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PREPARATION AND CHARACTERIZATION OF OXIDIZED VISCOSE RAYON AS ACTIVATED CARBON FIBER PRECURSOR

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ABSTRACT

Viscose rayon fibers prepared with phosphoric acid (H_3PO_4) were oxidized for 30 minutes in air at 150 to 250 °C for 30 min. Optical microscopy, density, elemental analysis, (DSC) calorimetry by differential scanning, (TGA) analysis of thermogravimetric, X-ray diffraction and IR spectroscopy measurements were used to follow and monitor the structural evolution during the oxidation of the phosphorylated viscose rayon fiber.

The physical and chemical properties of phosphorylated and oxidized viscose-rayon fibers changed as the temperature increased. The dehydration of cellulose caused an increase in linear density loss and fiber thickness with temperature. Carbon concentration increased, but hydrogen, oxygen, and phosphorous level decreased, according to elemental analysis. The findings demonstrate that dephosphorylation occurs as temperature rises.

As the results of the DSC and TGA measurements represent how the thermal stability was progressing. The TGA data further demonstrated that a char yield of 49.6% is achievable at 1000°C. The outcomes of X-ray diffraction examination demonstrated the removal of crystallinity brought by de-crystallization processes

An increasing number of ladder-like structures with aromatic moieties were also discovered by X-ray diffraction investigation. The study of (IR) spectroscopy data revealed a progressive and constant decline in the intensity of CH and OH vibration, which are strongly related to the dehydration process. As the results of X-ray diffraction were confirmed by IR spectrograph, which demonstrated the finishing of crystalline structure in the 2000-800 cm^{-1} region. In the 1590–1600 cm^{-1} range, (IR) spectroscopy also showed double bonds of C=C, which was responsible for the emergence of structures like a crosslinked ladder. The outcomes demonstrated that phosphorylation followed by oxidation in air resulted in the enhancement of the oxidative stability in viscose-rayon fibers following activation and carbonization.

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INTRODUCTION

Since its inception in the 1960s, (ACF) activated carbon fiber in knitted form, nonwoven, and woven fabrics have been thoroughly studied and produced commercially [1].

ACFs are widely used for environmental decontamination and purification purposes such as water and air purification, deodorizing, dechlorination, detoxification, fracking, and other industrial and medical applications due to their porous structure, excellent adsorption, and desorption performances, significant specific surface area, and ease of handling [2]. Viscose rayon is one of the precursors used to make ACF [3], polyvinyl alcohol [4], poly p-phenylene-terephthalamide (Kevlar®) [5], poly m-phenylene-terephthalamide (Nomex®) [6], phenolic resin [7] and polyacrylonitrile [8] as viable substitutes for activated carbons that have been based on granular or powder because of the higher adsorption rate and desorption properties, as well as better performance in terms of easier operation [9].

The procedure for producing viscose rayon-based ACF includes (i) chemical pretreatment, (ii) oxidation in air (at or around 300°C), (iii) carbonization at or about 900°C under an inert atmosphere (often nitrogen) and (iv) activation. [3]. The finished carbon or ACFs' microstructure, as well as their physical and mechanical properties, are directly influenced by oxidation. To prepare the precursor fibers for high carbonization and activation temperatures, the oxidative stabilization step is required, which is typically carried out at temperatures between 200 and 300 °C in an air-saturated environment [7,10]. Viscose rayon is typically subjected to low-temperature pyrolysis and when there is a reactive environment, like oxygen, to accelerate the rate of processing and carbonization yield [14]. The carbonized structure exhibits greater reactivity upon activation because the oxidation process produces functional groups containing oxygen [11].

Various chemical pretreatments with Strong acid nitrogenous salts, acidic salts, metallic halides, and phosphoric acid and its derivatives were reported to be used to impregnate viscose rayon fibers before oxidation [15]. These chemicals, being flame retardants, were designed to alter the course of the carbonization stage by acting as a catalyst in the removal of hydroxyl groups [13]. Flame retardants were also found to accelerate the carbonization process, spread the reactions over a wider temperature range, and improve the carbon yield [16]. Flame retardants change how cellulose breaks down by producing more char while producing fewer flammable gases and tars. [17]. Increasing the production of char also serves as a thermal barrier, preventing heat from transferring to the sample's interior and protecting the entire structure from heat. By using phosphate-based flame-retardants, after the

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carbonization, oxidative stabilization & activation phases, a broad & controlled microscopic spectrum of porosity [2 nm pore width] & mesopores (2-50 nm pw) can be formed [18].

Following low-temperature oxidation, oxidized viscose rayon can be subjected to high-temperature carbonization under an inert atmosphere, usually, nitrogen or argon, using high heating rates and temperatures between 1000 and 1500 °C leading to the elimination of non-carbon materials through gaseous volatilization [19,20]. During carbonation, about 50% of the carbon-free components are excreted as carbon dioxide, monoxide, water, hydrogen & methane [20]. Activation can be performed by using physical and chemical methods of activation. Activation aims to create a porous structure on the surface of ACF for adsorption and desorption purposes [27]. An oxidizing environment such as steam, O_2, or CO2 (air) at temperatures 800-1000 °C is usually used to activate material shortly after carbonization.

When compared to physical activation, chemical activation uses activating agents like phosphoric-acid, sulfuric-acid, or nitric-acid, a lower activation temperature (often between 400 and 500 °C), no air, and a larger carbon yield [21].

To carbonize and activate the carbon at the same time, the precursor is first treated with chemical reagents before being carbonized in an inert atmosphere. This is how chemical activation is often done. Higher yields and more micropores with shorter activation times are the key benefits of chemical activation. [20,22]. Physical activation, on the other hand, is used in commercially produced ACF due to its cheaper price [20,23]. Sometimes, it is possible to combine the activation processes of chemicals and physical agents. The precursor in this instance is first impregnated with the chemical activating agent before being carbonized activated at the temperature between 800 and 900 °C with a physical activating agent such as air, oxygen, carbon dioxide, or steam [35].

Numerous micro- and nano-sized pores are created on the surface of the fibers after carbonization and activation operations; they play crucial roles during the adsorption and desorption processes. [1,24]. As far as the physical activation methods are concerned, steam is often used in industry as an activating agent [24]. The irregularly formed aromatic structures split to cause the formation of pores during carbonization. Unfortunately, these pores tend to have poor adsorption characteristics due to the blockages or closures caused by the precipitates or substances produced during the disintegration of the carbon materials [25].

During the physical activation step, an oxidizing agent such as steam, air, or oxygen reacts preferentially with atoms of carbon in the amorphous regions of the carbon structures or on the edges of the graphite planes, resulting in the formation of numerous nano-pores, crevices and cracks leading to more surface area of pores per unit weight on the carbon fiber surface [24,25]. Large specific surface areas are produced by the introduction of microporous structures and the widening of pore diameters into the carbonized fiber during the activation stage. [20,22,26].

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The development of functional groups on the carbon surface during the activation phase produces adsorption selectivity for polar organic and inorganic adsorbates [20,27]. Micropores are those formed during the activation stage that has an effective width of less than 2 nm; mesopores are those that have an effective width in a range of 2 and 50 nm; macropores are those that have an effective width greater than 50 nm. Because pores are formed and created during the activation step, the carbon fibers' surface area is increased[28].

Because it is inexpensive, non-toxic, and environmentally benign, phosphoric acid was chosen as the impregnating agent in the current study. Activation temperature is also said to be lower (often between 400 and 500 °C) than physical activation (over 850 °C)[21]. Recovery and recycling of phosphoric acid are also worth consideration [21]. In earlier investigations, the impact and effectiveness of phosphorylation with phosphoric acid on the oxidative thermal stabilization of viscose rayon fiber were not thoroughly investigated

Phosphorylation of viscose rayon fiber with phosphoric acid was not thoroughly investigated in earlier studies [2]. To improve the Viscose rayon fiber's oxidative stability prior to the carbonization and activation phases, it was determined that further research on the use of phosphoric acid as a potential impregnating agent was necessary. The current investigation's goal was to determine how pretreatment with phosphoric acid affected the oxidation of viscose fibers between 150 and 250 °C. Measurements of fiber thickness, calorimetry by differential scanning, TGA, density, elemental analysis, X-ray diffraction & infrared spectroscopy (IR) were used to complete the structural characterization, to track and observe structural changes as a function of oxidation temperature. [1].

FORMULATION OF OXIDIZED SAMPLES

With a linear density of 32.6 texes in step with sixty-four filaments, the multifilament viscose rayon yarn is untreated. Untreated viscose rayon multifilament yarn was handled with a 5% (v/v) aqueous ethanol solution for half an hour at 50 °C to eliminate impurities from the fiber base. It was then washed thoroughly underneath running tap water for half an hour to remove all capping residues. Samples were given a chemical treatment by being submerged in 3% (w/v) phosphoric acid for 20 minutes at room temperature. At 25 degrees Celsius, the solution's pH was 1.19 [1]. On a dry basis, untreated viscose rayon contained 3.1% (w/w) phosphoric acid. Kimetsan AS provided phosphoric acid (85% conc.) for the experiment (Ankara, Turkey). After the samples were submerged, they were retrieved, the excess solution was wiped off using a towel, and they were then left to dry overnight at 50°C [3]. After that, oxidation was conducted for 30 minutes at isothermal temperatures of 150, 175, 200, 225, and 250°C in a circulating air oven. In the beginning, the samples were coiled around a stainless-steel frame to keep them constrained and stop both physical shrinkage and the loss of molecular orientation. The heating rate used for oxidation was 1°C per minute [2].

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EXPERIMENTAL DATA COLLECTION

The fiber thickness values of the untreated and oxidized viscose fiber had been measured with the use of a polarising microscope (Nikon ME600L, Japan) and a calibrated eyepiece. On average, 5 filaments, each with 20 locations along the fiber axis, were used to quantify the thickness. The linear density of a fiber is the most correct indicator of its fineness. Linear density becomes calculated because of the weight of a multifilament bundle in grams in step with unit length. The importance of a multifilament package in grams in keeping with one thousand meters is known as textual content on this take a look at and used as a linear density measure [2].

In a LECO TruSpec Micro Elemental Analyzer, combustion was used to do elemental analysis for O, H, C, and P. (Leco Corp., USA). High-temperature combustion was utilized to remove components from the substance in oxidized samples. The material was placed in a tin capsule with a weight of 2 mg earlier than being burned. Once the sample was loaded into the chamber, it was kept there until an oxygen dose was discharged. Then it was thrown into the furnace. In the warm, oxygen-rich condition, the sample caught fire and burned. For the oxygen content calibration, benzoic acid was utilized; acetanilide was employed for C & H content calibrations [3]. X-ray diffraction traces (Wide-angle) were taken with the help of a Bruker AXS D8 Advance X-ray diffractometer system have used nickel-filtered CuK* radiation (= 0.154056 nm) at constraints of 40 kV & 40 mA, respectively. Ten steps were taken when counting each degree [9]. The transmission mode measurements of the infrared spectrum were performed using a Perkin Elmer Spectrum 400 FT-IR spectrometer. In every instance, a sample's 50 interferograms were averaged, processed, and apodized using a medium Norton-Beer function. Using the same instrument parameters, every sample spectrum was ratioed as opposed to an equivalent amount of background scans. Finally, OMNIC software was used to evaluate each spectrum [2].

DSC measurements were made utilizing the Perkin Elmer Diamond DSC tool. Around 5 mg was the typical sample weight used. 350°C was chosen as the highest temperature range, with a heating-up rate of 10°C per minute. With indium (m.p. 156.6°C and H = 28.45 J/g), heat flow calibration was performed successfully. Indium and zinc standards (m.p. 419.51°C) were used to calibrate the temperature. The samples were assessed at a constant nitrogen flow rate of 50 mL/min,

Thermogravimetric/Differential Thermal Analyzer by Perkin Elmer was used to collect thermograms for thermographic analysis (TGA). The measurements typically used 4-6 mg of the sample weight. A maximum temperature range of 1150°C and heating up at the rate of 10°C per minute were selected. The melting temperatures of indium (156.6 °C), tin (231.8 °C), zinc (419.51 °C), aluminum (660.1 °C), and gold (1064.18 °C) were employed as reference points for calibration of temperature (TG/DTA). A reference weight of 20 mg at

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room temperature was used for the calibration of the weight balance in all systems. The trials employed a 200 ml/min nitrogen flow. TGA measurements of weight loss and the percentage of char production for raw, phosphoric acid-pretreated, and oxidized viscose rayon fibers were made [4].

EXPERIMENTAL DATA ANALYSIS

(i) X-ray data-curve fitting

Hindeleh et al. [29] developed a curve-fitting approach that turned into utilized to fit all the X-ray diffraction traces generated from oxidized viscose rayon samples a good way to separate the overlapped factors. [3]. Every profile is supposed to consist of a blend of Gaussian and Cauchy functions. The computer software generates a list of precise peak values, along with peak height, peak wide at half height, and peak place, while the observed and computed intensity strains converge to the high-quality to-be-had values. To mirror the equatorial X-ray diffraction traces, a linear baseline, three crystalline (110 and 120), and one amorphous peak was employed. It is believed that the zone below the baseline includes a background signal from the sensor that might possibly contain air and incoherent scattering. The peak height of an amorphous contribution was changed to closely match the tail regions to the experimental datasets. Peak location in the amorphous regions was consistent, although half-height breadth might fluctuate [4].

(ii) Determination of apparent X-ray crystallinity

The ratio of the built-in depth underneath the resolved peaks to the built-in depth of the average scatter below the experimental hint determines the obvious X-ray crystallinity [3]. This definition can be expressed in equation (1) as

$$\chi_c = \frac{\int_0^\infty \mathrm{I}_{cr}(2\theta)d(2\theta)}{\int_0^\infty \mathrm{I}_{tot}(2\theta)d(2\theta)} \tag{1}$$

It is vital to observe that the obvious X-ray crystallinity used to be mounted between two arbitrary angles and has to be viewed as the high-quality viable mathematical answer. This finds out about assessed the obvious X-ray crystallinity to be in the two vary between 5 and 35 [9].

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(iii) Determination of X-ray Conversion Index

The conversion index of X-rays is calculated by equation (2)

$$\text{X - ray - conversion index (\%)} = \frac{\Sigma I_o - \Sigma I}{\Sigma I_o} x100\% \qquad (2)$$

where $\Sigma I_o = I_{1\bar{1}0} + \mathbf{I_{110}} + \mathbf{I_{020}}$ is the sum of the intensities $1\bar{1}0$, 110 and 020 reflections from untreated rayon fiber and $\Sigma I = I_{1\bar{1}0} + \mathbf{I_{110}} + \mathbf{I_{020}}$ is the addition of the intensities of $1\bar{1}0$,110 and 020 reflections from the phosphoric acid pre-treated and oxidized viscose rayon fiber, respectively. After the process of curve fitting, the intensities' values are determined [1].

RESULTS AND DISCUSSION

Viscose rayon fibers that had been prepared with phosphoric acid (H_3PO_4) were heated in the range of 150 and 250 °C and exposed to the air for 30 minutes (Table 1). The samples gradually underwent oxidation in the air, turning from white to brown and reddish brown in the early stages to blackish and black in the late stages (Table 1). It is important to consider the samples' changing hue as clear proof that oxidation and dehydration processes are occurring [2]. The sample developed a light brown hue as the oxidation temperature reached 150 °C, indicating that a chemical process like the dehydration of water had taken place. The samples changed from light brown to darker hues of brown between 150 and 250 °C, and at 225 °C, they finally became black. Burning tests were performed on the samples. Table 1 presents the findings [1]. Samples that had been subjected to phosphoric acid pre-treatment and oxidation did the burning test at 250 °C, indicating that the samples had reached thermal equilibrium and were prepared for carbonization (Table 1) [2].

Table 1 shows the fiber thickness values of the oxidized samples as a function of oxidation temperature [1]. The result for the fiber thickness in the oxidized sample dropped from 28 m to 17.4 m at a temperature of 250 °C. After reaching 150°C for the first oxidation temperature, the fiber thickness value considerably dropped, and the decline persisted as the oxidation temperature rose. The greatest loss in fiber thickness was observed after 250 °C of oxidation, with an approximate 38% loss in fiber thickness occurring in comparison to the untreated sample. The large weight loss that occurred during the oxidation stage is most likely the primary reason for the abrupt fall in fiber thickness [4].

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Table 1 shows the change in linear density in the phosphoric acid-pretreated and oxidized viscose rayon fibers as a function of oxidation temperature. Linear density was shown to decrease with rising oxidation temperature [2]. 44% less linear density was present after the oxidation temperature of 250°C. Linear density suffers as a result of the considerable water loss caused by the dehydration of cellulose during oxidation [3].

Elemental Analysis

Carbon and hydrogen (C and H) contents as well as oxygen (O) content were measured separately. By difference, phosphorus content was obtained [8]. The findings of elemental analysis of viscose rayon fibers that had been oxidized between 150 and 250 C are shown in Figure 1. The findings indicate that carbon content rises as oxidation temperature rises (Figure 1. a) [29]. On the other hand, as the oxidation temperature rises, the concentration of hydrogen, oxygen, and phosphorus drops (Figure 1. b-d). With rising oxidation temperatures, the results clearly demonstrated the impact of phosphoric acid on the removal of oxygen and hydrogen [24]. The decline in oxygen and hydrogen content with rising oxidation temperature is probably caused by the liberation of water [26].

The data fell on a straight line with a gradient of 2.1 when the atomic ratios of H/C and O/C were plotted, as shown in Figure 2, indicating that the elimination of water is the net change in the transformation of the phosphoric acid pretreated and oxidized samples [11]. This chemical reaction is shown as follows:

$$(C_6H_{10}O_5)_n \rightarrow 6nC + 5(H_2O)_n$$

The dehydration of samples seems to depend greatly on the oxidation temperature. The origin of water seems to come from the cellulose structure [16]. In presence of phosphoric acid oxides of carbon are also reported to be released with maximum production at about 200 °C. Hydrogen is also likely to be released as molecular hydrogen as part of methane by production at high heat-treatment temperatures [31]. As will be shown later, apart from the loss of water, the conjugation of double bonds also occurs during the oxidation process. The results confirm the occurrence of dephosphorylation with increasing oxidation temperature.

Analysis of (DSC) Differential Scanning Calorimetry Measurements

DSC was used to analyze the thermal behavior of virgin and oxidized viscose rayon fibers at temperatures between 150 and 250 °C. The DSC thermogram of untreated viscose rayon in

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Figure 3. shows a near 100 °C endotherm associated with moisture evaporation driven by the presence of physically absorbed water [6]. A stronger endotherm that lasts till 340 °C begins at about 280 °C. The depolymerization (decomposition) of cellulose, which results in the formation and evaporation of levoglucosan and its breakdown products, is the main source of this endotherm's peak temperature, which reaches 328 °C. [32]. The dehydration and depolymerization events that always accompany the pyrolysis of untreated cellulose are well-documented in the literature. Levoglucosan is typically produced as a by-product of depolymerization, and it either exhibits an endotherm during the evaporation of breakdown products or an exothermic activity during aromatization [33].

According to minimal char yield, the depolymerization of untreated viscose rayon is thought to occur before pyrolysis [1]. The absence of exothermic peaks in the temperature range between 150 and 250 °C of the DSC thermogram of untreated material also suggests the presence of some exothermic reactions and along with dehydration and depolymerization reactions that result in the formation of vaporization by-products based on proposed and low char yield [28].

Two endothermic peaks are anticipated to occur when phosphoric acid is present, favoring dehydration over depolymerization. Due to the occurrence of primarily favorable dehydration processes, pre-treatment with phosphoric acid is anticipated to boost char output [34]. It is understood that a hydrogen atom is dissociated from and sandwiched between two hydroxyl groups throughout dehydration processes, producing water [35].

The findings of the DSC thermograms of the oxidized viscose rayon fibers revealed that the pre-treatment with phosphoric acid and subsequent oxidation in the air had a significant impact. Figures 3. b to 3. f exhibit the DSC thermograms of viscose rayon fibers that had been treated with phosphoric acid and oxidized in the air between 150 and 250 °C. The phosphorylation of cellulose caused a stronger endotherm that looked to be changed from 328 °C to a lower temperature of roughly 220 °C, according to the DSC thermograms, which first exhibited an endotherm of 100 °C approximately due to the evaporation of water [22]. A decrease in crystallinity may be the cause of this behavior (i.e., increasing amorphous fractions) [36] and also the poor thermally stable property of viscose rayon fiber pre-treated with phosphoric acid followed by oxidation in air. With a rising temperature of oxidation, the area beneath this endothermic peak shrank until it vanished entirely at oxidation temperatures of around 200°C. The decrease and eventual removal of the endotherm illustrate how the pre-treatment with phosphoric acid delays the depolymerization events that take place during the oxidative thermal treatments. This could help to explain why the char yields were higher after the phosphoric acid pre-treatment [19].

Three exothermic peaks were also seen in the DSC thermograms in addition to the two endothermic peaks at about 100 and 328 °C. Due to the dehydration of cellulose, the first exothermic peak was observed to be broad and extend from 120 to 200 °C, with a maximum

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temperature of up to 140 °C. The area associated with dehydration was observed to decrease with rising oxidation temperature and finally vanish entirely at 250 °C [6].

The greatest peak temperature of the third exothermic peak, which ranged from 270 to 400 °C, was determined to be around 305 °C and was connected to the oxidation of levoglucosan breakdown by-products [6]. With rising oxidation temperatures, the spread of this exotherm widened until it vanished entirely at 250 °C [8]. The findings indicate that levoglucosan production is suppressed and finally prevented at oxidation temperatures of 200 °C or higher, the benefit of boosting thermal stability before the carbonization step [6].

It is proposed that cellulose undergoes two different types of processes to undergo heat depolymerization [37]. Lower temperatures cause a slow decomposition reaction that includes dehydration, degradation, oxidation, the evolution of CO and CO2, and the creation of carbonyl and carboxyl groups, leading to the formation of carbonaceous residue [23]. It has been discovered that cellulose decomposes into a tarry and mixes with levoglucosan as its main part at higher temperatures [16]. Levoglucosan continues to break down into compounds that are combustible and volatile [14].

Analysis of TGA data

Thermogravimetric analysis was used in the current experiment to assess the temperature stability of untreated and pretreated viscose rayon fibers oxidized in the air [10]. It is well-informed that the manufacturing of activated carbon fiber or viscose rayon-based carbon fiber in an inert environment demands gradual pyrolysis, or carbonization, which is unfavorable. There is a decent chance that this will increase the side reactions that release volatile carbon-containing chemicals and reduced carbon yield [1].

It is well-recognized that fast pyrolysis causes the formation of several volatiles, like carbon dioxide, organic acids, and aldehydes which ultimately results in low carbon yields. Others maintain that air oxygen is necessary for the production of more carbon [3]. Years of experience have taught us that weight losses connected to relatively steeper and narrower temperature ranges imply faster decomposition reactions, which cause significant weight losses and lower carbon yields [4]. Utilizing TGA and derivative TG curves, the effect of pyrolysis of viscose rayon utilizing phosphoric acid pretreatment and oxidation at various temperatures is described [30]. Using the weight loss and its rate, the relative yields of the carbonization process might well be compared as a function of oxidizing temperature. The highest rates of weight loss and various peaks during the thermal breakdown of the samples under examination can both be determined with the aid of derivative TG curves [22].

The cellulose degradation reactions were altered, as revealed by a TGA study of viscose rayon fibers that had been prepared with phosphoric acid and then oxidized [2]. Viscose rayon that had not been treated displayed one area of fast disintegration [11]. According to Figure 4, the

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dehydration of cellulose at temperatures between 150 and 250 °C caused the second zone of weight loss [18]. Beginning at 250°C and continuing until 400°C, cellulose thermally decomposed (Figure 4). In a nitrogen atmosphere, weight loss increased steadily between 250 and 400 °C, peaking at 353, and peaked at 1000 °C, with a char yield of 12.7%. This suggests that a large quantity of thermal breakdown processes has been placed [2].

Viscose rayon fibers that had been prepared with phosphoric acid and then oxidized showed a widening of the weight loss curves and a shift in the temperature of the maximum weight loss to lower values (Figure 4). Crosslinking and aromatization processes caused a steady weight loss in the temperature 250 to 550 °C, according to TGA thermograms [1]. Dehydration and depolymerization processes are virtually always involved in the pyrolysis of untreated viscose rayon fiber. Levoglucosan is created during depolymerization events, and it can either evaporate and cause endothermic reactions or undergo aromatization and cause exothermic reactions [33].

The TGA thermogram of untreated viscose rayon revealed a low char production of 12.7% at 1000 °C, which indicates that depolymerization reactions are primarily involved in pyrolysis [35]. The findings indicate that phosphoric acid pretreatment followed by oxidation alters the pyrolysis process's course and avoids the development of tar and volatile chemicals, demonstrating the overall advantage of dehydration over depolymerization processes that leads to higher char yields [3]. The ultimate polymeric residue, according to Tang and Bacon, is composed of four-carbon building units that were originally present in the cellulose structure. The highest output is therefore anticipated to be 29.5% [2]. This indicates that a maximum weight decrease of 70.5% is possible [12].

Figure 5.a shows the derivative TG curves of untreated viscose rayon in a nitrogen atmosphere. Viscose rayon showed a sharp and strong peak with a maximum decomposition rate at 353 °C. The derivative TG curve from the sample oxidized at 150 °C (Figure 5. b) was showing a sharp peak with a maximum rate of decomposition at 278 °C and a small broad peak appeared as a shoulder at 240 °C [19]. This peak may be due to the broad distribution of molecular weight from the cellulose structure. In the case of the sample oxidized at 175 °C (Figure 5. c), it showed a sharp peak with a maximum rate of decomposition at 278 °C. In this case, these were no sign of any broadening or shoulder on the low-temperature side of the main peak [2]. The char yields of samples oxidized between 150-175 °C were 33.2 and 35.2%, respectively. On increasing the oxidation temperature to 200 °C (Figure 5.d), the derivative TG curve showed a much sharper and stronger peak with a maximum rate of decomposition at 284 °C and an increased yield of 36.1% up to 1000 °C. The sample oxidize at 225 °C showed a much-reduced peak intensity with a maximum rate of decomposition at 284 °C. This may be due to the increasing crosslink density and a higher yield of char 47.5% up to 1000 °C [27]. Finally, the sample oxidize at 250 °C shows a weakened peak due to almost total loss of cellulose and an increased amount of yield of 49.6% at 1000 °C [23]. Although Tang and Bacon showed that the maximum yield is 29.5%, the higher values of

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char yield obtained in the present investigation may be due to the presence of dehydrated phosphoric acid in the residue [33].

It is clear that the incorporation of phosphoric acid as an effective dehydration agent followed by oxidation resulted in a significant reduction in the maximum rate of cellulose degradation temperature, enhanced the char yield, inhibited the formation of tarry substances, and prepared the samples for high-temperature carbonization and the activation stages [19].

Phosphoric acid offers many advantages: it results in a high carbon yield and is a one-step process (i.e. impregnation, oxidation, and carbonization). Phosphoric acid is found to be a good dehydrating agent and inhibits the formation of levoglucosan [13].

Analysis of X-ray diffraction data

Viscose rayon fiber has a typical cellulose II crystal structure and is essentially regenerated cellulose, often known as cellulose hydrate. It is typically thought to have less crystallinity than cotton [37]. A cellulose II structure is also created when cotton is mercerized with strongly alkaline solutions. After heat treatment in glycerol at temperatures as high as 270°C, especially after heat treatment in nitrogen, water, or steam, a cellulose II structure in some situations may contain a cellulose IV structure [31]. When the cellulose long axis is parallel to the unit cell's c-axis and the unit cell comprises two cellobiose segments of two cellulose molecules, the unit cell of a cellulose II structure is monoclinic [38].

The equatorial X-ray diffraction patterns of the virgin, pretreatment with phosphoric acid, and oxidized viscose rayon fibers are shown in Figure 6 as a function of oxidation temperature [4]. The X-ray diffraction lines in Figure 6 are shifted along the ordinate for better understanding (Y-axis).

Three distinct reflections are seen when the equatorial X-ray diffraction trace of untreated viscose rayon fibers is qualitatively analyzed (Figure 6). A curve-fitting approach was used to acquire precise peak characteristics from the equatorial X-ray diffraction traces displayed in Figure 6. To calculate the apparent X-ray crystalline phase, amorphous percentage, and X-ray conversion index as a percentage of oxidation temperature, curve fitting of the equatorial X-ray diffraction traces was also used [10].

The pattern of equatorial X-ray diffraction untreated viscose rayon fiber may be separated into at least three unique peaks, each with a different index number between 110 and 020 and a different d-spacing between 0.728, 0.441, and 0.405 nm (Table 2) [39]. These peaks may be found in the cellulose II structure's monoclinic phase, which has unit cell 42 dimensions of a=0.810 nm, b=0.903 nm, c=1.031 nm, and =117.1. Table 2 shows the resolved peak properties of untreated viscose rayon fiber [3] and compares the calculated and observed d-spacings. To enhance the fit in the tail regions of the surrounding peaks, a second peak at

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around 19.4 (d-spacing of 0.441 nm) was included during the curve fitting stage [40]. Regarding the apparent crystallinity of the original cellulose II structure, phosphoric acid pre-treatment followed by oxidation in air at temperatures between 150 and 250 °C appeared to have significantly altered the molecular structure [1]. Based on equatorial X-ray diffraction traces, apparent crystallinity gradually decreased as oxidation temperature increased [17]. A normal decrystallization (also known as amorphization) process happened all through the oxidation stage with increasing temperature [29].

An equation to calculate the ratios of the cellulose II and amorphous structures might be developed using the peak areas from the curve fitting of the equatorial X-ray diffraction traces shown in Figure 6 [1]. Figure 8 illustrates an increasing amorphous percentage and a diminishing apparent X-ray crystallinity, as determined by the results of X-ray diffraction studies [33]. The amorphous phase increased from 59 to 100% after being exposed to oxidation at 250°C, while the cellulose II phase decreased from 41 to 0% [23]. The intensities of the 110 and 020 cellulose II reflections were utilized to develop a unique method for determining the X-ray conversion index [2]. According to the oxidation temperature, the measurements of the X-ray transformation values for the oxidized viscose rayon fibers are shown in Figure 9. The progressive rise in oxidation temperature from 150 to 250 °C caused the X-ray conversion score to grow from 34.4 to 100%. It is feasible to accurately anticipate how many ladder structures would be formed during the oxidation process by looking at the data of the X-ray conversion index, which was shown to rise with increasing oxidation temperature [40]. According to the studies done so far, phosphoric acid pre-treatment followed by air oxidation disrupted the crystalline structure of cellulose II and increased the amount of the amorphous phase and the percentage of ladder-like structures with aromatic groups [8].

Analysis of FT-IR spectroscopy measurements

The oxidation of viscose rayon fiber that had been prepared with the phosphoric acid solution was monitored using IR spectroscopy [2]. Figure 10 displays the IR spectra between 4000-2000 cm^{-1} for the untreated, phosphoric acid pre-treated, and oxidized viscose rayon fibers together with distinctive bands [26]. It is believed that cellulose II's free and intramolecular hydrogen-bond OH groups are responsible for the area from 4000 and 3000 cm^{-1}. The area from 3000 to 2800 cm^{-1} includes CH stretch vibrations. It is well known that cellulose's hydroxyl groups operate as the sites of phosphoric acid reactions [28]. The various infrared bands from the infrared spectra of the untreated viscose rayon fiber are listed in Table 3. Particularly the bands associated with OH and CH stretching and the crystalline bands present within the 2000-800 cm^{-1} range in the IR spectra revealed noticeable relative intensity changes following phosphoric acid treatment and oxidation in the air [13].

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The intensity of OH bands gradually decreased as the oxidation temperature rose. This is unmistakable proof that dehydration processes are taking place while the oxidation process is taking place [32]. The OH stretching region becomes more defined in the spectrum for the sample that underwent 150°C oxidation, whereas the location of the CH stretch band shifts 2895-2928 cm^{-1}. It demonstrates that even at a temperature of 150°C, oxidation in air results in molecular changes at the C-H bonds [22].

The CH-stretching area has a similar reduction of intensity as the oxidation temperature rises. This shows that in addition to oxidation processes, dehydrogenation reactions take place concurrently [21]. This implies that in addition to the oxidation reactions, parallel dehydrogenation reactions also take place. When the oxidation temperature approached 250 °C, the CH-stretching bands began to diminish significantly, but they never fully vanished. In contrast, the OH bands continued to be present but never totally evaporated [39].

Figure 11 displays the infrared spectra of untreated, phosphoric acid pre-treated, and oxidized viscose rayon fibers in 2000-800 cm^{-1} area along distinguished bands [2]. Table 3 also includes a list of the distinctive bands in this area. In contrast to the IR bands of untreated rayon fibers, which are known to be sensitive towards crystallinity type and lattice type and are positioned about 1420, 1375, 1337, 1318, 1280, 1228, 1165, and 895 cm^{-1}, the physically absorbed water generates a distinctive band around 1650 cm^{-1} [34].

Additionally, the spectrum exhibits a drop in cellulose II band intensity of about 895 cm-1, which is attributed to the cellulose ring's C-O-C stretching [13]. The carbonyl bands become more intense and emerge as a doublet at 175°C (Figure 11. c), the oxidation temperature. At 1620 cm^{-1}, a new band emerges [15].

This is consistent with the findings of the elemental analysis, which showed that hydrogen content dropped as oxidation temperature rose [33]. The carbonyl band at 1710 cm^{-1} becomes strong and wide at oxidation temperatures between 200-250 C, and a new band connected to the C=C band about 1590–1600 cm^{-1} also arises. When oxidized at 200–250°C, C=C stretching becomes predominant, indicating the presence of aromatic compounds in the oxidized cellulose [31].

A viscose rayon fiber that had been prepared with phosphoric acid and oxidized at 150°C could be seen in the IR spectrum as having bands between 993 and 1064 cm-1 [26]. The IR band at 1064 cm^{-1} in the polyphosphate chain P-O-P was ascribed to P-O symmetrical vibration [17]. The other band at 993 cm^{-1} is blamed for the P-O-C asymmetric stretching [24]. At oxidation temperatures of 175°C and higher, the strength of the bands going to decrease. This finding supports the results of the elemental analysis, which showed that dephosphorylation took place as the oxidation temperature rose and the phosphorus level declined [12].

The outcomes demonstrated that depolymerization and dehydration events were catalyzed by phosphoric acid, which also stimulated these activities. Phosphoric acid is frequently utilized

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in the manufacturing of activated carbon 1 for chemical activation purposes by forming phosphate or polyphosphate linkages between the cellulose chains [20].

CONCLUSIONS

Viscose rayon fibers that had been phosphorylated were subjected to a 30-minute oxidation process in the presence of air between 150 and 250 °C. Measurements of fiber thickness, density, elemental analysis, differential scanning calorimetry, thermogravimetric analysis, X-ray diffraction, and IR spectroscopy were used to investigate in detail the structural evolution that occurs during the oxidation of phosphorylated rayon fibers. As the temperature increased, the rayon fibers underwent both chemical and physical changes. The outcomes of elemental analysis confirmed the dephosphorylation with increasing temperature, as the carbon content increased and the hydrogen, oxygen, and phosphorous content decreased. The DSC and TGA readings' thermal analysis revealed an improvement in thermal stability. Additionally, a 49.6% char yield at 1000°C was demonstrated by TGA analysis. The loss of crystallinity caused by the amorphization processes was demonstrated by X-ray diffraction research. Additionally, X-ray diffraction revealed an expanding amount of ladder-like structures with aromatic moieties. The dehydration events caused the OH and CH vibrations to lose some of their strength, as demonstrated by IR spectroscopy. In line with X-ray diffraction findings, IR spectroscopy in between 2000–800 cm^{-1} area revealed the demise of cellulose structure. The development of C=C double bonds was also detected by IR spectroscopy in the 1590–1600 cm^{-1} range. The outcomes showed that, before carbonization and activation processes, phosphorylation followed by oxidation in the air increased the oxidative stability of viscose rayon fibers.

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REFERENCES

1. F. Zeng, D. Pan, and N. Pan, *J. Inorg. Organomet. Polym. Mater.*, 15, 261 (2005).
2. J.S. Mattson and H.B. Mark, *Activated Carbon*, Marcel Dekker, New York, 1971.

Received 26 Ocak 2023; Received in revised form 15 February 2023; Accepted 22 February 2023;
Available online 20 March 2023;
doi: 10.5281/zenodo.7955648

3. J-M. Huang, I-J. Wang, and C-H. Wang, *J. Polym. Res.*, 8, 201 (2001).

4. S-J. Zhang, H-Q. Yu, and H-M. Feng, *Carbon*, 44, 2059 (2006).

5. S. Villard-Rodil, F. Suarez-Garcia, J.I. Paredes, A. Martinez-Alonso, J.M.D. Tascon, Chem. Mater. 17 (2005) 5893.

6. F. Suarez-Garcia, A. Martinez-Alonso, and J.M.D. Tascon, *Carbon*, 42, 1419 (2004).

7. N. Worasuwannarak, S. Hatori, H. Nakagawa, K. Miura, Carbon 41 (2003) 933.

8. S. Y. You, Y.H. Park, and C.R. Park, *Carbon*, 38, 1453 (2000).

9. C-I. Su, C-C. Peng, C-Y. Lee, Textile Res. J. 81 (2010) 730.

10. M-C Yang, D-G Yu, Textile Res. J. (1996) 115.

11. A.C. Pastor, F. Rodriguez-Reinoso, H. Marsh, M.A. Martinez, Carbon, 37, 1275-1283 (1999).

12. F. Carillo, X. Colom, J.J. Sunol, and J. Saurina, Eur. Polym. J., 40, 2229 (2004).

13. R. Bacon, G.E. Cranch, R.O. Moyer, W.H. Watts, U:S. Patent, 3,305,315 (1967).

14. J. Siroky, R. S. Blackburn, T. Bechtold, J. Taylor and P. White, Cellulose, 17, 103 (2010).

15. D.R. Moore, S.E. Ross, and G.C. Tesoro, U.S. Patent, 3,527,564 (1970).

17. B.K. Kandola, A.R. Horrocks, D. Price, G.V. Coleman, J. Macromol. Sci. Rev. Macromol. Chem. Phys. C36(4) (1996) 721.

18. J.J. Freeman, F.G.R.Gimblett, R.A. Roberts, K.S.W. Sing, Carbon 26 (1988) 7.

19. H. Li, Y. Yang, Y. Wen, L. Liu, Comp. Sci. Tech. 67, 2675 (2007)

20. N. Yousof and A.F. Ismail, J. Anal. Appl. Pyrol., 93, 1-13 (2012).

21. Guo, Y; Rockstraw, D.A. Carbon 2006, 44, 1464.

22. Z. Ryu, H. Rong, J. Zheng, M. Wang, B. Zhang, Carbon, 40, 1144-1147 (2002).

23. S.-J. Park, K.-D. Kim, Carbon, 39, 1741-1746 (2001).

24. F. Zeng, D. Pan, Cellulose 15 (2008) 91.

25. C-I. Su and C-L. Wang, Fibers and Polymers, 8, 482-486 (2007).

26. S.-J. Park, K.-D. Kim, Carbon, 39, 1741-1746 (2001).

27. J. Lahaye, Fuel, 77, 543-547 (1998)

28. P.H. Wang, Z.R. Yue, and J. Liu, J. Appl. Polym. Sci., 60, 923-929 (1996).

Received 26 Ocak 2023; Received in revised form 15 February 2023; Accepted 22 February 2023;
Available online 20 March 2023;
doi: 10.5281/zenodo.7955648

29. Hindeleh A.M., Johnson D.J., and Montague P.E., 1983, *Fibre Diffraction Methods*, ACS Symp. No. 141 (Eds. A.D. French and K.H. Gardner), American Chemical Society, Washington DC, p. 149-181.

30. A.M. Hindeleh and D.J. Johnson, *Polymer*, 19, 27 (1978).

31. M. Jagtoyen and F. Derbyshire, *Carbon*, 36, 1085 (1998).

32. D.F. Arseneau, *Canadian J. Chem.*, 49, 632 (1971).

33. M.M. Tang and R. Bacon, *Carbon*, 2, 211 (1964).

34. K. Gurudatt and V.S. Tripathi, *Carbon*, 36, 1371 (1998).

35. M.V. Ramiah, *J. Appl. Polym. Sci.*, 14, 1323 (1970).

36. L.Y. Mwaikambo and M.P. Ansell, *J. App. Polym. Sci.*, 84, 2222 (2004).

37. F. Suarez-Garcia, A. Martinez-Alonso, and J. M.D. Tascon, Carbon, 42, 1419 (2004).

38. M.M. Tang and R. Bacon, *Carbon*, 2, 221 (1964).

39. J. Laine and A. Calafat, *Carbon*, 29, 949 (1991).

40. A.M. Hindeleh and D.J. Johnson, *Polymer*, 15, 697 (1975).

41. F. Carillo, X. Colom, J. Valldeperas, D. Evans, M. Huson, and J. Church, *Textile Res. J.*, 73, 1024 (2003).

Received 26 Ocak 2023; Received in revised form 15 February 2023; Accepted 22 February 2023;
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Table 1. Characteristics of untreated and oxidized viscose rayon fibers pretreated with the phosphoric acid solution

Stabilization temp (°C)	**Colour Change**	**Burning Test**	**Fiber thickness* µm**	**Fiber thickness change (%)**	**Linear density (Tex)**	**Linear density change (%)**
Untreated	White	Fail	28.1 (0.3)	0.0	32.6	0.0
150	Light brown	Fail	24.0 (0.4)	-14.6	31.7	-2.8
175	Dark Brown	Fail	23.4 (0.5)	-16.7	30.1	-7.7
200	Black	Fail	18.0 (0.5)	-35.9	27.3	-16.3
225	Black	Pass	17.8 (0.5)	-36.7	21.3	-34.7
250	Black	Pass	17.4 (0.5)	-38.1	18.2	-44.2

Values in parantheses are given as standard deviations

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Table 2. Resolved peak parameters of curve fitted equatorial X-ray diffraction trace of untreated viscose rayon fiber

Peak ref.	**f**	**A (Height)**	**W (Width)**	**Position (2θ)**	**d-obs spacing (nm)**	**d-calc spacing (nm)**
$\bar{1}10$	0.4	74.0	2.42	12.15	0.728	0.731
Amorph.	1.0	43.0	10.46	19.43	0.456	-
110	0.5	116.0	2.32	20.10	0.441	0.441
020	1.0	94.5	2.08	21.92	0.405	0.402

Table 3. Characteristic IR bands from the infrared spectrum of untreated viscose rayon fiber [47, 48]

Wavenumber (cm^{-1})	**Intensity**	**Assignment**
3455	very strong	OH stretching of intramolecular hydrogen bonds
3180-3170	shoulder	OH stretching of intramolecular hydrogen bonds
3000-2800 (2895)	very strong	C-H stretch in cellulose II
1650	strong	OH stretching of absorbed water
1424	strong	CH_2 symmetrical bending
1375	strong	CH bending
1337	medium	OH in-plane bending
1318	medium	CH_2 wagging
1280	weak	CH bending
1264	weak	-
1228	weak	OH in-plane deformation
1205-1200	weak	OH in-plane deformation
1165	broad	C-O-C asymmetric stretching
1110	broad	Ring asymmetric stretching
1055	broad	C-O stretch
1015	weak	C-O stretch
986	weak	C-O stretch
895	strong	C-O-C stretching of cellulose ring

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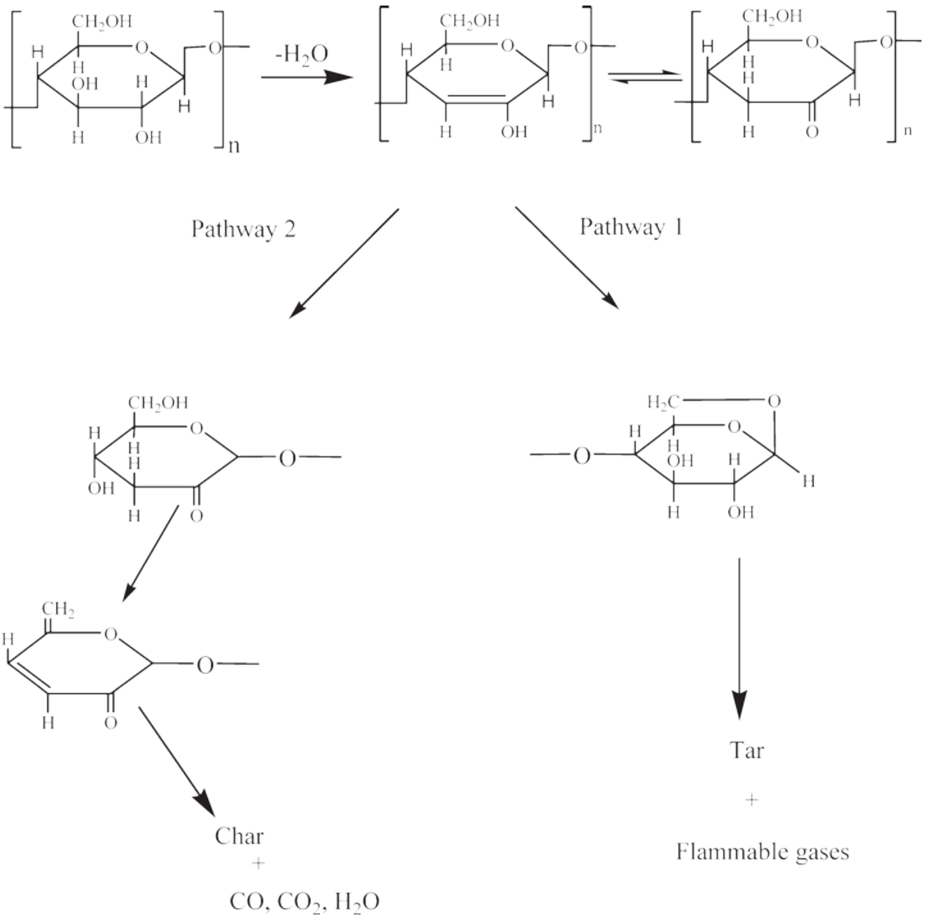

Figure 1. Reactions involved during the pyrolysis of viscose rayon fibers in the presence of a catalyst (Li, H.; Yang, Y.; Wen, Y.; Liu, L. *Comp. Sci. Tech.* 2007, *67*, 2675.)

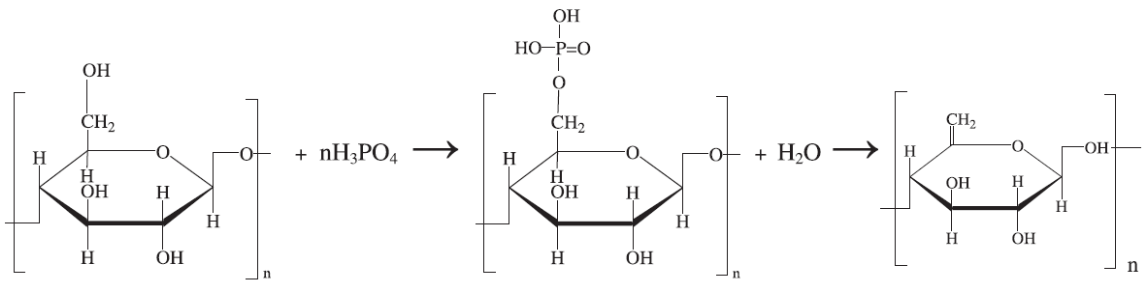

Fig. 9. Interaction of primary hydroxyl (CH_2—OH) groups in cellulose with phosphoric acid resulting from decomposition of DAP.

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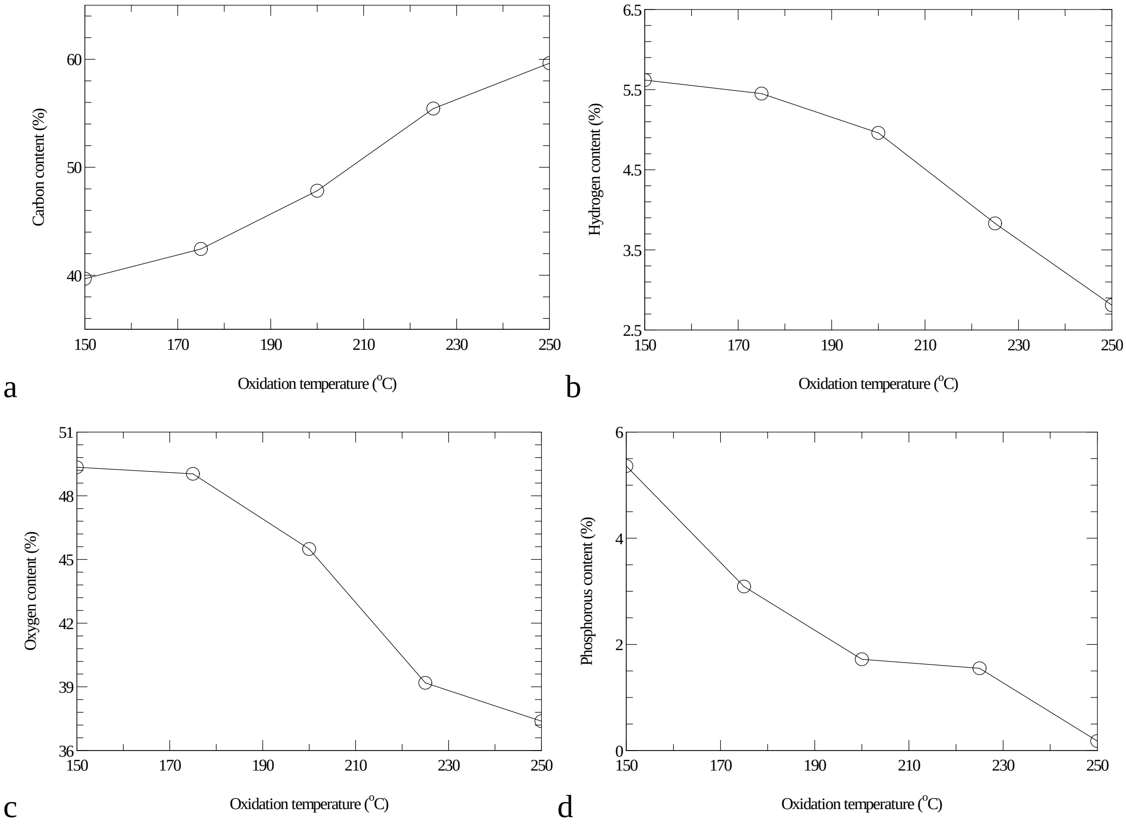

Figure 2. Elemental analysis of oxidized viscose rayon fibers pretreated with phosphoric acid as a function of oxidation temperature. (a) carbon (% wt.); (b) hydrogen (% wt.); (c) oxygen (% wt.); (d) phosphorous (% wt.).

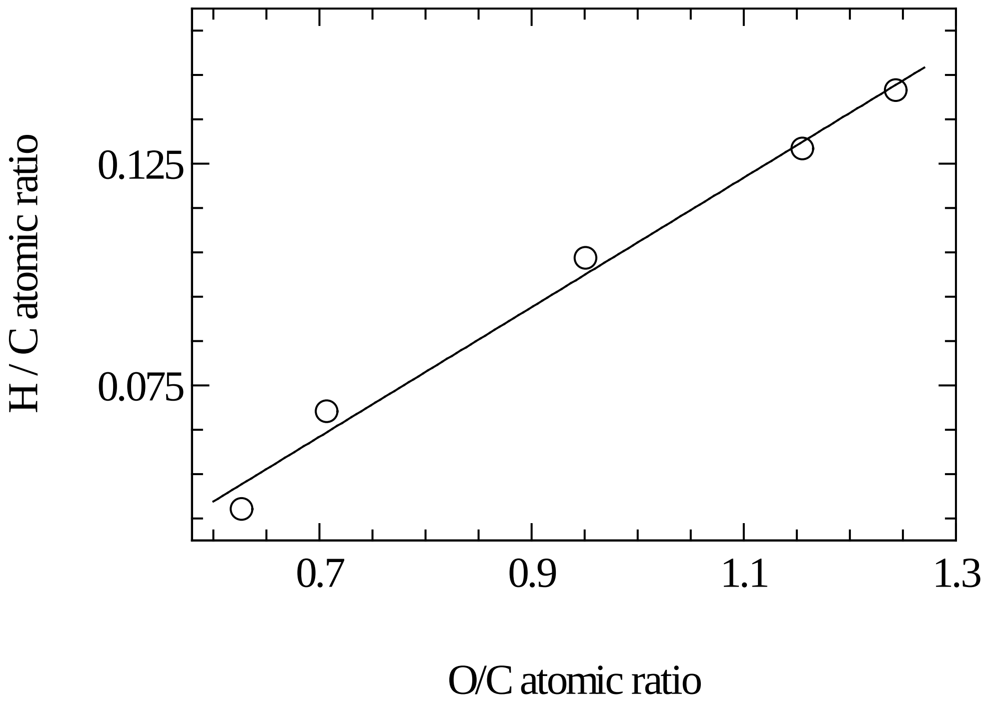

Figure 3. Comparison of H/C against O/C atomic ratios for the phosphoric acid pretreated and oxidized viscose rayon fibers as a function of oxidation temperature.

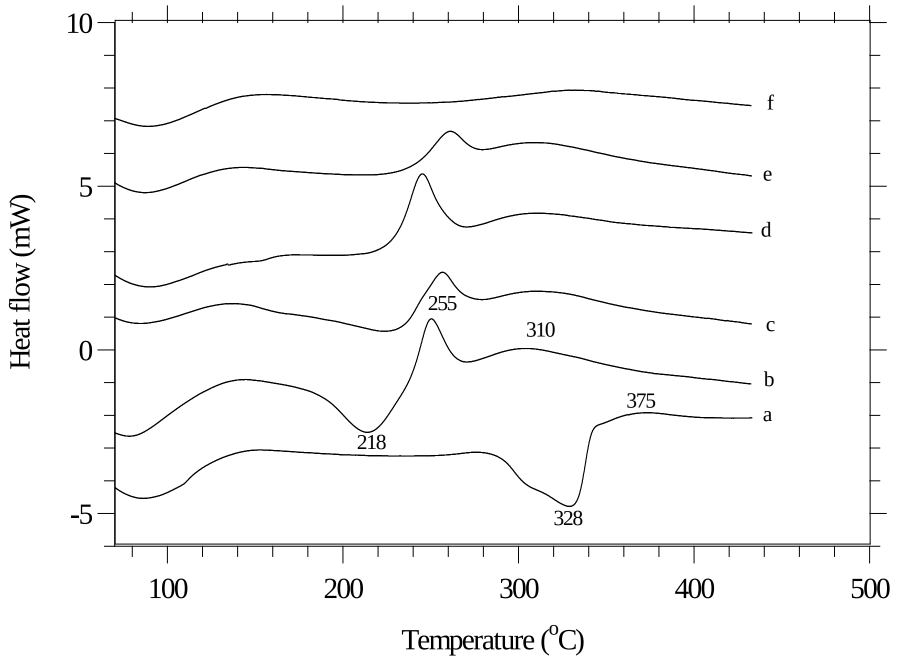

Figure 4. DSC thermograms of untreated (a) and phosphoric acid pretreated and oxidized viscose rayon fibers as a function of oxidation temperature. (b) 150 ºC, 30 min; (c) 175 ºC, 30 min; (d) 200 ºC, 30 min; (e) 225 ºC, 30 min; (f) 250 ºC, 30 min.

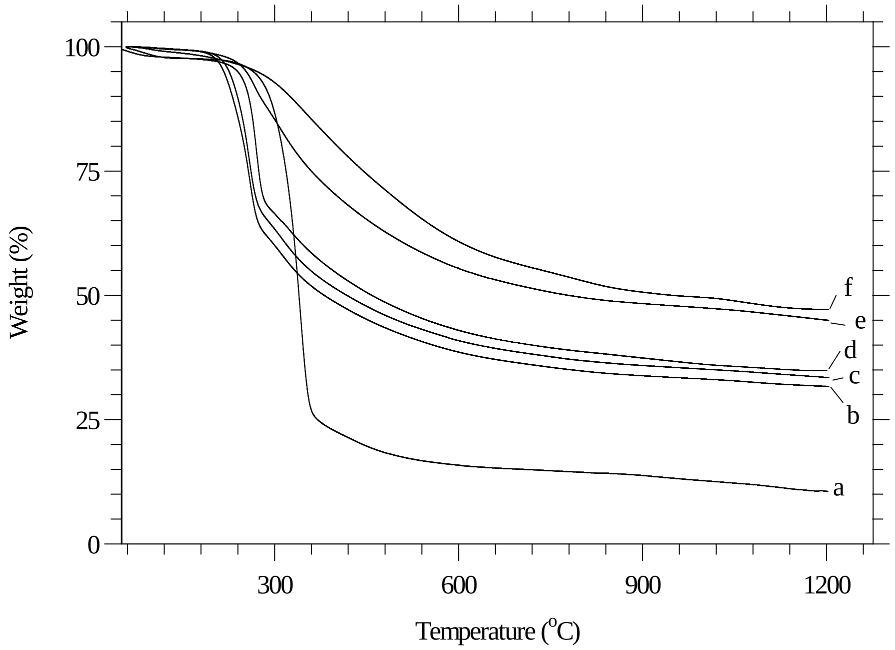

Figure 5. TGA thermograms of untreated (a) and phosphoric acid pretreated viscose rayon fibers oxidized at (b) 150 °C, 30 min; (c) 175 °C, 30 min; (d) 200 °C, 30 min; (e) 225 °C, 30 min; (f) 250 °C, 30 min.

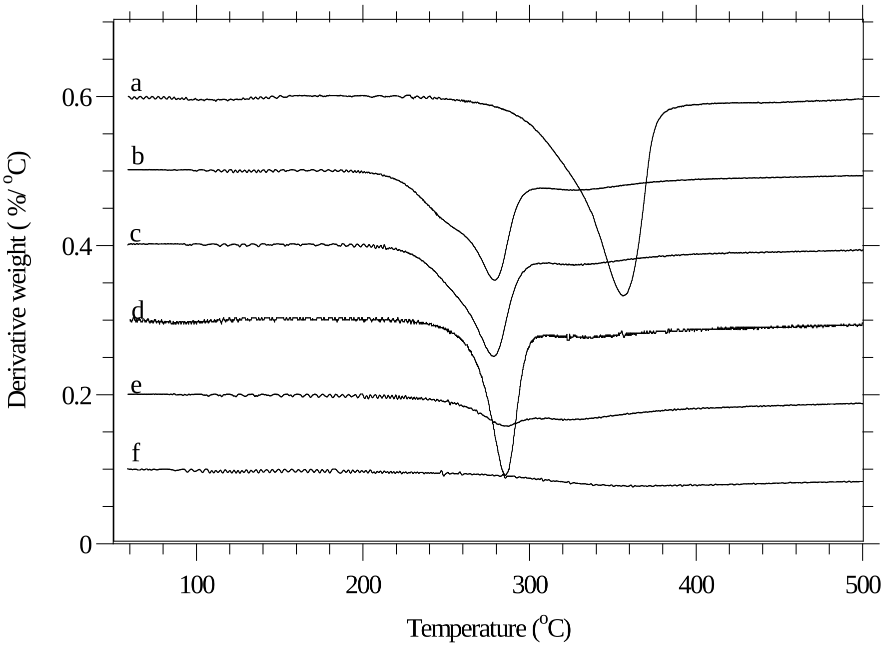

Figure 6. Derivative thermogravimetric curves of untreated (a) and phosphoric acid pretreated viscose rayon fibers oxidized at (b) 150 °C, 30 min; (c) 175 °C, 30 min; (d) 200 °C, 30 min; (e) 225 °C, 30 min; (f) 250 °C, 30 min.

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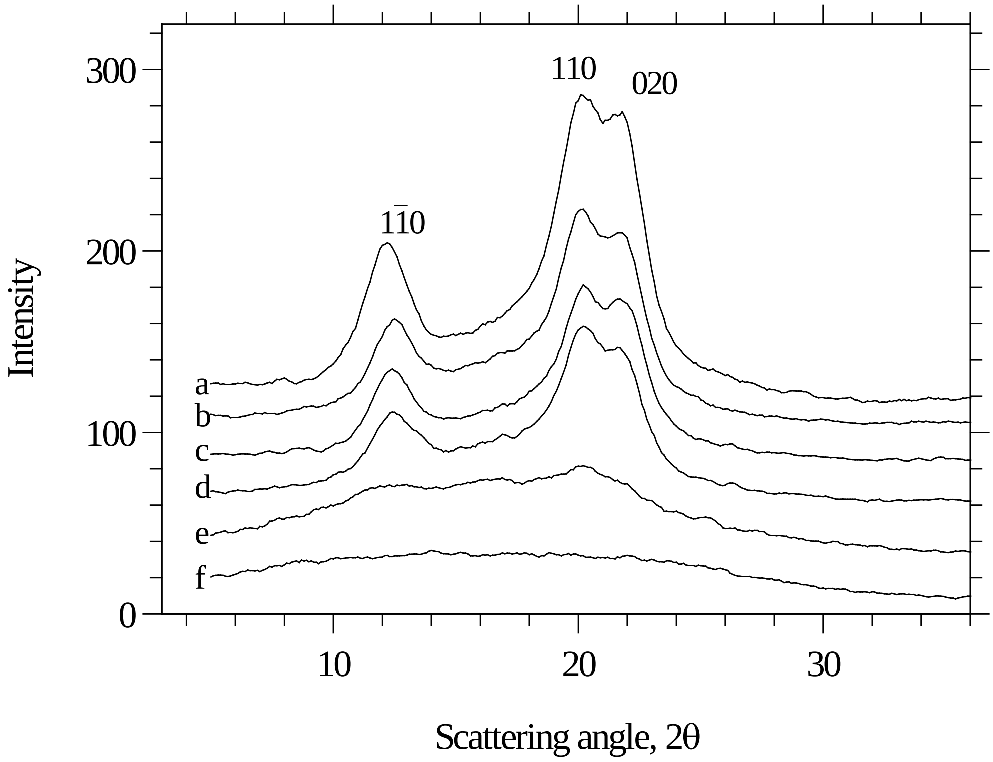

Figure 7. Equatorial X-ray diffraction traces of untreated (a) and phosphoric acid pretreated viscose rayon fibers oxidized at (b) 150 °C, 30 min; (c) 175 °C, 30 min; (d) 200 °C, 30 min; (e) 225 °C, 30 min; (f) 250 °C, 30 min.

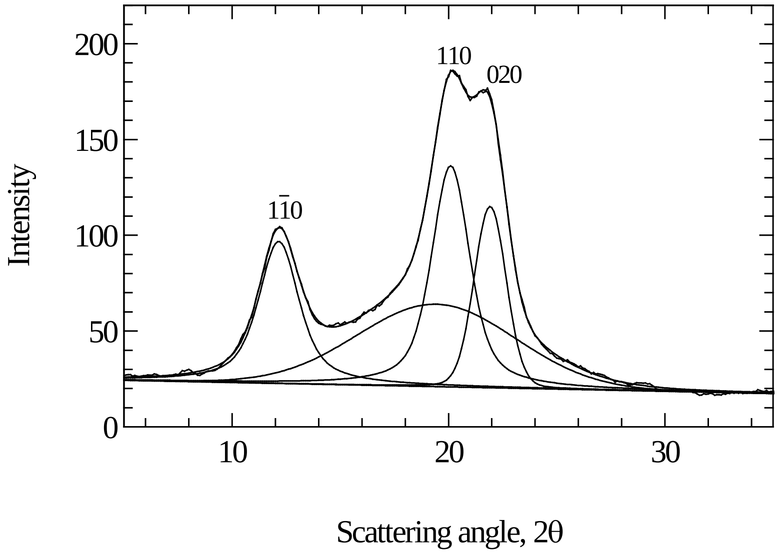

Figure 8. Curve fitting of equatorial X-ray diffraction trace of untreated viscose rayon fiber

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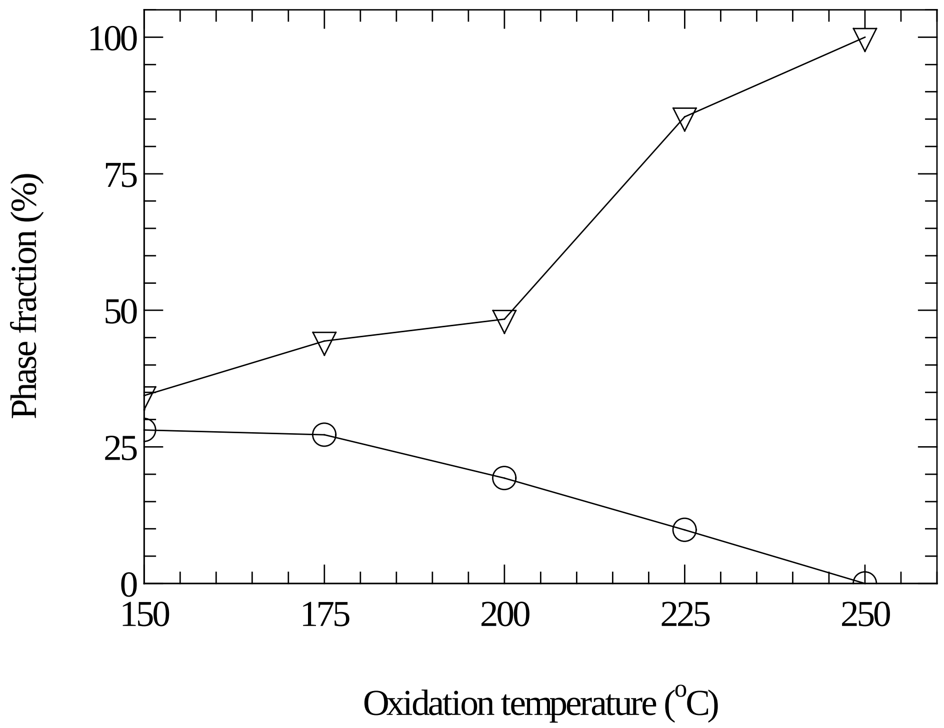

Figure 9. Variation of phase fractions of phosphoric acid pretreated and oxidized viscose rayon fibers as a function of oxidation temperature. (∇) amorphous phase; (O) crystalline (cellulose II) phase.

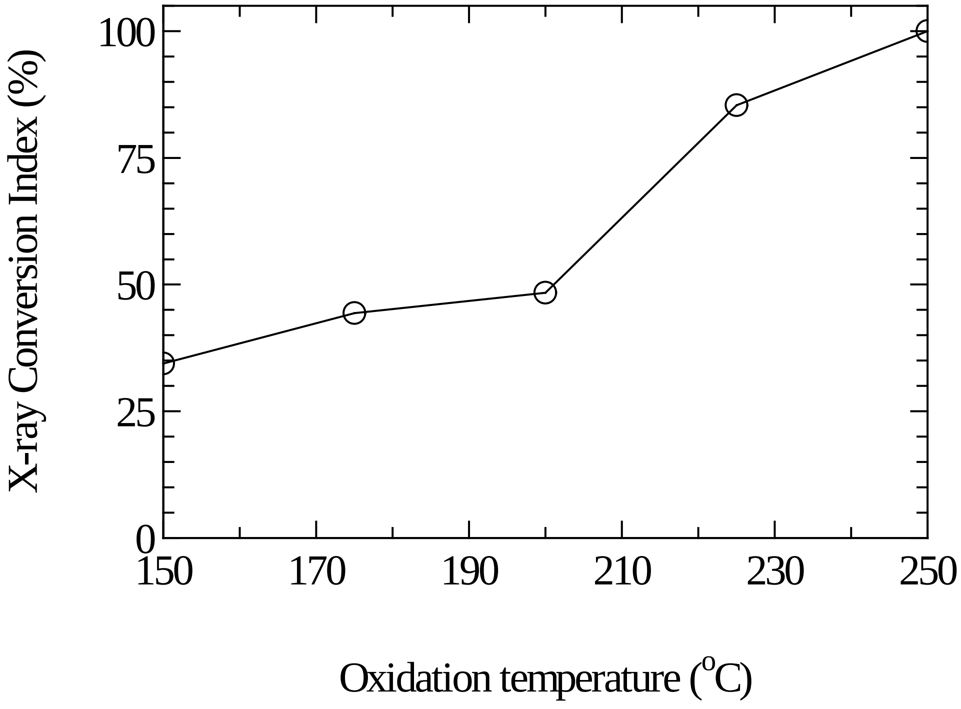

Figure 10. Variation of X-ray conversion indices of untreated and phosphoric acid pretreated and oxidized viscose rayon fibers as a function of oxidation temperature.

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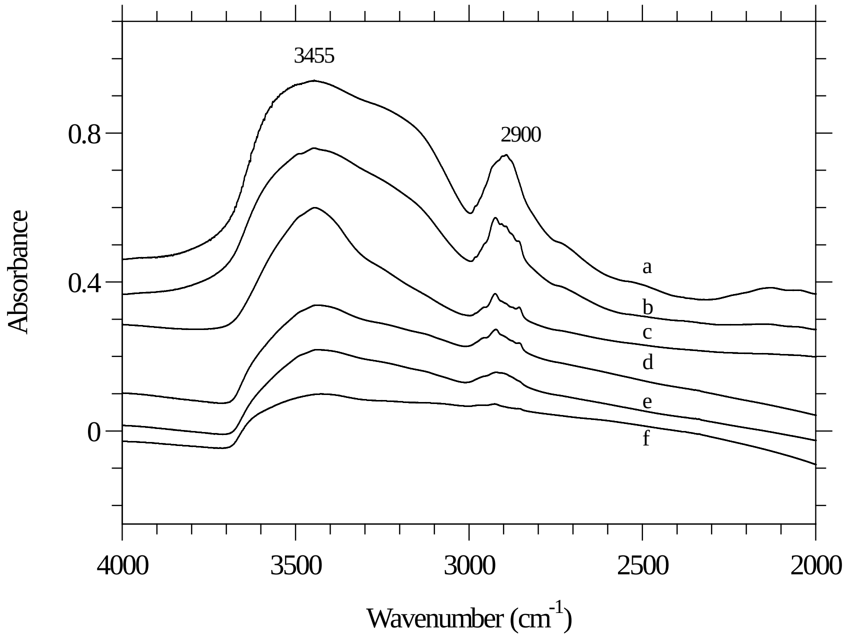

Figure 11. Infrared spectra of untreated (a) and phosphoric acid pretreated viscose rayon fibers oxidized in the air for 30 min as a function of oxidation temperatures. (b) 150 °C; (c) 175 °C; (d) 200 °C; (e) 225 °C; (f) 250 °C.

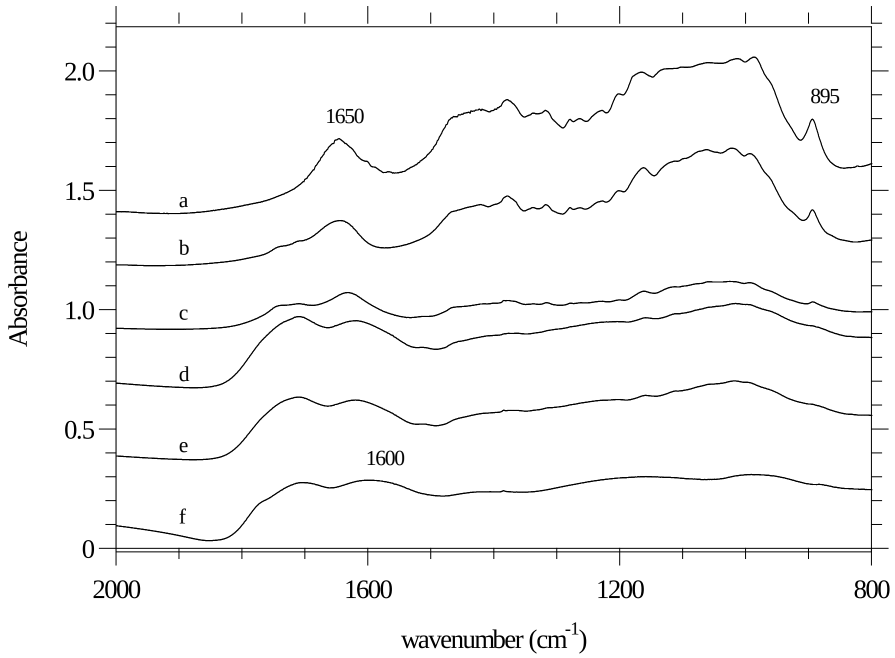

Figure 12. Infrared spectra of untreated (a) and phosphoric acid pretreated viscose rayon fibers oxidized in the air for 30 min as a function of oxidation temperatures in the 2000-800 cm^{-1} region. (b) 150 °C; (c) 175 °C; (d) 200 °C; (e) 225 °C; (f) 250 °C.

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