



Production of pulp from *Salix viminalis* energy crops using the FIRSST process

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ABSTRACT

In this work, isolation of the cellulose fibres was carried out via the Feedstock Impregnation Rapid and Sequential Steam Treatment process (FIRSST). The latter allows the separation of extractives, hemicellulosic sugars and lignin isolating the cellulose fibres. Quantitative data on the constitutive macromolecules of biomass was obtained using ASTM or TAPPI standard methods. Carbohydrates found in the hemicelluloses were also quantified using HPLC. Kraft pulp from whole biomass has also been produced at a bench scale (few kg per batch) using known and established pulping conditions. The pulps from both pulping techniques were tested following ATPC standard methods. Pulp yields were of 34% for the classical Kraft processes (using whole biomass) while the FIRSST process showed yields around 30%. The average fibre lengths were similar for FIRSST pulp (0.39 mm) and Kraft pulp (0.41 mm) and the mechanical properties of the FIRSST pulp were as good as those of the Kraft pulp.

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1. Introduction

Residual biomass (woody or herbaceous) could be a suitable pathway for the production of second generation biofuels and so are the 'energy crops', grown in marginal lands, which could also be considered as a potential lignocellulosic feedstock for biofuels while contributing to CO₂ fixation. In Canada, barked willow (*Salix viminalis*) has shown promising growth potential (Labrecque and Teodorescu, 1999) and our group has shown that this species could be used for the production, from whole biomass, of Kraft pulp even with specimen as young as 3 years old which were composed of wood, bark and foliage (unpublished results).

The isolation of the constitutive fractions of lignocellulosic biomass has been and continues being a subject of scientific, engineering and economic interest. To this purpose, the techniques that are being developed and/or used actually comprise organosolv, aqueous/steam technological platforms and even combinations of both. The organosolv technique is particularly interesting if the production of a high grade lignin is targeted as a co-product and recent studies that used this technique have focused on poplar (Pan et al., 2007; Kirci et al., 2003), on wheat straw (Sun and Chen, 2008) and on bagasse (Tu et al., 2008). Another alternative is the aqueous/steam treatments which requires biomass impregnation with water (specific catalysts can be added) to reach fibre saturation, cooking under pressure at temperature around 200 °C via

addition of steam. With a sudden decompression to atmospheric pressure through a well designed nozzle the treated biomass exits the treatment vessel, and part of the water flashes out as steam forcing destructuring and defibration of the lignocellulosic tissues. During the treatment, acetyl groups are converted to acetic acid which serves as a source of protons along the process. This technique, to work effectively, requires a careful management of the water in contact with the biomass material. In literature, it has been efficiently applied to bagasse (Martin et al., 2008), *Miscanthus* (Sorensen et al., 2008), Aspen (Li et al., 2007) and *Salix* (Sassner et al., 2008) with the target being the production of carbohydrates for ethanol.

The present study centers on the comparison between two types of pulp generated from *S. viminalis*. The biomass used for this experiment was not sieved nor debarked to ensure that the conditions were representative of a potential industrial process that will use whole biomass as input. The same 3 years old *S. viminalis* shrubs were used for the production of both Kraft and FIRSST pulps. Such a comparison will allow determining whether the isolation of the constitutive fractions of the biomass is possible without appreciable fibre damage.

2. Methods

2.1. Sampling

Three year-old samples of *S. viminalis* were obtained from Agro Énergie in St.-Roch de l'Achigan (Que., Canada). Same species have been reported to produce close to 45.28 t/(ha year) of biomass

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(Labrecque and Teodorescu, 1999). The samples were harvested and shipped fresh to the Chair's laboratory in Sherbrooke (Québec). They were stored in a freezer at -15°C until utilization. Since the overall potential of the plant was considered during this experiment, the different tissues from the woody plant were not separated one from another and the basket willow was evaluated as a whole.

Table 1

Procedures used for the quantification of the macromolecular compounds from *Salix viminalis*.

Procedure	Corresponding protocol	Complementary informations
Extract content	ASTM D1107-56	–
Ash content	TAPPI T-15	–
Holocellulose	TAPPI T-9	ASTM D1104
Hemicelluloses	TAPPI T-212	H ₂ SO ₄ replaced CH ₃ COOH for acidification also see HPLC protocol
α -Cellulose	ASTM D1103-60	TAPPI T203 05-1974
Lignin	ASTM D1106-56	–

2.2. Chemical analysis of the biomass

The protocols used for the chemical analysis of the *S. viminalis* biomass are summarized in Table 1 below.

2.3. HPLC protocol for carbohydrate analysis

Quantification of the sugars was made using a Dionex HPLC system model DX-500 equipped with an AS50 autosampler, a GP50 pump and a ED40 electrochemical detector. Column used to this purpose was a Dionex PA-10 (4×250 mm) with a guard column model Dionex PA-10 (4×50 mm). Detection was made with a pulsed amperometry detector (PAD). Elution was made with a 4 mM of an aqueous mixture of sodium hydroxide with a 1.0 mL per minute flow. Identification of carbohydrates was made by comparison with pure compounds: arabinose, glucose, galactose, xylose and mannose all bought from Sigma. Quantification was made using fucose (also bought from Sigma) as an internal standard.

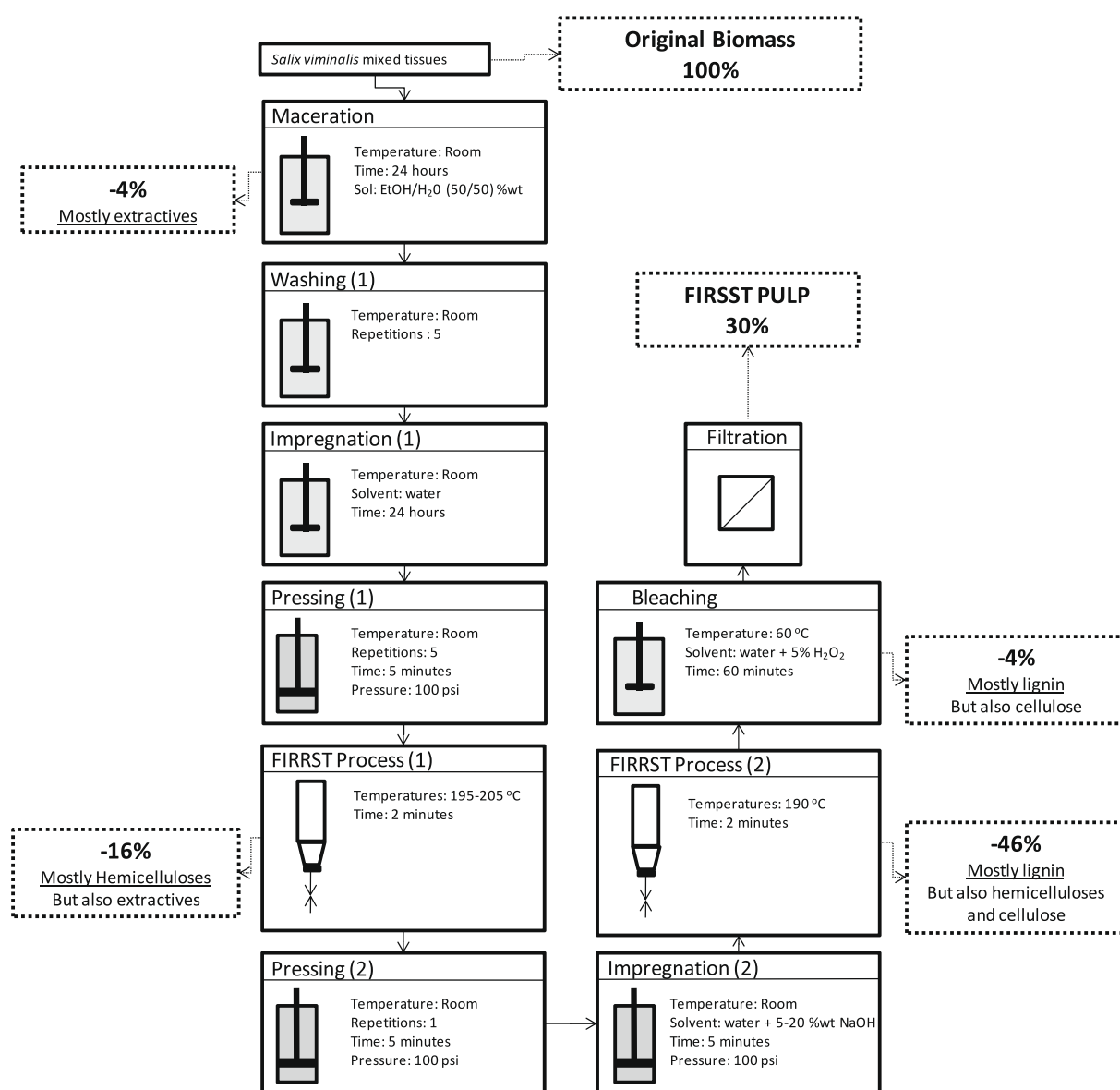


Fig. 1. Flow diagram of the FIRRSST process and operational conditions used in each stages.

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