



# **Evaluating the Role of Antioxidants in the Stabilization of Hydroxypropyl cellulose by ATR-FTIR Spectroscopy**

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#### ARTICLE INFO

Article history: Received: 03 Mar 2018 Final Revised: 24 Apr 2018 Accepted: 25 Apr 2018 Available online: 1 May 2018 Keywords: Polymer degradation Hydroxypropyl cellulose (HPC) Oxidation Antioxidant ATR-FTIR

# ABSTRACT

This study is aimed at evaluating the performance of tetrakis[methylene-3-(3,5-di-tert-butyl-4-hydroxyphenyl) propionate] methane (S1010), tris(2,4-di-tert-butylphenyl) phosphite and ascorbic acid antioxidants in stabilizing hydroxypropyl cellulose. For this purpose, films with weight percent ratio of 0.4 % (w/w) were prepared in 1 % isopropanol solution (w/v). Then, the films were tested under three accelerated aging conditions, i.e. light (UV; 216hr), drv-heat (120°C; 240 hr) and moist-heat (70°C; RH: 95%; 240 hr). During the aging processes, structural changes were studied using attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy based on the defined destruction indices. In addition, color changes of the films were evaluated by colorimetry in CIELab system before and after aging processes. According to the results, S1010 phenolic antioxidant improved color and structural stabilization of HPC against environmental destructing factors in photooxidation, thermooxidation and hydrothermal degradation processes. Prog. Color Colorants Coat. 11 (2018), 93-101© Institute for Color Science and Technology.

### 1. Introduction

Cellulose is a linear crystalline polymer of  $\beta$ 1,4-D-Glucose that, with its derivatives, forms one of the most important natural macromolecular groups [1, 2]. Despite various cellulose derivatives, cellulose ethers have been constantly developing in world markets because of their availability, easy transportation, small toxicity effects and diversity [3]. With the help of etherization process of hydroxyl groups of anhydroglucose, it is possible to produce cellulose

(which is insoluble in water) derivatives that are soluble in water such as methyl cellulose (MC), hydroxypropyl cellulose (HPC), hydroxypropyl methyl cellulose (HPMC), hydroxyethyl cellulose (HEC) and ethyl hydroxyethyl cellulose (EHEC) [4]. HPC is one of the most important chemical etheric derivatives of cellulose. HPC is a non-toxic solid biodegradable polymer [5, 6]. It is mainly used as coatings, excipients, encapsulations, binding materials, foaming agents, protection colloids, prodrugs, flocculants, and a

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wide range of applications in food, medicine, paper, ceramic and plastic industries [7-10]. It is a non-ionic material that possesses a lower reactivity with active ingredients in comparison to ionic excipients [11]. This material, being a hydrophilic derivative of cellulose, not only shows a specific phase transfer behavior in aqueous solutions and some other solutions, but also owns a large number of privileges such as excellent film forming characteristics, decomposition and environmental compatibility [12-15]. The ability to increase the viscosity of aqueous solutions appears to be among its most outstanding features [3]. On the other hand, characteristics of this material has led to its widespread use in conservation of historic objects as well as its application as a gel forming agent, consolidant agent, sizing factor and glue [16].

Generally, HPC film can act as an acceptable barrier to moisture and oxygen [7]. However, polymers do not have a stable structure and may be destructed under different conditions, which decreases their efficiency and performance. Polymer degradation processes can be totally categorized as bio-, chemical, thermal, radiolytic, mechanical and photo- degradation [17]. Meanwhile, environmental factors happen to be of utmost effect in polymer degradation and their efficiency under various conditions. Environmental effects such as changes in temperature, relative humidity and UV ray can lead to oxidation, hydrolysis and breaking of macromolecules chains and intermolecular bonds [18]. Photodegradation of polymers is known as the main reason for their limited applications in outdoor environments [11]. Photodegradation, which occurs due to the UV ray, is a serious problem in sunniest climates. Photooxidation releases free radicals that initiate a series of fastdeveloping chain reactions that result in fast deterioration of mechanical characteristics [11, 19]. Thus, almost all synthetic polymers need to be stabilized against incompatible environmental effects, which in turn, expedite development of new stabilizers. These compounds attempt to target probable destruction ways as thermolysis, thermooxidation, photolysis, such photooxidation and radiolysis to elongate the lifetime of polymers [17]. Generally, main active reactions on the structure of cellulose, that tend to create structural changes on it, include photodegradation, acid hydrolysis, oxidation and biodegradation which are all caused by environmental effects [20]. Hence, it looks necessary to find ways for reducing or ceasing the damage caused by factors such as heat, light, humidity or oxygen. On this basis, this study

aims to investigate the stability of HPC under three conditions of photo, thermal and hydrothermal degradation to evaluate the performance of tetrakis[methylene-3-(3,5-di-tert-butyl-4-hydroxyphenyl) propionate] methane (S1010), tris(2,4-di-tert-butylphenyl) phosphite (S1680) and ascorbic acid (AA) as antioxidation agents in stabilization of HPC based on Fourier transform infrared (FTIR) spectroscopy.

#### 2. Experimental

#### 2.1. Materials

Hydroxypropyl cellulose (HPC) with a commercial name of Klucel G was purchased from Lascaux Colours & Restauro Co. (Brüttisellen, Switzerland). Tetrakis [methylene-3-(3,5-di-tert-butyl-4-hydroxyphenyl) propionate] methane with a commercial name of Songnox 1010 (S1010), as a phenolic antioxidant, and tris (2,4-ditert-butylphenyl) phosphite with a commercial name of Songnox 1680 (S1680), as a phosphite antioxidant, were purchased from Songwon Industrial Co., Ltd. (Ulsan, South Korea). Ascorbic acid (AA) was obtained from Acros Co. (Geel, Belgium). Isopropanol was purchased from Merck chemical company (Darmstadt, Germany).

#### 2.2. Preparation of film samples

S1010, S1680 and AA as antioxidant agents of 0.4 % ratio (m/m) were added to HPC. Thereafter, compounds (HPC, HPC+1010, HPC+S1680 and HPC+AA) were prepared with a concentration of 1 % (m/v) in isopropanol and then were settled as a film layer on a glass plate ( $2 \times 5$ cm). After drying in laboratory air for 72 h, the effects of antioxidants on HPC films characteristics were studied.

#### 2.3. Accelerated aging

Structural changes of films were studied under three accelerated aging conditions. A UV lamp (UV-C) was used to observe photooxidation of the prepared films at a distance of 20 cm from UV source for 216 hours. Thermooxidation changes were also checked at a temperature of 120 °C for 240 hours. Furthermore, films endured a temperature of 70 °C and a relative humidity of 95% for 240 hours in order to assess the hydrothermal degradation.

#### 2.4. ATR-FTIR Spectroscopy

ATR-FTIR analysis were carried out using a Nicolet

470 FTIR spectrometer and OMNIC 6.1a software (Nicolet instrument corporation, USA) equipped with PIKE MIRacle attenuated total reflectance (ATR) accessory with zinc selenide (ZnSe) crystal plate. All Spectra were collected in the range of 4000-650 cm<sup>-1</sup> at 4 cm<sup>-1</sup> resolution with 32 scans. To avoid any spectral variation due to various factors, air background spectrum was collected before each sample analysis.

#### 2.5. Colorimetry

Colorimetry in the CIE lab color spaces is used to study the color changes in sample during aging process. Meanwhile, 5 points were selected for each film and their average was reported. In this survey, a colorimeter device model Salutron® Colortector Alpha (made in Germany) was used. The total color difference ( $\Delta E$ ),  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ , the chroma coordinate ( $C^*$ ) and  $\Delta C^*$  were obtained as Eq. 1-6 [21].

$$\Delta E = \sqrt{\left[\left(\Delta L\right)^2 + \left(\Delta a\right)^2 + \left(\Delta b\right)^2\right]}$$
(1)

$$\Delta L^* = L^*_{2} - L^*_{1} \tag{2}$$

$$\Delta a^* = a^*_{2} - a^*_{1} \tag{3}$$

$$\Delta b^* = b^*_{2} - b^*_{1} \tag{4}$$

$$C = \sqrt{a^2 + b^2} \tag{5}$$

$$\Delta C^* = C^*_{2} - C^*_{1}$$

(6)

#### 3. Results and Discussion

#### 3.1. ATR-FTIR spectroscopic studies

#### 3.1.1. Photooxidation

Figure 1 exhibits the ATR-FTIR spectra pertaining to HPC and other films containing antioxidants in the range of 775-1850 cm<sup>-1</sup> for various irradiation times. Spectra changes during aging process shows the increase in the absorption peak around 1730 cm<sup>-1</sup> which corresponds to stretching vibrations of carbonyl groups [20-23]. This peak is known as the carbonyl index in the FTIR spectrum. Normally, when polymer is exposed to UV irradiation, this peak increases with the exposure time, similar to the spectra shown in Figure 1 [11]. Formation of this peak, which itself admits oxidation [20], is considered as the oxidation index of cellulose and cellulose ethers in the FTIR spectrum [24, 25].

The oxidation reaction of cellulose structures usually includes interactions with hydroxyl groups that results in the formation of carbonyl [20]. This is suggested in Figure 2 as one of the proposed procedures for HPC photooxidation. For this reason, HPC photooxidation leads to a decrease in C-OH absorption and to an increase in C=O absorption in the FTIR spectrum. Reduction of absorption peak at 1325 cm<sup>-1</sup>, which corresponds to the bending vibration of C-O-H, can be seen in Figure 1 [18, 22].



Figure 1: ATR-FTIR spectra of films during accelerated photoaging process; a: HPC, b: HPC+AA, c: HPC+S1680 and d: HPC+S1010.

The increasing rate of the area under the peak at 1730 cm<sup>-1</sup> and the decreasing rate at 1325 cm<sup>-1</sup> during the aging process has been evaluated and reported in Figure 3a,b with respect to the area corresponding to 1460 cm<sup>-1</sup> as the internal standard peak. This assessment reveals the inability of AA and S1680 in preventing photooxidation of HPC, while S1010 phenolic antioxidant considerably prevents the decrease of 1325 cm<sup>-1</sup> absorption band (related to bending vibration of C-O-H) which in turn diminishes formation of carbonyl groups during oxidation.

Of course, oxidation of HPC does not solely result in reduction of 1325 cm<sup>-1</sup> absorption band; it also causes intensity deductions in absorption bands including 840, 935 and 1117 cm<sup>-1</sup> all related to asymmetrical stretching vibrations of C-O-C in the pyranose ring [26, 27], 1150 cm<sup>-1</sup> related to asymmetric stretching vibrations of C-O-C in the oxygen bridge [27, 28] and 1265 cm<sup>-1</sup> related to the stretching vibration of C-O [23]. In most of these cases, the changes were similar to those occurring in the case of 1325 cm<sup>-1</sup> and only S1010, as an additive, reduced the reaction rate. For instance, Figure 3c represents the changes in peak area around 1117 cm<sup>-1</sup> which is related to C-O-C in the pyranose ring. The reduction in this absorption band demonstrates the opening of pyranose ring during exposure to UV irradiation. In this case (i.e. again), the phenolic antioxidant has more efficiently prevented the destruction of ring structure.

#### **3.1.2.** Thermal oxidation

Figure 4 reports the ATR-FTIR spectra of films under study during dry-heat accelerated aging. As looking into HPC absorption bands admits, carbonyl groups become more in a wave number of about 1730 cm<sup>-1</sup> during the accelerated aging process which itself is evidence of oxidation [20]. Earlier studies also showed that thermooxidation of cellulose structures is usually accompanied by an increase in absorption of carbonyl in the FTIR spectrum [29].



Figure 2: Reaction mechanism for the photooxidation of HPC [11].



**Figure 3:** The ratios of area under absorption bands fit at 1730 (a), 1325 (b) and 1117 (c) to 1460 cm<sup>-1</sup> as oxidation indices in ATR-FTIR of films during accelerated photoaging.



Figure 4: ATR-FTIR spectra of films during dry-heat accelerated aging process; a: HPC, b: HPC+AA, c: HPC+S1680 and d: HPC+S1010.



**Figure 5:** The ratio of area under absorption bands fit at 1730 to 1460 cm<sup>-1</sup> as an oxidation index in ATR-FTIR of films during dry-heat accelerated aging

The ratio of areas corresponding to 1730 and 1460 cm<sup>-1</sup> peaks is studied and reported in Figure 5, as the evaluation index of oxidation extent and with the purpose of assessing the performance of antioxidants in preventing the thermooxidation. Generally, all three antioxidants under research have had acceptable performances in prevention of HPC oxidation. At the same time, the oxidation index of the film without any antioxidant shows a considerable raise with time. This issue can be attributed to the formation of destruction products during the accelerated aging process. However, for the film containing S1010, temporary formation of absorption band in the vicinity of 1716 cm<sup>-1</sup> can be due to the formation of unstable products during thermooxidation. Anyhow, this film shows the

least fluctuation intensity among others, especially around  $1060 \text{ cm}^{-1}$  related to C-O vibration [3, 10].

#### 3.1.3. Hydrothermal degradation

Figure 6 exhibits the ATR-FTIR spectra of films during the accelerated aging process at a relative humidity of 95 % at 70 °C. The most outstanding change is the increase of absorption in the range of 1550-1700 cm<sup>-1</sup>, especially after 240 h. The absorption peak at 1630 cm<sup>-1</sup> is related to the bending vibration of absorbed water molecules [29, 30]. In addition, it is known that hydrothermal degradation of cellulose structures generally increases the absorption band intensity around 1610 cm<sup>-1</sup>. This absorption band corresponds to ketone groups formed during hot-humid

aging process, act as chromophore groups [31], and overlap with the absorption band related to the bending vibration of water molecules.

Proniewicz et al. showed that in FT-IR spectrum of the cellulose structure during hot-humid aging, the maximum changes are observed at 1420 cm<sup>-1</sup> and 1320 cm<sup>-1</sup> [22]. Their study revealed the raise in the absorption band at 1420 cm<sup>-1</sup> and its fall at 1320 cm<sup>-1</sup>. Thus, in the study of hydrothermal degradation of HPC, the ratio of areas corresponding to 1415 and 1325 cm<sup>-1</sup> was considered as the destruction assessment index (Figure 7a). Moreover, the ratio of areas under 1415 and 1265 cm<sup>-1</sup> peaks was also investigated. The results are shown in Figure 7b. Results imply the degradation rate decrease in the film containing phenolic antioxidant. Meanwhile, other antioxidants seem to have no significant effect on controlling the destruction trend.

Commonly, humidity at high temperatures causes hydrolysis of glycosidic [29] and breaks connections 1-4 in the cellulose chain. Consequently, the absorption band related to asymmetric stretching vibration of C-O-C in the oxygen bridge decreases and, based on the procedure proposed in Figure 8, the formation of OH groups is yielded. For this reason, the ratio of the area under 3500 cm<sup>-1</sup> peak (related to the stretching vibration of OH [2, 32]) to that under 1150 cm<sup>-1</sup> (related to the stretching vibration of C-O-C) was considered as the hydrolysis degradation index. The results obtained from these tests are presented in Figure 7c. Generally, antioxidants prevent destruction to different extents among which the phenolic one shows a better efficiency in preventing the hydrolysis of HPC.



Figure 6: ATR-FTIR spectra of films during hot-humid accelerated aging process; a: HPC, b: HPC+AA, c: HPC+S1680 and d: HPC+S1010







Figure 8: The hydrolysis mechanism of HPC.



Figure 9: Color changes of films after accelerated photoaging (a), accelerated dry-heat (b) and hot-humid aging (c).

#### 3.2. Colorimetric studies

Figure 9 indicates the color evaluation of films before and after aging processes according to CIELab color coordinates system. In this Figure,  $\Delta L$  is indicative of the brightness difference index,  $\Delta C$  shows chroma difference index and  $\Delta E$  is the total color difference of the films [33].

This study suggests the small difference between color indices in films containing AA and the S1680 phosphite antioxidant after accelerated photoaging process. However, they cannot be referred to as antioxidation agents for HPC against photodegradation due to low structural stability of the films. This is while not only does S1010 does decelerate the photooxidation of HPC, but also reduces the color changes caused by the aging process.

One can recognize that the least color change in accelerated heat-aging occurs for the film containing phenolic antioxidant. The color changes are most evident for HPC+AA, especially in the color parameter b<sup>\*</sup>. The color of this film after heat aging was yellowish. This can be of importance in cases where color conserving and visual features are required.

In accelerated hot-humid aging, the film containing

phenolic antioxidant seems to undergo a lower color change because of the reduction in the hydrothermal degradation of HPC and its products which act as chromophore factors. This, regarding decrease in the total color difference according to  $\Delta E$  proposed in Figure 9, can be readily observed.

#### 4. Conclusions

The findings of this study showed the structural instability of HPC against destructive environmental factors. According to the ATR-FTIR spectroscopy, the stretching vibrations due to the presence of carbonyl increased under the effect of thermo- and photooxidation which was later assigned as a scale for degradation evaluation. Investigating the performances of antioxidants revealed the decrease in degradation of HPC via photooxidation by tetrakis[methylene-3-(3,5di-tert-butyl-4-hydroxyphenyl) propionate] methane (S1010). This antioxidant not only reduced the destruction rate in thermooxidation of HPC, but also caused its color stabilization. In addition, the S1010 phenolic antioxidant controlled the hydrothermal and hydrolysis destruction of HPC and reduced the extent of its color change. Hence, according to the results of performance assessment of the three ascorbic acid, phenolic and phosphite antioxidants, the S1010 phenolic antioxidant remarkably improved the stability of HPC in all three photooxidation, thermooxidation and hydrothermal degradation processes.

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#### Acknowledgment

The authors gratefully acknowledge Mohsen Mohammadi Achachlouei, assistant professor in art university of Isfahan, Isfahan, Iran, for his helpful collaboration in supplying Songwon products.

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How to cite this article:

H. Ahmadi, S. Mallakpour, A. Koochakzaei, Evaluating the Role of Antioxidants in the Stabilization of Hydroxypropyl cellulose by ATR-FTIR Spectroscopy. Prog. Color Colorants Coat., 11 (2018), 93-101.

