



# THEORETICAL INTERPRETATION OF THE CHANGES OCCUR ON CELLULOSIC WASTES AS A RESULT OF DIFFERENT CHEMICAL TREATMENTS

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**Keywords:** CAChe Program applications, cellulose modelling and chemical treatment.

In an attempt to find out what is happening during the chemical treatments of some agricultural residues (rice straw), as well as to link the theoretical aspects and the practical side, in an attempt to explain those changes that occur and thus control the quality and derivatives of the products of these treatment. Although theoretical studying cellulosic wastes, but this aspect of the studies and research has not obtained right after. The idea of this research on the essential elements, namely the first CAChe Program computer to design and expected IR behaviour for these materials, the second is what happened in the experimental treatments.

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## Introduction

Fourier transform infrared spectroscopy (FTIR) is a powerful technique for studying molecular structures. FTIR and molecular modelling proves that functional group like COOH could enhance the ability of the dry plant for mediating heavy metals.

The advantage of PM3 model as well as other semi-empirical methods that it provide a good spectroscopic data with moderate accuracy in appropriate computational time using personal computer.<sup>1</sup> Another advantage of semi-empirical models that the ability for providing structural and spectroscopic data for many systems and molecules mainly for understanding the vibrational spectrum of these systems.<sup>2,3</sup>

The interaction between cellulose and lignin is tried in the represent model through their O-linkage. The obtained model indicates that, the molecular point group is corresponding to C1 for cellulose, lignin as well as their mixtures. This indicate that no change in the symmetry of both cellulose and lignin as a result of their interaction. Further details of the interaction will be followed from their calculated spectra as indicated in Figure 9.

## Experimental Part

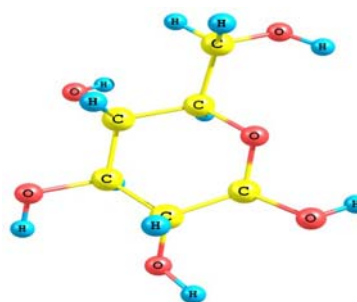
### Modelling the Spectra of cellulose and lignin

Calculations were carried out on a personal computer, performed using semi-empirical quantum mechanics package, MOPAC 2002 which was implemented within the CAChe Program by Fujitsu.<sup>4</sup> Cellulose model molecule was chosen as B-D glucose 1-4 linkage. Lignin structure was guaiacylpropane. The geometry and vibrational spectra of both cellulose and lignin then mixture of them as (1:2), (1:1)

and (2:1) were studied. The initial geometry optimizations of each structure were performed with the molecular mechanics force field to get approximate minimum energy structures. The lowest energy conformations obtained by the molecular mechanics method then were further optimized with the semi-empirical PM5 method.<sup>5</sup>

### Infrared spectrophotometric study of bleached cellulose from high yield soda rice straw pulps

The perhydroxyle anion (HOO<sup>-</sup>) is generally accepted as being the principal active species in peroxide bleaching. This anion is a strong nucleophile which, during bleaching converts electron-rich chromophores typified by  $\alpha$ - $\beta$ -unsaturated aldehydes and ketones, and phenolic ring-conjugated ethylenic or carbonyl groups to their non-chromophoric counterparts. The reactions of lignin with peroxide are not reversible and lead to the permanent removal of most of the chromophoric groups present in the lignin molecule.<sup>6</sup>

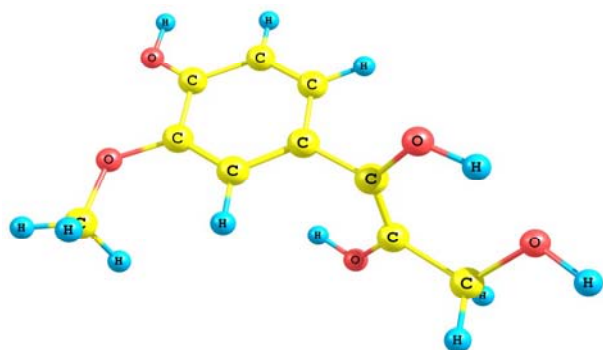


**Figure 1.** Optimized structure of cellulose (glucose unit) which is optimized at PM3 semi-empirical quantum mechanical method.

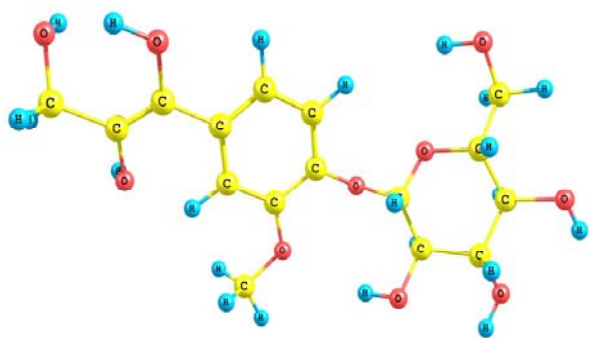
### IR spectral analysis for xanthated cellulose samples

By complete removing of lignin and hemicelluloses dissolvable pulp is formed that is used for manufacturing of cellulose derivatives, such as, cellulose xanthate.

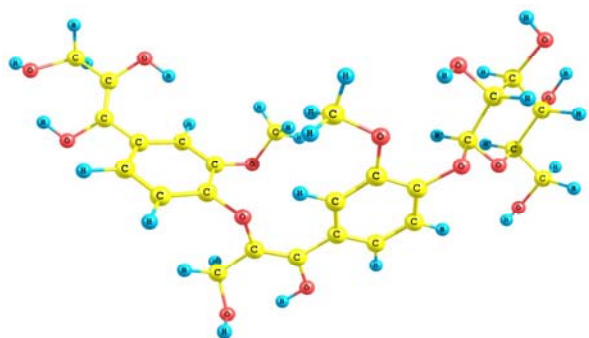
In this work for derivatization of rice straw xanthation reaction was used in which the ONa groups of cellulose were converted to O=C(S)SNa groups respectively. By this conversion some hydrogen bonds between cellulose chains are broken.



**Figure 2.** Optimized structure of lignin which is optimized at PM3 semi-empirical quantum mechanical method.



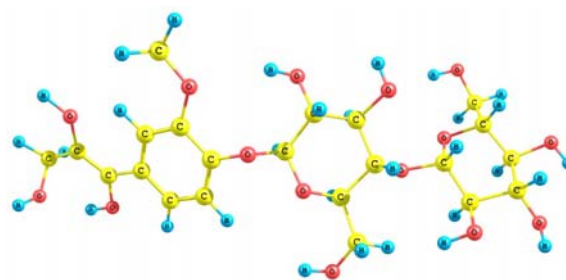
**Figure 3.** Optimized structure of cellulose and lignin (1:1) which is optimized at PM3 semi-empirical quantum mechanical method.



**Figure 4.** Optimized structure of cellulose and lignin (1:2) which is optimized at PM3 semi-empirical quantum mechanical method.

## Results and Discussions

Figure 6 indicating cellulose/lignin ratios and its effect upon calculated IR. Regarding the OH band around 3450  $\text{cm}^{-1}$  increasing the lignin content leads to a broadening in the OH while decreasing lignin content leads to small broadening in the OH followed by a shoulder of OH.



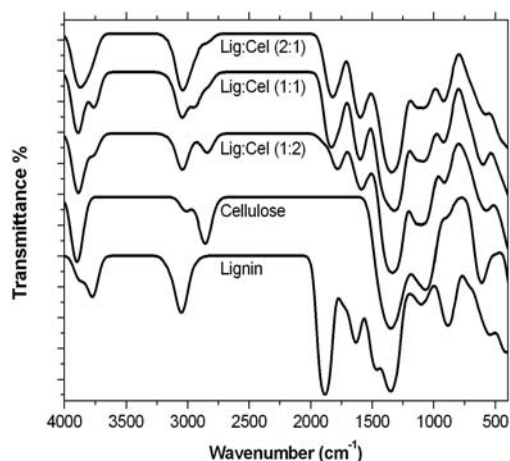
**Figure 5.** Optimized structure of cellulose and lignin (2:1) which is optimized at PM3 semi-empirical quantum mechanical method.

The broad OH is coming as a result of  $\text{CH}_2\text{OH}$  ( $1^{\text{ry}}$  alcoholic OH) while the shoulder is coming from side chain OH's.

Lignin exhibits CH of  $\text{CH}_3$  towards higher wave-number (around 3050  $\text{cm}^{-1}$ ). While cellulose has two respective CH bands toward lower frequencies. Increasing lignin ratio leads to a broad CH band includes  $\text{CH}_3$ ,  $\text{CH}_2$  and CH bands. While splitting of CH bands is coming as a result of increasing the cellulose content.

**Table 1.** Assignments of bands in the IR spectrum of rice straw pulp

$\nu$ , $\text{cm}^{-1}$	Assignment	$\nu$ , $\text{cm}^{-1}$	Assignment
3600	$\nu\text{OH}$ free	1475	$\delta\text{CH}$ in pyran ring
3560	$\nu\text{OH}$ (w) H-bond	1460	$\delta\text{CH}_2$ in pyran ring
3450	$\nu\text{OH}$ (m) H-bond	1450	$\delta\text{CH}_2$ internal deform.
3380	$\nu\text{OH}$ (s) H-bond	1435	$\delta\text{OH}_3$ and R-O-Ar ether
3250	$\nu\text{OH}$ (s) H-bond	1385	$\delta\text{CH}_2$ external deform.
3100	$\nu\text{CH}$ asym. aromatic	1340	$\delta\text{CH}_2$ external deform.
3050	$\nu\text{CH}$ asym. aromatic	1285	$\delta\text{OH}$ phenolic
2980	$\nu\text{CH}$ asym. aliphatic	1270	$\delta\text{CH}_2$ external deform.
2960	$\nu\text{CH}$ asym. aliphatic	1255	$\delta\text{CH}_2$ external deform.
2900	$\nu\text{CH}$ sym. aliphatic	1235	$\delta\text{OH}$ carboxylic
2880	$\nu\text{CH}$ sym. aliphatic	1185	$\nu\text{C-OH}$ phenolic
1750	C=O acetyl linkage	1165	$\delta\text{CH}$ aromatic and C-O-C
1730	C=O $\beta$ -keto structure	1140	$\nu\text{C-OH}$ carboxylic
1675	C=O $\beta$ -keto structure	1115	$\delta\text{CH}$ aromatic
1630	$\delta\text{OH}$ (water)	1065	$\delta\text{CH}$ aromatic
1610	C=C aromatic ring	1040	Lignin band
1595	C=C aromatic ring	1165	C-O cellulose
1530	C=C aromatic ring		



**Figure 6.** PM3 semi-empirical quantum mechanical calculated vibrational spectra for cellulose, lignin and cellulose/ lignin mixtures.

The rest of the spectra below  $2000\text{ cm}^{-1}$  show no variation in the characteristic bands of cellulose as a result of cellulose lignin mixing. From this point we can conclude that the foundation of lignin with cellulose samples could be study from IR spectra as there will be a significant effect upon OH and CH bands.

In order to use the previous study which is a case study for definite conditions of treatment of rice straw (cellulose – lignin), the practically measured IR for all samples under study conditions were subjected and studied.

The infrared absorption spectra of all treated samples were recorded within the range  $4000\text{--}500\text{ cm}^{-1}$  using KBr technique. The extremely broad absorption band within the region  $3400\text{--}2400\text{ cm}^{-1}$  is characteristic to the OH groups. The broadness of this band increases as the extent of hydrogen bonding increases. The absorption of the stretching vibrations of the C-H group is obscured being found within this range. This band can be used to measure the total OH content.

Our study was following the changes occurs on cellulose lignin system (agricultural waste) and cellulose itself through three steps.

### Step one

Based on the above knowledge, we try to model the interaction of Na with cellulose / lignin (2:1) (case study as representations for samples contain high cellulose content). First one Na atom is coordinated instead of hydrogen atom of (C=C) in the main chain of cellulose. Two Na atoms to coordinate instead of (C=C) hydrogen bond in the lignin structure. Other one is coordinated instead H of OH ( $1^{\text{ry}}$  alcoholic) in cellulose.

A third Na atom could coordinate through weak hydrogen bonding into the –O– linkage between cellulose and lignin. Regarding the calculated IR of cellulose / lignin mixture when treated with NaOH, no significant change in the characteristic bands cel:lign mixture as a result of NaOH treatment.

In order to model the true structure of cellulose, a model of 8 glucose units is presented as in Figure 6., some changeable sites with Na atoms was done in model figure 7.

a) Infrared spectrophotometric study of cellulose from high yield soda rice straw pulps

The plant pulp obtained from different plant sources using different cooking conditions is essentially composed of cellulose. The IR spectra of cellulose materials were the subject of many studies.<sup>7</sup>

In this point of our study we would discuss the assignment of the bands in the IR spectra of rice straw and the changes in the infrared spectra which take place during the pulping process.

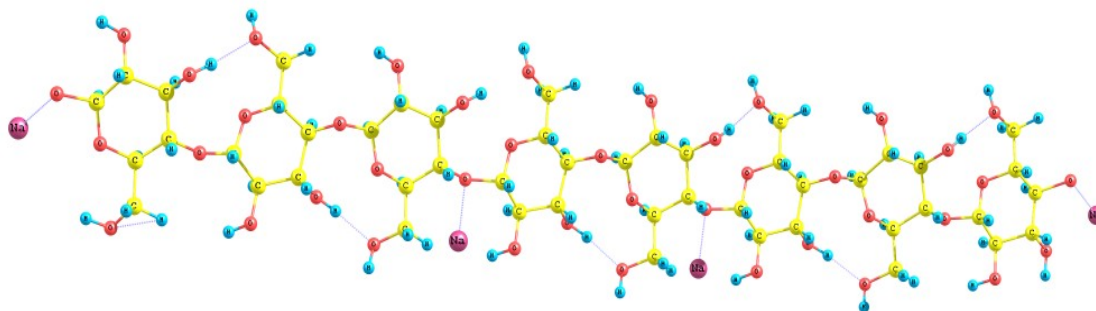
On comparing the spectrum of the raw material with those of high yield soda rice straw pulp (Figs. 3 and 4) the following can be pointed out:

a- The broad  $\delta\text{OH}$  band at  $1600\text{ cm}^{-1}$  tends to be less broad in the spectra of the pulps, meanwhile the band envelopes become more symmetrical. The bands due to the stretching vibrations of the CH-groups tend to be sharper in the spectra of the pulps with the loss of absorption on the higher frequency side where the bands due to aromatic CH-groups appear. The group of bands within the  $1400\text{--}1200\text{ cm}^{-1}$  becomes sharper with rise of either NaOH concentration or cooking temperature. The intensity of most bands decreases in the same direction while the peaks at  $1630\text{--}1370\text{ cm}^{-1}$  tend to be sharp and display a slight increase in peak intensity. The bands within the  $1200\text{--}900\text{ cm}^{-1}$  region tend to be less broad in the spectra of the pulps, yet the most obvious change in this region is the appearance of some sharp peaks near  $1100\text{ cm}^{-1}$ . These changes recall more or less those observed with mercerization of cellulose and can be similarly accounted.<sup>8</sup> The absorption bands within the  $800\text{--}625\text{ cm}^{-1}$  region show some intensity changes on cooking rice straw with NaOH. These spectral changes can be due to dissolution of lignin and as well as the removal of some low molecular forms of carbohydrates from rice straw during the pulping process. This is accompanied by the large decrease in the intensity of the absorption bands due to such components in the various regions; hence the other absorption bands appear sharper. The sharpness of these bands is more obvious with samples cooked for longer times or when the NaOH concentration was increased. Also the rise of cooking temperature brings almost similar effects.

b- The strong band at  $1735\text{ cm}^{-1}$ , assigned to the stretching vibration of the C=O group of the acetyl residue from polyurinoind of the hemicelluloses, displays an obvious decrease in intensity when rice straw is cooked in 6% NaOH for one hour at  $100\text{ }^{\circ}\text{C}$  on increasing the time of cooking to two hours, the band is turned to a very shallow broad one but it vanishes completely when the rice straw is cooked with 8% NaOH for 2 hrs. when boiling. The band appears as a rudimentary peak in the spectrum of samples cooked for one hour at boiling temperature with 8 % NaOH. Also this absorption peak is not observed in the spectra of samples cooked with 10% NaOH. The decrease of C=O band intensity and its vanishing in the spectra of the pulps is due to the hydrolytic rupture of the acetyl linkage between lignin



**Figure 7.** Optimized structure of cellulose (8 glucose units) which is optimized at PM3 semi-empirical quantum mechanical method. COOH is replaced with OH of CH<sub>2</sub>OH.

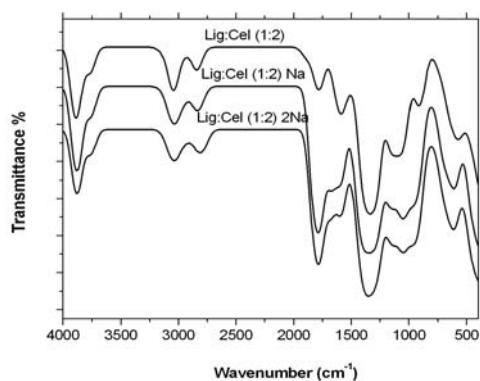


**Figure 8.** Optimized structure of Cel8-4Na units which is optimized at PM3 semi-empirical quantum mechanical method. Two sodium are proposed to interact through hydrogen bonding into the end terminal OH of cellulose. A third Na atom is interacting with O-linkage through hydrogen bond.

and hemicellulose during the pulping of rice straw with NaOH. Thus the removal of the acetyl group from rice straw increases with time of cooking or with rise of NaOH concentration and also by increasing the temperature of the cooking bath as well.

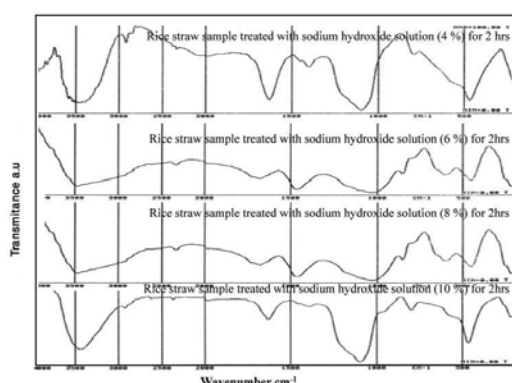
### Step two

We tried to model also cellulose after different conditions of treatment as the following. To model the bleached cellulose with H<sub>2</sub>O<sub>2</sub> (oxidation of some sides mainly alcoholic OH), the model was modified replacing CH<sub>2</sub>OH with COOH in units 1, 5 and 8 respectively. The structure of cel8-COOH is indicated in Figure 8.



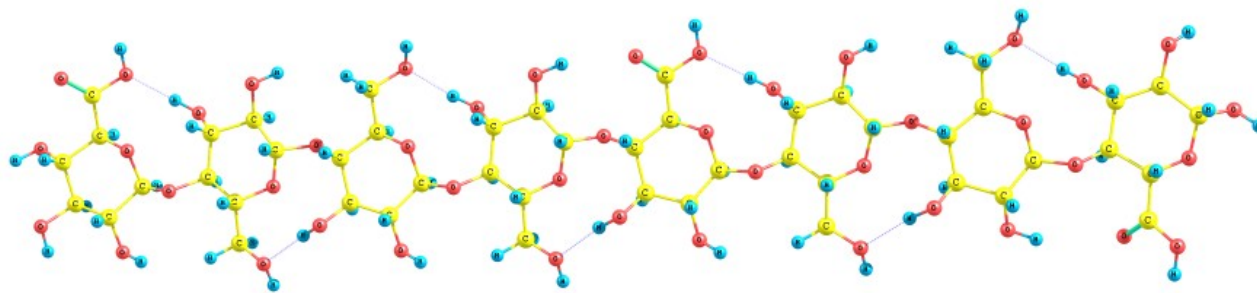
**Figure 9.** PM3 semi-empirical quantum mechanical calculated vibrational spectra for cellulose/ lignin (2:1) as compared with cellulose/ lignin (2:1) in the presence of Na.

c- The bands at 1595-1600 cm<sup>-1</sup>, which correspond to the skeletal vibrations of the aromatic rings of the lignin part, show a gradual decrease on increasing the cooking time or with rise of temperature and or alkali concentration. The drop in the intensity of this band denotes that the lignin content of the pulps decreased during cooking with various degrees depending on the prevailing experimental conditions.

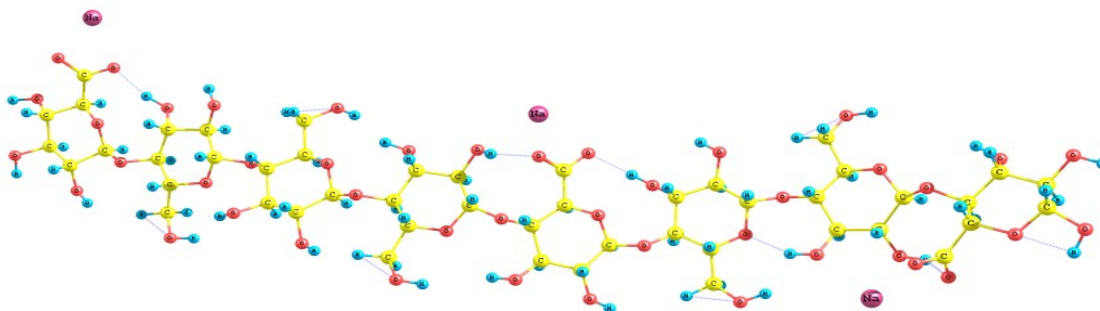


**Figure 10.** IR absorption spectra of soda treated rice straw samples

As the bleaching process was done in alkaline medium with NaOH solution another modification was done introducing another model in which Na is substituting H atom of COOH group. So that Figure 9 presents the sodium salt of the oxidized cellulose as cel8-COONa.

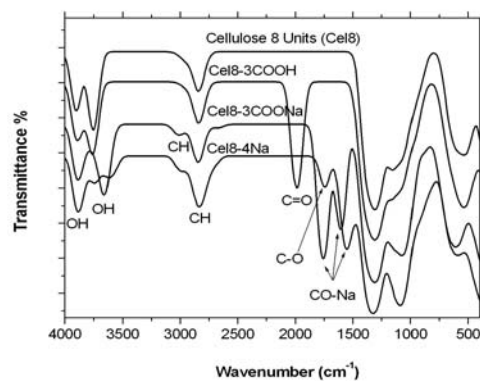


**Figure 11.** Optimized structure of Cel8-3COOH units which is optimized at PM3 semi-empirical quantum mechanical method.



**Figure 12.** Optimized structure of Cel8-3COONa units which is optimized at PM3 semi-empirical quantum mechanical method. Three sodium atoms were substituted with H-atom of the COOH.

Figure 13 represents the calculated IR spectra of cellulose 8 units (cel8) as compared with pri-pulped and bleached cellulose. As far as COOH is replaces CH<sub>2</sub>OH a slight shift in OH band towards lower frequencies are regarded. A significant appears of C=O band around 1800 cm<sup>-1</sup> is attributed to COOH. Regarding cel8-COONa the shift on OH band remains toward lower frequencies with a splitting in CH band.

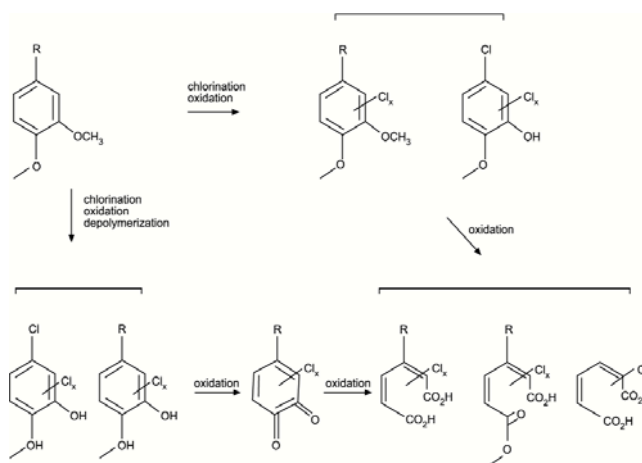


**Figure 13.** PM3 semi-empirical quantum mechanical calculated vibrational spectra for cellulose 8 units (Cel8) as compared with Cel-3COOH, Cel8-3COO-Na and Cel-4Na.

This splitting leads to the appearance of a new CH band around 2980 cm<sup>-1</sup> while the principle CH band remain unchanged around 2820 cm<sup>-1</sup>. As Na is coordinated with COOH to form cel8-COONa. The shift of OH band toward lower frequency is significant. While C=O is changed into metal carboxylate around 1750 cm<sup>-1</sup>. This band is asymmetric metal carboxylate. Another one which is the symmetric metal carboxylate is found around 1610 cm<sup>-1</sup>.

Finally as 4 Na atoms introduced into cel8 units a splitting in the side chain OH took place with a rise to a band of C=O around 1740 cm<sup>-1</sup>.

The infrared spectrum is characteristic for the entire molecule. It is known that certain groups of atoms give rise to bands at or near the same frequency regardless of the structure of the rest of the molecule. It is the persistence of these characteristic bands that permit us to obtain useful structural information by simple analysis of the spectrum and reference to generalized charts of the characteristic group's frequency.



**Figure 14** Oxidation of residual lignin during bleaching sequences.

As was mentioned before in the experimental part, there are many sequences for producing white pulp for several uses. The aim for which pulp is needed determines the stages and the kind of treatment which would be preferred. At this point of our study the pulp obtained from rice straw was treated with sodium bisulphate (4 % and 8 %) solution concentrations and samples from these stages were then

treated with hydrogen peroxide (2 %, 4 % and 8 %) solution concentrations, samples obtained from the treatment mentioned above were subjected to IR measurements and the results are collected in Fig. 14.

From investigation of IR spectrum of the bleached samples, it is clear that the mean value of absorbance is higher than that for un-bleached pulp. The characteristic absorbance in the wave number region of free OH and bonded OH stretching group namely 3300-3500  $\text{cm}^{-1}$  reveals that the OH groups are not free, since the bands are very broad. Comparing between the mean value of the absorbance of second and primary OH groups of soda only, or both of soda and bisulphate, or soda and bisulphate and  $\text{H}_2\text{O}_2$  the lignin peaks have lower values due to the oxidation effect of  $\text{H}_2\text{O}_2$  on the pulp during the bleaching process causing an increase in degradation of lignin.<sup>9</sup>

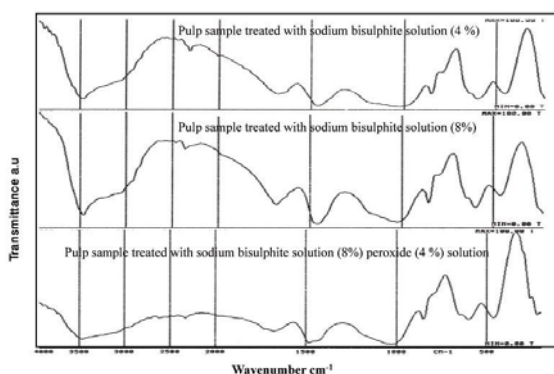


Figure 15. IR absorption spectra of bleached pulp samples

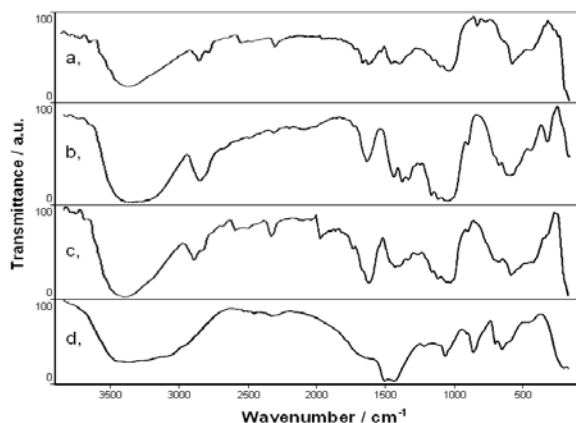


Figure 16. IR absorption spectra of xanthated cellulose samples

The 1800-1500  $\text{cm}^{-1}$  region includes several broad bands which are not well resolved, the bands at 1750 - 1670  $\text{cm}^{-1}$  correspond to the stretching vibrations of the C=O groups of various types namely those of the acetyl linkage (1750  $\text{cm}^{-1}$ ), and those at (1730 and 1670  $\text{cm}^{-1}$ ) are due to  $\beta$ -keto structure. The shoulder near 1630  $\text{cm}^{-1}$  is due to in-plane deformation of the water molecules, while the bands at 1610, 1590 and 1520  $\text{cm}^{-1}$  are due to skeletal vibrations of the aromatic ring of lignin.

The band at 1470  $\text{cm}^{-1}$  is assigned to C-H bending of the pyran ring, while that at 1430  $\text{cm}^{-1}$  is considered to be the inplane bending of the  $\text{CH}_3\text{O}$ - groups. The inplane bending of the  $\text{CH}_3$ -groups leads to the broad band at 1350  $\text{cm}^{-1}$ . The two peaks at 1280 and 1240  $\text{cm}^{-1}$  are assigned to the deformation vibrations of the  $-\text{COOH}$  and phenolic OH-groups respectively. The other types of vibrations involving the OH groups lead to the absorptions bands at 1180 and 1130  $\text{cm}^{-1}$ . However, it was stated that the first band also includes the stretching mode of the aliphatic-aryl-ether linkage. The broad bands within the 1300-1000  $\text{cm}^{-1}$  region would also involve some types of bending vibrations of the aromatic C-H and those of the aliphatic groups.<sup>10</sup>

The characteristic absorption bands of phenyl ring of the benzyl group appear at 1700-2000  $\text{cm}^{-1}$  region. The C=O to the C=S occurs due to xanthation in addition to that the C=O band itself which is more sharp and boarder for sample (b), this may be due to bleaching with  $\text{H}_2\text{O}_2$  as had been stated above broke some hydrogen bonds and freed more positions to be xanthated.

The IR spectral bands of cellulosic material are relatively diffuse because of the high molecular weight. So, to follow the change of the molecular structure of the cellulose, the use relative absorbance (ratio of any band intensity / band intensity at 1325  $\text{cm}^{-1}$ ) is more correct than the use of changes that takes place for characteristic band. Also, infrared spectra can give us an idea on the crystallinity of cellulose by calculating the ratio of band intensity at 1425  $\text{cm}^{-1}$  which is characteristic of CH vibration of crystallinity region to band intensity characteristic of CH vibration of amorphous region at 900  $\text{cm}^{-1}$ . Thus, crystallinity index is an IR parameter that gives information about the ratio of crystalline region to amorphous region. The parameter was measured according to the absorbance ratio  $A_{1430} / A_{900}$ .<sup>11</sup>

Besides the measuring of the crystallinity index, the mercerization depth which occurs by swelling of cellulose by sodium hydroxide can also be determined. The mercerization depth gives us a good idea about swelling of cellulose and its decrystallization. It can be measured by using the ratio of band intensity at 1371  $\text{cm}^{-1}$  to band intensity at 1325  $\text{cm}^{-1}$ . Cellulose was mercerized in aqueous solution. Results of cellulose mercerizing were determined by  $A_{1429}/A_{893}$  and mercerization depth from  $A_{1375}/A_{1325}$ .<sup>12</sup> Due to the treatment of cellulose pulp with sodium hydroxide and heating, some degradation and oxidation of OH group can occur. The relative absorbance of OH group at 3425  $\text{cm}^{-1}$  decreased due to the oxidation to COOH. So, a new band appeared at 1715  $\text{cm}^{-1}$  which characteristic of C=O of carbonyl group. On the other hand, the relative absorbance of C-O-C band at 1114  $\text{cm}^{-1}$  which is characteristic of the (1-4)  $\beta$ -O-glucosidic linkage decreased due to the degradation of cellulose chains which occurred by sodium hydroxide solution and heating.

On mercerization many processes take place, e.g. swelling with some exothermic effect and dissolution of hemicellulose as non-forming components. Because degradation of cellulose occurs to a slight extent, an addition compound of cellulose and sodium hydroxide is formed and intermolecular hydrogen bonds become loose. This phenomenon brings about structural changes in cellulose which depends on sodium hydroxide concentration. The

extent of mercerization is affected by some inorganic and organic compounds such as zincate, aluminate or urea.

**Table 2.** General scheme of the samples under study

Sample No.	SHC %	Time, h	Characterization
1	4	1	Rice straw sample treated with NaOH solution (4 %) for 1 h.
2	6	1	Rice straw sample treated with NaOH solution (6 %) for 1 hr.
3	8	1	Rice straw sample treated with NaOH solution (8 %) for 1 hr.
4	10	1	Rice straw sample treated with NaOH solution (10 %) for 1 hr.
5	4	2	Rice straw sample treated with NaOH solution (4 %) for 2hrs.
6	6	2	Rice straw sample treated with NaOH solution (6 %) for 2 hrs.
7	8	2	Rice straw sample treated with NaOH solution (8 %) for 2 hrs.
8	10	2	Rice straw sample treated with NaOH solution (10 %) for 2 hrs.
9			pulp sample treated with sodium bisulphate solution (4 %).
10			pulp sample treated with sodium bisulphate solution (8 %).
11			pulp from rice straw sample treated with sodium bisulphate solution (4 %), and hydrogen peroxide (4 %) solution.
12			RSA remained after burning rice straw at 600 °C for 2 hrs.
13			Rice straw ash remained after burning rice straw at 800 °C for 2 hrs.
14			The content of black liquor (lignin and sodium silicate) obtained by complete drying.
15			Black liquor content treated with acetic acid.
16			Black liquor content treated with hydrochloric acid.
17			Black liquor content treated with benzene.
18			Black liquor content treated with carbon tetrachloride.
19			Rice straw ash sample remained after complete burning.
20			Black liquor treated with calcium hydroxide solution.
21			Black liquor treated with calcium carbonate suspension.
22			Pulp treated with CS <sub>2</sub> solution.
23			Bleached pulp with sodium bisulphate treated with carbon disulfide solution.
24			Bleached pulp with hydrogen peroxide treated with carbon disulfide solution.

change of molecular structure can be followed by infrared spectroscopy. The main parameters affected by treatment of cellulose with sodium hydroxide are crystallinity index, swelling of cellulose chain (related to the measurement of mercerization depth), intensities of OH groups, C=O band (which was related to COOH at 1715 cm<sup>-1</sup>) and CHO at 1654 cm<sup>-1</sup>, and the intensity of ether linkage which bonds the glucose molecule (1-4) β-O-glucosidic links.<sup>13,14</sup>

## Conclusion

Spectral trace of the changes that have occurred in the rice straw as a result of the chemical treatments, as well as the use of computer programs has shown that treatments caused a break in the link between the lignin and cellulose units at the same time freed terminals in cellulose units, making it the most active chemically. Theory and simulation process, which relied on the prediction what, happened during the treatment as well as the cellulose lignin ratio.

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