A comparison of methods for quantification of eucalypt pulp xylan content

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Abstract

Hemicelluloses, a major constituent of wood, are present between the cellulose fibrils in cell walls. In connection with the production of pulp, hemicelluloses play an important role, because its preservation as well as being desirable in paper manufacturing, increases yield in pulp production. To compare methodologies that are suitable for quantification of hemicellulose in the brown pulp of eucalyptus, tested three different methods. The aim of this study was to evaluate the correlation between these methods. The most used techniques to measure hemicellulose in the pulp of eucalyptus have been indirectly through the pentosan method or directly through xylan analyses by GC or HPLC techniques. But it is known that pulp hemicelluloses can be extracted with NaOH at various concentrations, and quantified by colorimetric and gravimetric techniques. In this study a comparison was made between the more traditional methods (pentosan and xylan analyses by HPLC) and the indirect method of NaOH 5% and NaOH 18% extraction followed by colorimetric analyses of the extract for five samples of eucalyptus brown pulp. It was observed that the content of NaOH 5% extract correlates very well with the levels of xylans by HPLC (R^2 = 0,98) and reasonably well with the pentosan content (R^2 = 0,93). The levels of soluble materials in NaOH 18% are positively correlated with the levels of xylans by HPLC (R^2 = 0,98) and pentosans (R^2 = 0,99). Therefore, the method of solubility in NaOH 5% and 18% are quite reliable and can be used to replace the other two since it is faster, simpler and les costly to carry out than the others.

Keywords: eucalyptus; hemicelluloses; indirect method; HPLC.

Introduction

Hemicelluloses have been a component of wood that has aroused great interest worldwide, is usually associated with other cell wall components, such as cellulose, proteins, cell wall, lignin and other phenolic compounds by covalent and hydrogen bonds both by ionic and hydrophobic interactions (Mine & Suckow, 1986; Rybka, SITARSKI & Raczynski, Bojanowski, 1993).

In the Kraft process, in addition to a large fraction of lignin, the hemicelluloses are also lost to the flow of black liquor during the cooking process (Bhaskaran AND VON Koeppen, 1970).

The hemicellulose can be quantified indirectly by measuring the solubility of the pulp in sodium hydroxide solutions at certain concentrations (BROWNING, 1967). In the case of short-fiber pulp, the material removed by 18% NaOH solution is practically the xylans. The methods used for measuring hemicellulose in hardwood pulps has been the pentosans and of xylans by liquid chromatography and / or gas. However, these techniques require time and cost. It should be emphasized that the value does not match the pentosan content of hemicellulose in lignocellulosic material. But in the case of eucalyptus, the pentosans content is very close to the hemicellulose content, since in these eucalyptus wood are mainly composed of pentoses. Thus, the method of pentosans approaches in much of the method by ion chromatography, providing very similar results.

The aim of this study was to compare the direct methods (pentosans xylan and analysis by HPLC) and methods indirect extraction by NaOH 5% and 18% NaOH followed by colorimetric analysis of the extract for five samples
eucalyptus pulp brown.

Experimental

Were used for this analysis five samples of industrial brown kraft pulps of eucalyptus. The pulp used was subjected to alkali treatment by varying the alkali charge of 0, 10, 30, 50 and 70g / L, with the zero reference.

For direct determination of the content of pulp xylan was hydrolyzed with H_2SO_4 (72%), led to an autoclave for one hour, and the filtrate was analyzed using a Dionex ion chromatography with pulse amperometric detector, Model 3000 ICS.

To obtain the content of xylan in the pulp were used, too, the indirect method of extraction in NaOH 5% and 18% followed by colorimetrie analysis of the extract, and pentosans.

For the solubility S5% and S18% weighed approximately 1.5 g of pulp, which were transferred to a 250mL Erlenmeyer flask. It is added through a beaker, 100 mL of solution of sodium hydroxide, keeping the set at 25 ° ± 0.2 ° C in the water bath for 60 minutes. After exactly 60 minutes, filter the mixture. Then the pipette was 20mL and 10mL of filtered solution of 0.5 N potassium dichromate and transferred to an Erlenmeyer flask. Carefully add 45mL H_2SO_4. Add to ferroin and titrate with ferrous sulphate solution 0.1 N until the appearance of purplish stable for a few seconds.

The methodology describes the determination of pentosans in cellulose will be followed by the method ABCP C8/70.

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Results and Discussion

The percentages of xylans,solubility in NaOH 5% and 18% and pentosans can be seen in table 1. Since the relationship between the percentages of pentosans and xylan are shown in figure 1.

Table 1.Percentages of pentosans and xylans

Samples	Xylans (%)	Pentosans (%)	$S_{5\%}$ (%)	$S_{18\%}$ (%)
Reference	15,6	14,9	9,81	6,76
10g/L	14,5	14,7	9,36	6,45
30g/L	10,8	12,0	5,87	4,85
50g/L	8,1	8,0	2,32	2,66
70g/L	5,9	4,7	1,68	1,27

The percentages of pentosanas and xylan showed good correlation (R^2 = 0.96). A level of 10% of xylan is equivalent to 9.8% pentosans. Therefore, these two techniques can be used interchangeably to measure the content of hemicelluloses brownstock eucalyptus, since the presence of other hemicelluloses in these pulps is negligible.

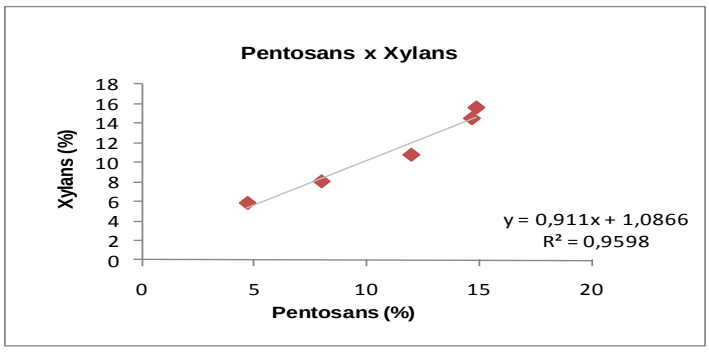

Figura 1. Correlação entre as percentagens de pentosanas e xilanas.

The correlation (R^2 = 0.98) between the solubility of the pulp in sodium hydroxide and 5% and xylan content was very high.). It was determined that that a NaOH 5% solubility value of 10% equivalent to 15,56% xylan.

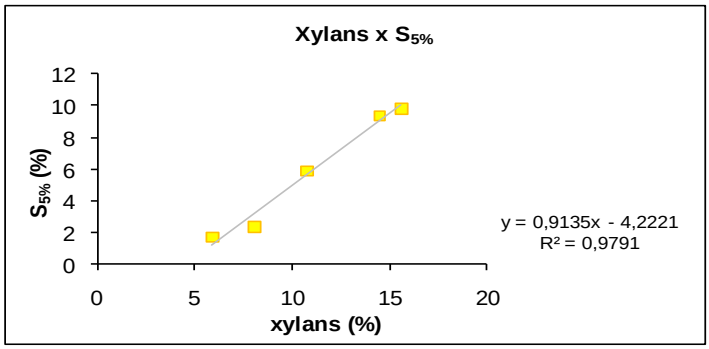

Figure 2. Correlation between soluble material in 5% NaOH and xylans.

You can see, in Figure 3, there was a good correlation between solubility and soda 5% and pentosanas (R2 = 0,93). It was determined that that aNaOH 5% solubility value of 10% equivalent to 15.9% pentosans.

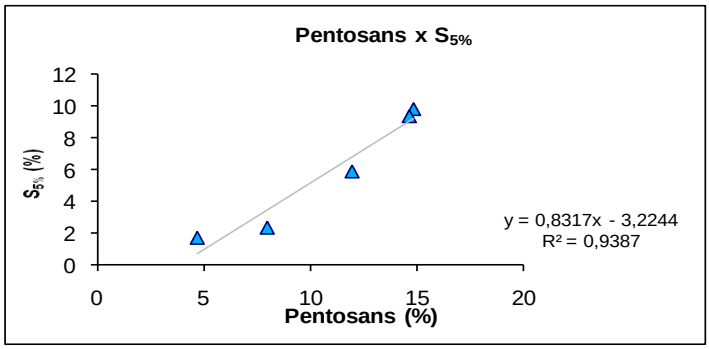

Figure 3. Correlation between soluble material in 5% NaOH and pentosans.

Similarly, Figure 4, there was a good correlation between solubility in sodium hydroxide and 18% pentosans (R^2 = 0,99) . It was determined that that a NaOH 18% solubility value of 10% is equivalent to 21,3 pentosans.

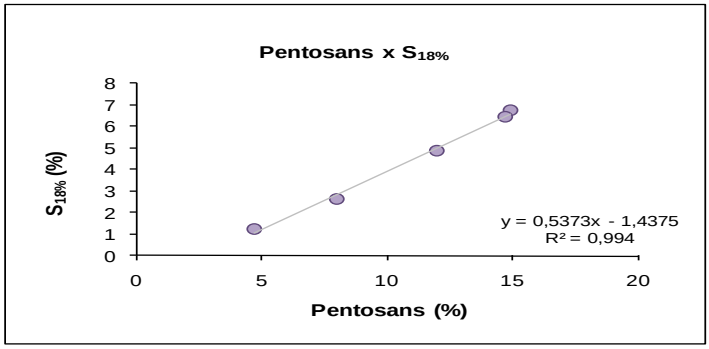

Figure 4. Correlation between soluble material in 18% NaOH and pentosans.

The correlation (R^2 = 0.98) between the solubility of the pulp in sodium hydroxide and 18% and xylan content was very high (Fig. 5). It was determined that that a NaOH 18% solubility value of 10% equivalent to 20,5% xylan.

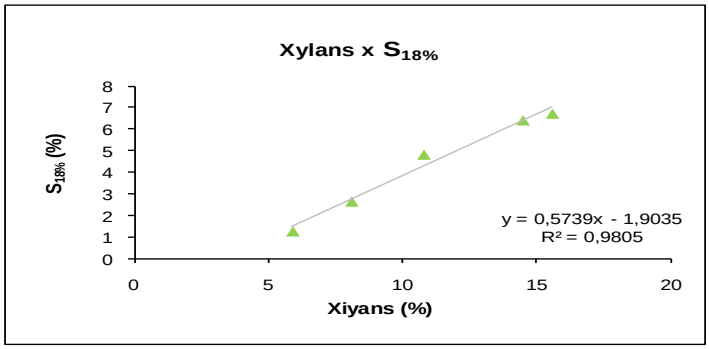

Figure 5. Correlation between soluble material in 18% NaOH and xylans.

Conclusions

The method of solubility of pulp in a solution of sodium hydroxide 5% and 18% was adequate for quantification of hemicellulose brown eucalyptus pulp. Being a simple, fast and inexpensive, it presents a good alternative to traditional methods of pentosans anddirect analysis of xylans by HPLC.

References

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