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Isolation and purification of concentrated and non-concentrated hemicellulose alkaline extracts

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ABSTRACT

The xylan rich filtrate obtained from alkaline extraction of bleached pulp was isolated by precipitation followed by washing and drying of the precipitate. This isolation/purification process was applied to a filtrate as is (non-concentrated) and to a filtrate previously concentrated by ultrafiltration (UF). The advantages achieved by implementing the UF concentration process are significant when compared with results obtained with the non-concentrated filtrate.

The membrane used in the concentration step, UFX5pHt, (MWCO 5 kDa), has an average flux of 30 L/h/m^2 for previously optimized conditions of cross flow velocity (1 m s^{-1}) and operating pressure (6 bar). Xylans were isolated from the concentrated filtrate using two different methods: neutralization/precipitation with HNO_3 (7.5 N), and precipitation with methanol (20, 30, and 50 wt/wt%). For the concentrated filtrate a reduction of up to 61% in the consumption of nitric acid for the precipitation was achieved. On the other hand, products of lower purity were obtained with methanol as precipitating agent. The molecular weights of the obtained xylans were determined by SEC leading to values between 24,100 and 31,000 Da.

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1. Introduction

In the last decades the research effort to optimize processes aiming at the isolation of hemicelluloses from various sources has increased substantially. The main objective has been the production of chemicals and fuels from sustainable resources which could, in the near future, be an alternative to those of fossil origins.

Among other sources, hemicelluloses can be obtained from the wood pulp. This approach serves three main purposes, namely: the production of xylan for commercial terms; the possibility to reuse the resultant hemicellulose poor wood pulp as raw material for other products; and, finally, as an alternative to face a hypothetical excess of wood pulp production in the future.

The production of xylans (a hemicellulose largely found in nature) from the wood pulp is, nowadays, a subject which arouses much interest. This is mainly due to their enormous potential as raw material for the production of high value-added products. The range of xylans application is enormous. It can be used to produce important chemicals such as, among many others, furfural, xylose and xylitol [1]. On the other hand, the use of xylan in packaging films, food coating, papermaking, and as an emulsifier and protein foam stabilizer are examples of applications which are under intense research evaluation [2–4].

The xylans in bleached kraft pulp can be recovered by the so called cold caustic extraction, a simple alkaline extraction using sodium hydroxide (NaOH) followed by filtration, from which an alkaline filtrate (in what follows designated by FTQ) rich in xylans is obtained [5–9]. From this filtrate, xylans are isolated and purified by antisolvent precipitation followed by the washing of the precipitate. Precipitation is achieved in most of the cases by acidic neutralization of the FTQ or by alcohol addition. The most used acids are acetic, formic, sulfuric and hydrochloric [10–15]. As to alcohols either for precipitation or for the washing of the precipitates, the most referred in literature are methanol and ethanol [11,12,16–19]. Water has been used in the washing step as well [7,16]. This whole isolation procedure can be performed in two different ways namely, acting directly upon FTQ or acting upon an FTQ previously concentrated by ultrafiltration (UF). Despite the fact that there are several studies about the application of membrane processes in the concentration of the alkaline filtrate [20–23] in none of them is reported, in detail, what happens next to the concentrate of xylans, regarding its posterior isolation and purification. Most of the papers dealing with this subject are mostly dedicated to the opti-

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mization of the UF operation, namely membrane and operating conditions selection.

It is expected that this UF concentration of FTQ could be an attractive alternative resulting in the reduction of the anti-solvent quantity used in the isolation step. Aiming the obtainment of higher purities for the final product diafiltration (DF) can also be performed in association with ultrafiltration. DF has also been used as a mean of decreasing pH of filtrate and/or preventing alkaline extract degradation during long term storage [23].

UF operation could also provide the recycling of a substantial amount of NaOH in the permeate to the extraction process. Conditions for this recycling are discussed is some papers [23,24].

In this context the main goal of this work is the evaluation of the advantages achieved by implementing a FTQ concentration process prior to isolation and purification of xylans. The advantages, if any, are evaluated by comparison with results obtained by exactly the same isolation and purification procedures applied to a non-concentrated FTQ and taken as reference data (RD).

2. Materials and methods

2.1. Raw material

The bleached kraft pulp from *Eucalyptus globulus* used in this experiment was supplied by RAIZ (Paper and Forest Research Institute, Aveiro, Portugal). Its characteristics based on pentosans and humidity content are shown in Table 1. Pentosans is an indirect method for xylans quantification (Tappi T223 cm-84 (1984)) based on its xylose content.

2.2. Equipment

2.2.1. Membrane

The UF concentration tests were performed with a UFX5pHt flat sheet polysulphone membrane (Alfa Laval, Denmark, Nakskov) having a MWCO of 5 kDa.

2.2.2. Membrane filtration setup

One membrane pair with an effective membrane area of 0036 m^2 was mounted in a plate-and-frame LabStak M20 unit (Alfa Laval, Denmark, Nakskov).

2.3. Experimental procedure

2.3.1. Membrane preparation and washing

Preceding first use, the membrane was cleaned in order to remove chemical preservatives. The membrane was then first submitted to a washing procedure with deionized water (30 min at 2 bar pressure and at 25 °C) followed by an alkaline cleaning with Ultrasil 10 (0.5 wt.% for 30 min at 25 °C and 2 bar). The alkaline detergent was removed from the system by washing it with deionized water until neutral pH.

After compaction (2 h at 20 bar) pure water flux (PWF) of the membrane was measured. For its hydraulic permeability, L_P, a value of $L_P = 5 \times 10^{-11}$ m s^{-1} Pa^{-1}, at 25 °C, was found.

The cross-flow velocity was kept at 1 m s^{-1} in every experiment.

Table 1
Characteristics of the kraft pulp.

Sample	Pulp 1	Pulp 2
Pentosans (%)[a]	16.4	17.2
Humidity (%)	70.6	69.1

[a] Calculated according to Tappi T223 cm-84 (1984).

After each experiment membrane was rinsed with DI water until neutral pH was achieved. After this washing, the PWF was measured always at the same conditions (6 bar, v = 1 m s^{-1}, T = 25 °C). The PWF was always recovered, its value being 108 ± 5% (L/h/m^2).

2.3.2. Alkaline extraction of hemicelluloses from pulp

Xylans were recovered from the *Kraft* pulp with NaOH according to the procedure previously optimized by RAIZ. NaOH content was set to 10% (w/w) in the suspension (2 g NaOH/g dry pulp). Temperature and consistency (defined as g pulp/g total solution), were 25 °C and 5% respectively. Extraction time was 1 h under an agitation of 1100 rpm.

Prior to extraction a certain amount of pulp is weighed. For this pulp weight, its humidity and consistency, the amount of total suspension was calculated. Depending on the weight of the suspension the adequate quantity of sodium hydroxide (10%) was added to it. Finally, in order to obtain the required amount of suspension, a given quantity of water was weighed and added. After 1 h, the suspension was filtered (G-3 filter 15–40 μm), and the alkaline extract (alkaline liquor) separated from the extracted pulp.

2.3.3. Concentration and diafiltration tests

The concentration experiments (C), aiming at the filtrate concentration, was carried out in batch mode under the conditions optimized by parametric studies. The transmembrane pressure was kept at a constant pressure of 6 bar and a cross-flow velocity of 1 m s^{-1} was applied in all tests. The volumetric concentration factor (VRF), which is defined as the ratio between the initial and the final volume at the membrane feed tank, varied between 3.6 and 4.6. For the concentration tests with diafiltration (C + DF), after a first concentration, a given mass (equal to the mass of collected permeate during the concentration) of water was added. After this water addition a second concentration was performed until the same VRF was reached.

2.3.4. Isolation of xylans in the UF concentrate

The xylans present in the alkaline UF concentrate were isolated by two independent methods:

- Precipitation by HNO_3 (7.5 N) until pH = 7 under a constant agitation of 300 rpm.
- Precipitation with 20, 30 and 50% methanol under a constant agitation of 300 rpm.

After addition of the anti-solvents, the resulting suspensions were centrifuged (B. Braun Sigma 4K10) at 8000 rpm for 20 min. The precipitates, mostly xylans, salts or NaOH and humidity, were further purified while the supernatants were rejected.

2.3.5. Purification of xylan

In order to reduce/remove the inorganic matter of the precipitates, they were washed either with water or methanol.

The washing/purification procedure using water, is differently controlled depending on the previous precipitation step. In the cases where precipitation was carried out with methanol, the addition of water was pursued until a pH of 7, for the resultant solution (water plus precipitate), is attained. When the precipitation was made by neutralization with HNO_3 the amount of water to be used in the washing operation was controlled by the conductivity (until ≈ 5 μS/cm) of the resultant solution.

Methanol washings were carried out using fixed volumes of the alcohol in 3 steps. At each step a centrifugation and separation of the supernatant was done.

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