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Trace the exploitation of Egyptian rice straw through spectral and thermal measurements

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KEYWORDS

Cellulose; Xanthation; IR spectra; TGA; DTA and SEM analyses **Abstract** Cellulose from rice straw obtained at low sodium hydroxide solution concentration with high quality was used to obtain different cellulose derivatives through xanthation. Cellulose was then treated with carbon disulfide in the presence of sodium hydroxide. The viscose obtained was characterized with both chemical and instrumental analyses, namely, IR spectra, TGA and DTA analysis as well as SEM (scanning electron microscopy).

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1. Introduction

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Production of cellulose from Egyptian rice straw could be attained through many methods known generally for the extraction of cellulose from its sources (Abou-Sekkina et al., 2010) and especially from rice straw (Changa et al., 2011). In this study, cellulose was obtained using a semi-chemical method

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(treatment of mechanically divided rice straw with sodium hydroxide). The produced cellulose was described using IR analysis (Isaa and Saad, 1978; Saad et al., 1980). Cellulose cannot be dissolved in water. Introducing hydrophilic groups along the chain of cellulose cleaves the hydrogen bonds and renders its derivatives soluble in conventional solvents, widening its applications to, for example, functional cellulose ethers and esters (Issa et al., 2009; Abdel Mohdy et al., 2009; Tappi Committee, 1999; AARC, 1997).

Commercial cellulose derivatives are either ethers or esters that are soluble in water or organic solvents. The three free hydroxyl groups in the AGUs react with various functional substitution groups. The resultant substituents therefore disturb the inter- and intra-molecular hydrogen bonds in cellulose, reduce the hydrophilic character of the numerous hydroxyl groups, and increase the hydrophobicity. Introducing ester and ether groups separates the cellulose chains almost completely so that the fiber structure is either altered or destroyed. The solubility of a cellulose derivative in a solvent or in water depends on the type of substituents, the degree of substitution

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and the molecular weight. These cellulose derivatives are grouped according to the processes and chemical substituents (British Columbia Pellet Fuel Manufacturers Association, 1997; California Air Resources Board and California Department of Food and Agriculture, 1997; Clean Fuels Development Coalition and American Bioenergy Association, 1998; Fibre Crete International, 1997; Mwaikambo and Ansell, 2002).

The technology of the synthesis of viscose from raw materials has certain problems (Stenius, 2000; Abdullaeva, 1993) connected with low rate of the process and impossibility of preparation of a product with high homogeneity on some qualitative indices. This is due to low reactivity and mass-diffusion of rice straw cellulose during the treatment with aqueous solution of sodium hydroxide. It has been established, as a result of experiments that, with the use of raw material mixtures for complete treatment of rice straw, the arising problems studied must be solved.

In the present research work, the experiments for the preparation of laboratory parts of cellulose pulps from rice straw were carried out. The obtained samples were allowed for IR spectra, TGA and DTA analysis as well as chemical analysis (permanganate number determinations) for comparison between xanthated and original samples. Scanning electron microscopy (SEM) was also employed for micro-structural studies.

2. Experimental

2.1. Cooking conditions

In these tests, samples from rice straw were treated with NaOH solutions of different concentrations for different time periods at different temperatures.

2.1.1. Effect of sodium hydroxide concentration

Sodium hydroxide acts as a solubilizing agent for both silica and lignin found in raw materials. Samples (10 g) were boiled with 100 ml NaOH solution of certain concentrations (4%, 6%, 8%, 10% and 12%) for 2 h.

2.1.2. Effect of time at optimum alkalinity

Samples (10 g) were leached with 100 ml of 10% NaOH solutions and were boiled for different time periods (1, 2, 3 and 4 h).

2.1.3. Effect of weight/volume ratio

Samples (10 g) from rice straw were treated with 10% NaOH solutions while boiling for 2 h in different weight per volume ratios 1/5, 1/10, 1/20 and 1/50 (w/v).

2.1.4. Effect of cooking temperature on the yield of pulp

Samples (10 g) from rice straw were treated with 100 ml 10% NaOH solutions for 2 h at different degrees (40, 60, 80 and 100 $^{\circ}$ C).

2.1.5. Effect of the nature of rice straw on the pulp yield

Samples (10 g) from rice straw were treated with 100 ml of 10% NaOH solutions for 2 h at boiling point, length of samples used were the whole plant (80–120 cm length), 20 cm, 10 cm, 5 cm and mechanically divided plant (less than 1 cm).

2.2. Bleaching

The pulps produced by NaOH pulping are pale to intense yellow in color and require bleaching to reach acceptable brightness. The brightness of the bleached pulp depends on the bleaching sequences and conditions. So in this study the pulp was treated with sodium bisulfate solution (4% and 8%), and with sodium hydroxide solution (4%) then with hydrogen peroxide solution (4%) (Gustafsson and Peltonen, 2003).

2.3. Xanthation

Pulp (0.5 g) was exactly weighted, placed in a wide mouth bottle of 150 ml capacity, 100 ml of water was added to the pulp and it was allowed to swell in the closed bottle for at least 1 h. Fifty milliliters of NaOH solution (280.6/g/L), and 3.5 ml of carbon disulfide were added in this sequence, then the bottle was stoppered. The glass stopper must be fixed on the bottle as quickly as possible; the bottle was then shaken for 15 min and allowed to rotate in the thermostat at room temperature for 6 h.

2.4. Permanganate number (Nada et al., 1994)

It is a method of expressing the bleachability of pulp. It is determined by the number of mls of 0.1 N KMnO₄ consumed by one gram of moisture free pulp under certain specific conditions of time, temperature and acidity. Required volume of 0.1 N KMnO₄ (20-40 ml depending upon the rawness of the pulp) were paced in one beaker, an equal amount of 4 N H₂SO₄ is placed into another beaker and enough water combined with H₂SO₄, so that, the final reaction mixture of the permanganate solution will be 1/300 N. When the reagent is ready, the pulp specimen is added to the reaction beaker, followed by addition of sulfuric acid and then by addition of permanganate. After exactly 5 min at 25 °C an excess of KI is added to stop the reaction. The residual KMnO₄ in the mixture released an equivalent weight of iodine from the iodide salt solution. The liberated iodine is then titrated against standard sodium thiosulfate solution (Fahmy et al., 1979). The volume of permanganate consumed by the pulp is then calculated. The permanganate number is obtained by dividing the number of mls of 0.1 N KMnO₄ consumed by the moisture free weight of the test specimen; permanganate number $=\frac{25-V}{W}$ where V is the number of milliliters of 0.1 N Na₂S₂O₃ consumed in the titration, W is the weight of moisture free pulp and 25 is the number of ml of 0.1 N KMnO₄.

3. Equipments

IR absorption spectra were recorded as KBr discs within the 4000–200 cm⁻¹ range on a Perkin Elmer 1430 infrared spectrophotometer. The thermal gravimetric analysis (TGA) was carried out on a Shimadzu TG 50 thermogravimetric analyzer from room temperature up to 1000 °C using 10 °C/min heating rate under nitrogen as atmosphere. The differential thermal analysis (DTA) was performed on a 990 Du-Pont differential thermal analyzer of 1200 °C cell using Al₂O₃ as a reference. The surface of rice straw and pulp from rice straw samples imaged with the (SEM) scanning electron microscopy type, JEOL JEM-850 operating at 35 kV employed in the Central Laboratory, National

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