퍼라이오데이트 산화된 셀룰로오스의 양이온화

요리용, 최형민, 김주용
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Cationization of Periodate Oxidized Cellulose

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1. Introduction

Periodate oxidation of cellulose leads to a selective cleavage of C-2 and C-3 vicinal hydroxyl groups within glucose to yield dialdehyde units. The dialdehyde cellulose is an important derivative for additional reactions.[1] In this paper, cationized cellulose was prepared from dialdehyde cellulose with choline chloride (CC) for a potential affinity membrane application. CC is inexpensive and very safe chemicals approved for a food additive. The cationized cellulose was analyzed for its characteristics by FTIR, TGA, SEM (with energy dispersive spectroscopy attachment), elemental analysis, and dye affinity measurement.

2. Experimental

2.1 Materials

Filter paper, CI Acid Red 4, and all chemicals such as sodium periodate, CC, aluminum sulfate, glycerol, acetic acid, and sodium sulfate used were of analytical grade. Deionized water was used throughout the work.

2.2 Methods

Filter paper (10g) was immersed in 500mL water, and 10.7g (0.1mol) sodium periodate was added to the bath. The bath containing the filter was stirred gently in the dark and oxidized under room temperature. After oxidation, the filter sample was suspended in 0.1mol/L aqueous glycerol solution for 0.5h and washed up to neutral. Lastly, the sample was placed in deionized water for 24h at an ambient temperature and then air-dried. The oxidized cellulose filter was immersed in the aqueous bath containing CC (20g/L) and aluminum sulfate catalyst (10g/L). Liquor ratio was 30:1. The padded sample with wet pick-up 80-85% was dried at 85℃/10min, cured at 150℃/5min in the curing oven, then thoroughly washed, and dried at a room temperature. CI Acid Red 4 dye was used to confirm the presence of cationic group in the treated cellulose. Dyeing solution contained dye (10g/L) and sodium sulfate (6g/L) was prepared, the pH was adjusted to 3-4 with acetic acid solution (0.1mol/L), and liquor ratio was 30:1.

3. Results and discussion

Fig.1 shows spectra of the samples. A spectrum (a) is for the untreated cellulose filter paper. Characteristic cellulose peaks such as 3300cm^{-1} for O–H stretching, 3000–2800cm^{-1} region for C–H stretching are shown. On the other hand, periodate-oxidized cellulose in spectrum (b) shows 1733cm^{-1} absorption peak for C=O stretching of aldehyde group. Intensity of the 1733cm^{-1} peak in dialdehyde cellulose, however, is rather

weak, because of hemiacetal formation between aldehyde and neighboring hydroxyl groups (or water).[2] The presence of hemiacetal was further confirmed by 890cm^{-1} peak in the spectrum (b). The intensity of this peak was enhanced after oxidation. Meanwhile, the absorption peaks at 1054cm^{-1} (−OH distortion vibration), 1203cm^{-1} (−OH in-plane bending), 1108cm^{-1} (C−H deformation stretch vibration), and 1160cm^{-1} (asymmetry C−O−C stretch vibration) became weak after oxidation. This suggested partial decomposition of cellulose during sodium periodate oxidation.[3] The presence of attached CC to oxidized cellulose was not able to confirm by FTIR as shown in spectrum (c), *i.e.*, no C-N bond, because of quaternized nitrogen. However, the intensity of peaks around 1733cm^{-1} and 890cm^{-1} became weak, indicating decrease in aldehyde contents after cationization. Other analytical methods needed to investigate evidence of cationic CC.

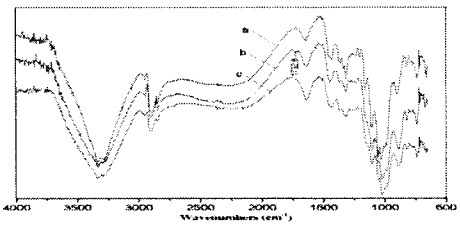

Fig.1 FTIR spectra of the cellulose filter samples: a)untreated; b)oxidized; c) cationized

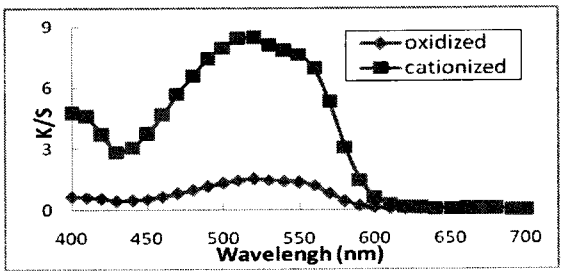

Fig. 2 K/S value

Fig.2 shows the K/S value of the samples. The K/S value is directly related to the color intensity of the fabric. The higher K/S value means the greater absorption in dyeing. The K/S value of cationized cellulose around 520nm was much greater than that of oxidized cellulose, indicating higher absorption of Acid Red 4 dye in the cationized cellulose. Cationic cellulose has an outstanding capacity of adsorption of anionic group in the dye through ionic bond between cationized cellulose (Cell-R-N^+) and acid dye (Dye-SO_3^-).[5] This revealed that the reaction occurred between CC and dialdehyde cellulose. Further analyses by using elemental analysis and SEM-EDS are underway to confirm the presence of nitrogen.

4. Conclusion

The cationized cellulose was prepared by the reaction of CC with the periodate-oxidized cellulose. FTIR showed the presence of aldehyde group after oxidation and the dye affinity analysis confirmed the reaction between aldehyde and hydroxyl group in CC. Cationization of periodate-oxidized cellulose is an efficient process to generate potential affinity membranes. The works are underway to confirm the presence of cationic molecule in the cellulose by more direct way.

REFERENCES

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