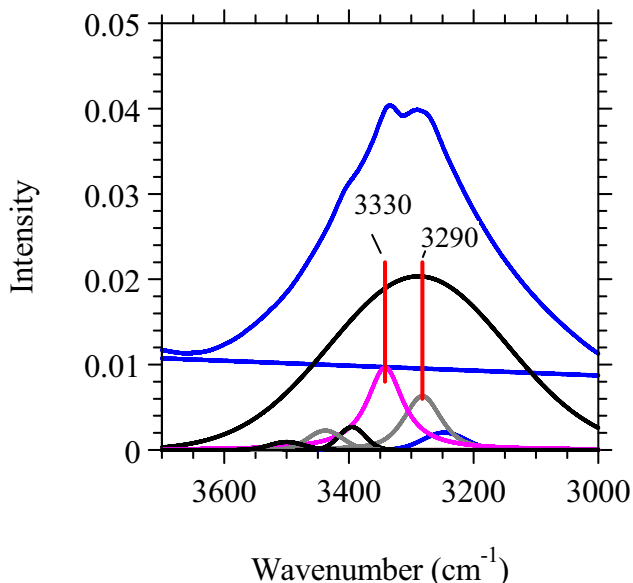
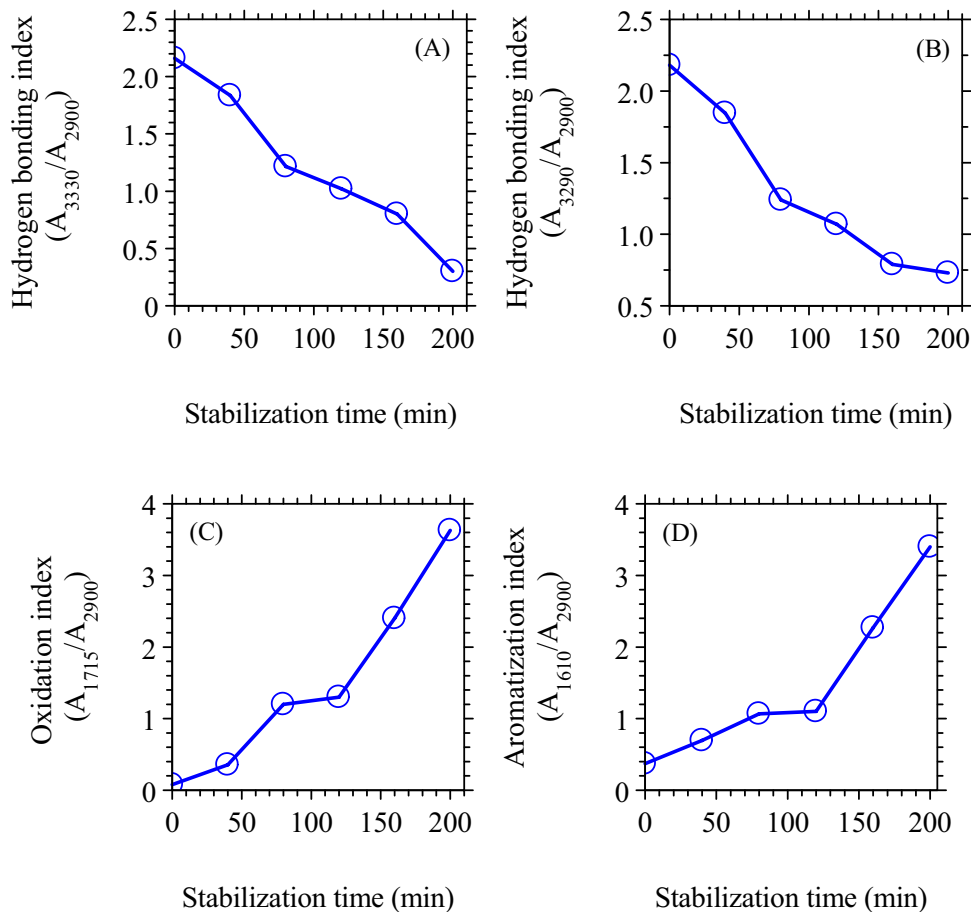
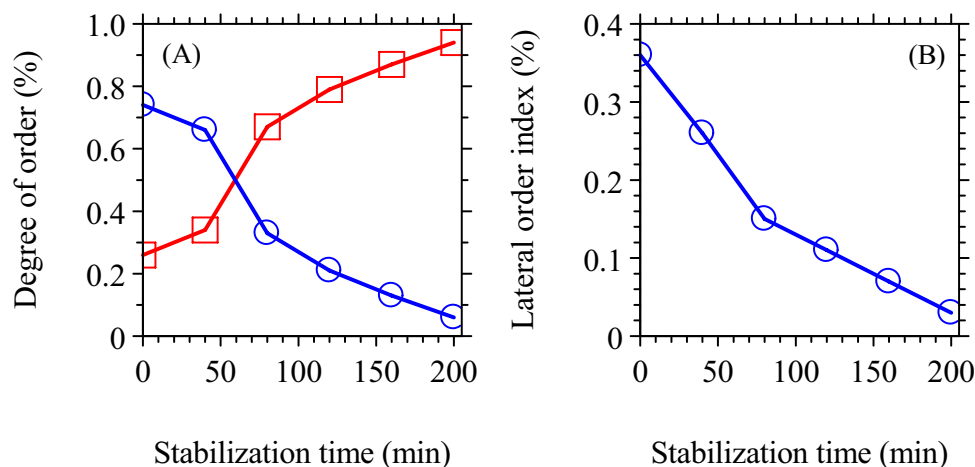


Fig. 6 Peak fitting of the IR spectrum of untreated cotton sample

Fig. 7 Hydrogen bonding indexes A<sub>3330</sub>/A<sub>2900</sub> (A), and A<sub>3290</sub>/A<sub>2900</sub> (B), oxidation index (C), and aromatization index (D) of the untreated and DAP-BA-U impregnated and then stabilized cotton yarns for various stabilization periods



**Fig. 8** **A** Total crystallinity and amorphous fraction values (O) crystalline content, (□) amorphous content. **B** Variation of lateral order index values obtained from untreated and DAP-BA-U impregnated and then stabilized cotton yarns



increase with prolonged heat treatment time, indicating high oxygen content.

The 2000–400  $\text{cm}^{-1}$  region of the IR spectrum of the cotton sample, known for its utility from both qualitative and quantitative perspectives, revealed notable changes. The IR-aromatization index, defined as  $A_{1610}/A_{2900}$ , displayed an increasing trend with prolonged heat treatment time (Fig. 7D). The IR band at 897  $\text{cm}^{-1}$ , attributed to C–O–C, confirmed a decline in intensity of peak as heat treatment time increased. This indicates the substitution of hydrogen atoms within lignin, hemicellulose, and cellulose structures, or the formation of ring structures [30].

The IR band at 1372  $\text{cm}^{-1}$ , related to the  $\text{CH}_2$  bending vibration, was utilized to assess the total crystallinity index (TCI). Results showed decreasing crystallinity and increasing amorphous content with extended oxidation time (Fig. 8A). The lateral order index (LOI), determined by the absorbance ratios of  $A_{1430}/A_{897}$ , demonstrated a continuous decrease for both untreated and oxidized cotton samples, indicating a reduction in lateral order (Fig. 8B). In summary, IR spectroscopy served as a powerful tool to elucidate the complex structural changes occurring during the stabilization reactions of cotton fibers treated with DAP-BA-U solution. The detailed analysis provided insights into the disruption of hydrogen bonding networks, aromatization, oxidation, and changes in crystallinity, enhancing our understanding of the evolving molecular structure during the oxidation process.

## 4 Conclusions

The work examined how DAP-BA-U affects the structure of thermally stabilized organic cotton yarns in an air atmosphere at temperatures of 150 °C to 245 °C and stabilization times of 40 to 200 min. Analyses involving various techniques, such as XRD, DSC, TGA, and IR

spectroscopy, highlighted the positive effects of incorporating DAP-BA-U into organic cotton fibers in terms of thermal stability. XRD analysis showed a gradual decline in crystallinity, attributed to amorphization processes during stabilization reactions disrupting the inter-chain hydrogen bondings. Organic cotton yarns reached thermal stability with 200 min of stabilization at 245 °C. TGA results indicated a char yield ranging from 23 to 53% at 1000 °C after heat treatments. IR spectroscopy analysis revealed a gradual decrease in the intensity of CH groups and hydroxyl groups associated with hydrogen bonds. IR spectra detected the appearance of carbon–carbon double bonds, indicating the development of a crosslinked ladder-like structure. Examination of infrared spectra in the region of 2000 to 450  $\text{cm}^{-1}$  confirmed that amorphization in the structure of organic cotton yarns increased with increasing heat treatment times, consistent with the XRD findings. The organic cotton yarns stabilized at 245 °C, as evidenced by DSC and TGA measurements, displayed noteworthy thermal stability, rendering it suitable for subsequent carbonization and activation processes. The carbonization and activation of organic cotton yarn samples are not covered in this work and are expected to be performed later.

**Author Contributions** Hiba Hariri: Conceptualization, investigation, data acquisition; Kemal Şahin Tunçel: Methodology, data analysis, review and editing; İsmail Karacan: Supervision, writing — original draft, editing.

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**Data Availability** The authors declare that the data supporting the findings of this work are available within the paper. If any raw data files are needed in another format, they are available from the corresponding author upon reasonable request.



## Declarations

**Conflict of Interest** The authors declare no conflicts of interest with the research presented in this article. No ethical approval was necessary for this manuscript as it does not involve human or animal subjects.

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