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Malek Khadraoui, Ramzi Khiari, Nicolas Brosse, Latifa Bergaoui, Evelyne Mauret. Combination of steam explosion and TEMPO-mediated oxidation as pretreatments to produce nanofibril of cellulose from *Posidonia oceanica* bleached fibres. *Bioresources*, 2022, 17 (2), pp.2933-2958. 10.15376/biores.17.2.2933-2958 . hal-03691626

HAL Id: hal-03691626

<https://hal.univ-lorraine.fr/hal-03691626>

Submitted on 9 Jun 2022

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Combination of Steam Explosion and TEMPO-mediated Oxidation as Pretreatments to Produce Nanofibril of Cellulose from *Posidonia oceanica* Bleached Fibres

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Posidonia oceanica is the dominant sea grass in the Mediterranean Sea. This biomass has great potential for use as a novel lignocellulosic material on an industrial scale. In this work, an innovative approach was applied to produce cellulose nanofibril (CNF) from *Posidonia*. First, fibres were isolated by a delignification-bleaching process, followed by refining and TEMPO-mediated oxidation to facilitate their further microfibrillation. Cellulose nanofibril suspensions were then produced by steam explosion or grinding (Masuko supermasscolloider). Next, CNF gel-like suspensions were characterized by several techniques such as morphological analysis (Morfi, optical microscopy, atomic force microscopy, transmission electron microscopy) and turbidity measurements. Nanopapers, prepared by filtration, were used to perform tensile tests. Finally, the efficiency of the combination of TEMPO-mediated oxidation and steam explosion was discussed. Obtained results show that the steam explosion process allows the production of CNF with a width between 4 and 10 nm and properties close to those obtained by the grinding process.

DOI: 10.15376/biores.17.2.2933-2958

Keywords: *Posidonia oceanica*; Cellulose nanofibril; Steam explosion; Grinding; TEMPO-mediated oxidation; Quality index

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INTRODUCTION

Cellulosic fibres are used in various fields, such as pulp and paper industries, and for several applications in packaging, automotive industry, building construction, *etc.* (Belgacem and Gandini 2011; Khiari and Belgacem 2017).

Of all the available wide variety of lignocellulosic sources, wood is the most commonly used in the world (Klemm *et al.* 2005). However, in forest-deficient areas, the utilization of other types of fibres has been developed. Currently, great attention is focused on the exploitation of residues and agricultural crops as alternative sources of cellulosic fibres: kenaf (Villar *et al.* 2009; Valdebenito *et al.* 2017), hemp bast (Puangsin *et al.* 2012),

flax (Cao *et al.* 2007; Alila *et al.* 2013), wheat straw (Josset *et al.* 2014), sisal (Siqueira *et al.* 2010), rice straw (Jiang and Hsieh 2013), bamboo residue (Liu *et al.* 2010), bagasse (Bhattacharya *et al.* 2008; Afra *et al.* 2013; Grande *et al.* 2018), date palm tree (Benhamou *et al.* 2014), alfa (Marrakchi *et al.* 2011), corn cobs and husk (Shogren *et al.* 2011), sugar beet pulp (Habibi and Vignon 2008; Li *et al.* 2014), banana rachis and culinary banana peel (Zuluaga *et al.* 2009; Khawas and Deka 2016), and *Posidonia oceanica* (Bettaieb *et al.* 2015).

This study deals with the investigation of fragments of *Posidonia* (*Posidonia oceanica*), an abundant marine plant in the Mediterranean Sea. The debris from this marine biomass are released by the sea in the form of balls and/or leaves and are accumulated in large quantities on the coasts during each summer season, leading to regular beach cleanings. Therefore, the valorisation of this waste as a source of fibres can be of interest.

Some studies have already been conducted on the delignification of *Posidonia* to produce cellulose, cellulose derivatives (Aguir and M'Henni 2006; Khiari *et al.* 2010), or composites (Rammou *et al.* 2021). Other researchers suggest the exploitation of *Posidonia* waste as an adsorbent material for dyes (Ncibi *et al.* 2006, 2007; Guezguez *et al.* 2009; Dural *et al.* 2011; Douissa *et al.* 2013). These materials have also been studied to produce a renewable and biodegradable nanomaterial called nanocellulose (Bettaieb *et al.* 2015). Since the 1980s, cellulose nanofibril (CNF, also called nanofibrillated cellulose) has been produced, traditionally by the application of specific mechanical treatments, such as microfluidization (Zimmermann *et al.* 2004), homogenization (Dinand *et al.* 1999; Dufresne *et al.* 2000), grinding (Abe *et al.* 2007; Afra *et al.* 2013; Nair *et al.* 2014), or cryo-crushing (Dufresne *et al.* 1997; Chakraborty *et al.* 2005; Alemdar and Sain 2008). Although these processes allow for the preparation of CNF exhibiting good properties, a major problem in all cases, regardless of the processes applied, is the high energy requirement (Spence *et al.* 2011). This is why great attention has been paid to the development of pretreatments leading to a reduction of the energy cost by facilitating the mechanical microfibrillation.

The most common pretreatments are biological and chemical: enzyme-assisted hydrolysis (Henriksson *et al.* 2007; Pääkkö *et al.* 2007), TEMPO-mediated oxidation (Saito *et al.* 2006), carboxymethylation (Heinze and Koschella 2005), and/or solvent-assisted pretreatments (Sirviö *et al.* 2015). In addition, other studies have been oriented towards the use of extrusion with the objective of increasing the concentration of the CNF gel-like suspensions (Ho *et al.* 2015; Rol *et al.* 2017).

Steam explosion (SE), which is considered to be inexpensive and clean, has also attracted several researchers for the production of CNF (Cherian *et al.* 2010; Deepa *et al.* 2011; Diop *et al.* 2015; Bansal *et al.* 2016; Phinichka and Kaenthong 2018). This is a thermomechanical process that allows the fragmentation and/or break-up of the lignocellulosic material by the combination of several factors, namely temperature in the reactor and high shear forces resulting from the sudden pressure drop when the treated material is blown into the final tank. The steam explosion process was firstly introduced for wood disintegration in the manufacture of panels (Mason 1926). Moreover, to produce fermentable sugars and alcohol from wood, Babcock and Kenvii (1932) used this process as a pretreatment of the raw material. In severe conditions, such as long residence time in the reactor or with the use of chemicals, this process leads to the depolymerisation of some lignin and the hydrolysis of hemicelluloses to elementary sugars and water-soluble oligomers (Fernández-Bolaños *et al.* 2001; Sun *et al.* 2005). Recently, this thermo-mechanical process has gained attention again for the separation of cellulose fibres from

different lignocellulosic sources (Kaushik and Singh 2011; Bansal *et al.* 2016; Saelee *et al.* 2016; Luo *et al.* 2018). At the same time, this treatment was proposed for producing CNF from different lignocellulosic materials (Table 1).

Table 1. Various Processing Conditions for Fibres and CNF Production Using Steam Explosion Apparatus or Modified Autoclaves System

Biomass and Reference	Apparatus - Conditions	Objective
Steam Explosion Apparatus		
Softwood pulp (Yamashiki <i>et al.</i> 1990)	<i>T/P</i> : 183 to 252 °C/10 to 49 bar <i>t</i> : 15 to 300 s	Obtention of alkali-soluble cellulose
<i>Miscanthus sinensis</i> (Salvadó <i>et al.</i> 2003)	<i>T/P</i> : 203 °C/16 bar* <i>t</i> : 4 to 14 min	Pretreatment for fibreboard production
Corn cobs and cotton gin waste (Agblevor <i>et al.</i> 2007)	<i>T/P</i> : 220 °C/22 bar* <i>t</i> : 2 min	Fibre extraction
Lotus petiole (Yuan <i>et al.</i> 2013)	<i>T/P</i> : 170 to 190 to 200 °C/10 to 15 to 20 bar <i>t</i> : 10 to 60 to 110 to 160 to 210 s	Fibre extraction
<i>Salix miyabeana</i> (Diop <i>et al.</i> 2015)	<i>T/P</i> : 190 to 205 to 220 °C/12 to 16 to 22 bar* <i>t</i> : 3 to 5 to 10 min	Fibre extraction
Sugarcane (Saelee <i>et al.</i> 2016)	<i>T/P</i> : 195 °C/13 bar <i>t</i> : 15 min	Fibre extraction
Windmill palm (Luo <i>et al.</i> 2018)	<i>T/P</i> : 212 °C/20 bar <i>t</i> : 2 min	Fibre extraction
Pineapple leaves (Tanpichai <i>et al.</i> 2019)	<i>T/P</i> : 215 °C/20 bar <i>t</i> : 5 min	Fibrillation (5 times)
Sugarcane bagasse waste (Hongrattanavichit and Aht-Ong 2020)	<i>T/P</i> : 195 to 200 to 205 to 210 °C/13 to 15 to 17 to 19 bar <i>t</i> : 5 to 10 to 15 min	Fibre extraction
Autoclave		
Banana leaf fibres (Cherian <i>et al.</i> 2008)	<i>T/P</i> : 126 °C*/1.4 bar <i>t</i> : 60 min	First step: Fibres extraction
	<i>T/P</i> : 126 °C*/1.4 bar <i>t</i> : 15 min	Second step: Microfibrillation (8 times)
Wheat straw (Kaushik and Singh 2011)	<i>T/P</i> : 200 °C/20 bar <i>t</i> : 240 min	Fibre extraction
<i>Helicteres isora</i> plant (Chirayil <i>et al.</i> 2014)	<i>T/P</i> : 130 °C*/1.7 bar <i>t</i> : 60 min	First step: Fibre extraction
	<i>T/P</i> : 130 °C*/1.7 bar <i>t</i> : 15 min	Second step: Microfibrillation (8 times)
Jute (Manhas <i>et al.</i> 2015)	<i>T</i> : 115 °C/1 bar* <i>t</i> : 30 min	Microfibrillation
Fresh waste of oil palm empty fruit bunch (Solikhin <i>et al.</i> 2019)	<i>T/P</i> : 121 °C/1.5 bar <i>t</i> : 60 min	First step: Fibre extraction
	<i>T/P</i> : 121 °C/1.5 bar <i>t</i> : 60 to 120 to 180 min	Second step: Microfibrillation
Alfa (Labidi <i>et al.</i> 2019)	<i>T/P</i> : 180 °C/9 bar* <i>t</i> : 20 min	Fibre extraction

Note: *P* is the pressure (bar), *T* is the temperature (°C), *t* is the residence time (min), and * is calculated relative pressure from temperature and *vice versa*.

Various processing conditions of steam explosion were used to separate and/or defibrillate the fibres. However, several authors have worked with modified autoclave systems that do not allow applying high steam pressure, constituting a real limitation. Subsequently, the depressurization stage is not well controlled and its intensity not sufficient. In the literature, it was referred that steam explosion is composed by two steps: steam cracking and explosive decompression. Steam cracking consists in the penetration of water into the material in the form of steam under high pressure and at a high temperature. During the second stage called “explosive decompression”, the wet saturated lignocellulosic material “bursts” as a result of the sudden release of pressure in the reactor. It was shown that the pressure difference between the inside of the reactor and atmospheric pressure is proportional to the intensity of the shear forces applied to the biomass. So, taking advantage of this effect, the authors decided to use steam explosion for microfibrillation.

This paper proposes to study the steam explosion process SE to produce CNF from *Posidonia oceanica* (balls and leaves). A steam explosion pilot with a well-controlled decompressive stage was used. It was coupled to a conventional chemical treatment, *i.e.*, TEMPO-mediated oxidation. Then, the obtained CNF was characterized and compared to TEMPO-oxidized CNF produced by conventional grinding. The objective is to understand the impact of the steam explosion on CNF morphologies (optical microscopy, atomic force microscopy, transmission electron microscopy, turbidity, and nanosized fraction) and mechanical properties (Young’s modulus). Finally, the results were compared with available data from the literature dealing with wood, non-wood, and agricultural crop-based sources. The novelty of this study was to use steam explosion for the first time as the main process for microfibrillation of delignified and bleached *Posidonia* fibres (combined or not to a chemical pretreatment). This could be considered as a short-time process with less reduced environmental impact. The difference with the previous studies is that the explosion conditions were different mostly realized in autoclaves, not in steam explosion pilots with a well-controlled decompressive stage.

EXPERIMENTAL

Materials and Chemical Products

Posidonia oceanica balls (POB) and leaves (POL) were used as a starting raw material to produce CNF fibres. These wastes were collected in Monastir, Tunisia in December 2020. After a washing step, to eliminate sand and contaminants, the material was dried under ambient conditions for one month (average relative humidity: 71%; average temperature: 26 °C). All materials and chemicals were used as received from the suppliers without further purification.

Delignification and Bleaching of *Posidonia oceanica* Fibres

Bleached fibres from *Posidonia* balls (F-POB) and leaves (F-POL) were obtained after two operations, namely, delignification of the raw material and bleaching of the fibrous suspension. Delignification was carried out using soda in the presence of a small quantity of anthraquinone. This method has been commonly used for pulping non-wood plants (Antunes *et al.* 2000). More precisely, the delignification of the *Posidonia* was performed according to the procedure described by Khiari *et al.* (2010). For this purpose, 60 g (dry material) of raw material was treated at 160 °C with an alkali charge (sodium

hydroxide) of 20% (w/w, based on dry material) and a liquor to solid ratio fixed at 10. Additionally, 0.1% (w/w, dry material) of anthraquinone was added as a catalyst. Pulping was carried out in electrically-heated rotating autoclaves (one-litre volume) for 120 min. Afterwards, the pulp was washed and filtered using a nylon mesh (100 μm) and finally was stored in a freezer.

After delignification, the bleaching step was completed with sodium chlorite (NaClO_2) in a buffer medium. This treatment mainly consisted of two phases: a sodium chlorite bleaching phase followed by an alkaline extraction. For the bleaching phase, 50 g of the delignified pulp were treated in a heat-sealable bag with buffer solution (pH <4.7) prepared from sodium acetate trihydrate and acetic acid. Then 3.95 g of NaClO_2 dissolved in 100 mL of deionized water were added and the bag was filled with water to 1 kg. Then, it was kept in a water bath for 90 min at 80 °C and shaken manually every 5 min. At the end, sodium sulfite was added to neutralize the chlorine dioxide, and then the pulp was filtered with a nylon sieve of 40 μm mesh size and washed with water.

An alkaline extraction is then performed to facilitate lignin extraction. In a heat-sealable bag, the treated pulp is introduced into 400 mL of a sodium hydroxide solution (3% w/w). The bag was filled up to 1 kg with deionized water, then closed, kept in a water bath at 70 °C for 60 min and stirred every 5 min. Afterwards, the pulp was filtered with a nylon sieve of 40 μm mesh size and washed with water. Those two steps were repeated 5 times.

CNF Production

Fibres refining

Using the PFI mill, as per ISO 5264-2 (2011) standard, 300 mL of an aqueous suspension containing 30 g of dry bleached pulp (10% w/w) were refined until a drainage index of 82 °SR (Schopper Riegler degree), which corresponds to 6000 and 4000 revolutions of the mill for F-POB and F-POL, respectively.

TEMPO-mediated oxidation of fibres

Chemical modification of the fibres by TEMPO (2,2,6,6-tetramethylpiperidine-1-oxide radical)-oxidation was performed to selectively convert the primary hydroxyl of cellulose to carboxylate functions (Saito *et al.* 2006; Isogai *et al.* 2011). The bleached and refined pulp at a concentration of 1% (w/w) was treated according to the protocol proposed by Saito *et al.* (2006). The TEMPO/NaBr/NaClO system was used at 25 °C with 0.1/1/6.22 mmol of the reactants per gram of cellulose (mmol/g), respectively. The ensuing TEMPO-oxidized cellulose fibres were filtered and washed using deionized water until the filtrate conductivity reached a value below 5 $\mu\text{S cm}^{-1}$.

Microfibrillation by steam explosion or grinding

Steam explosion experiments were conducted at LERMAB, Nancy, France. These experiments involved three main steps. First, 100 g of dry fibres were placed in a 4.8-L reactor that once closed, was saturated with steam until the target couple of temperature/pressure (170 °C/8 bar) was reached. These conditions were kept for 30 s. The release regulator was then opened, and the material was subjected to a sudden pressure drop (depressurization) during its rapid ejection from the reactor to be finally recovered in the blow tank. These conditions were selected after a preliminary study. In particular, the low residence time was chosen to limit the degradation of cellulose and hemicelluloses.

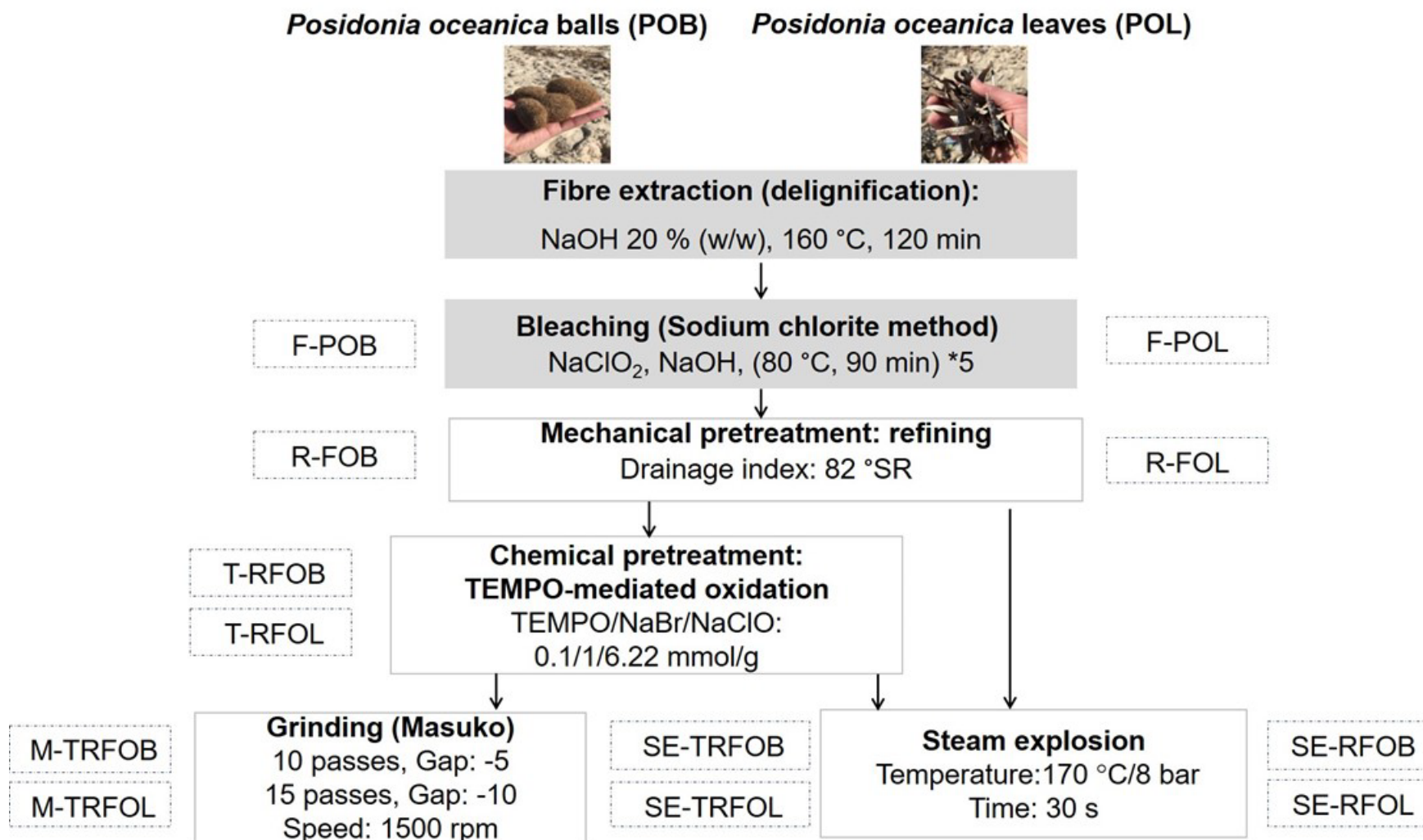


Fig. 1. Schematic diagram of the proposed pathways to produce CNF from POB and POL

In this study, the main objective of the steam explosion was to take advantage of the sudden drop of pressure and to assess if a possible effect on the fibres (expected breakage of the cell wall) may be sufficient to produce CNF from TEMPO-oxidized fibres. Grinding was also done using the ultrafine friction grinder Supermasscolloider (model MKZA6-2, disk model MKG-C 80, Masuko Sangyo Co., Ltd., Saitama, Japan), available at LGP2, Grenoble, France. The ultrafine grinder was used at 1500 rpm to treat the fibre suspension. The nominal gaps between the rotating and the static stones were -5 for 10 passes and -10 for 15 passes. Figure 1 presents the proposed routes leading to the preparation of CNF using the different selected processes (cooking, bleaching, TEMPO oxidation, steam explosion, or grinding). Table 2 summarizes all sample labels.

Table 2. Abbreviations of the Different Samples

Label	Samples
POB	Raw <i>Posidonia</i> balls
POL	Raw <i>Posidonia</i> leaves
F	Separation and bleaching
R	Refining
T	TEMPO-mediated oxidation
SE	Steam explosion
M	Grinding by Masuko
F-POB	Bleached fibres from <i>Posidonia</i> balls
F-POL	Bleached fibres from <i>Posidonia</i> leaves
R-FOB	Refined and bleached fibres from <i>Posidonia</i> balls
R-FOL	Refined and bleached fibres from <i>Posidonia</i> leaves
T-RFOB	TEMPO-oxidation of refined and bleached fibres from <i>Posidonia</i> balls
T-RFOL	TEMPO-oxidation of refined and bleached fibres from <i>Posidonia</i> leaves
SE-RFOB	Steam explosion of refined and bleached fibres from <i>Posidonia</i> balls
SE-RFOL	Steam explosion of refined and bleached fibres from <i>Posidonia</i> leaves
SE-TRFOB	Steam explosion of TEMPO-oxidized, refined, and bleached fibres from <i>Posidonia</i> balls
SE-TRFOL	Steam explosion of TEMPO-oxidized, refined, and bleached fibres from <i>Posidonia</i> leaves
M-TRFOB	Masuko grinding of TEMPO-oxidized, refined, and bleached fibres from <i>Posidonia</i> balls
M-TRFOL	Masuko grinding of TEMPO-oxidized, refined, and bleached fibres from <i>Posidonia</i> leaves

Characterisation

Several methods and techniques were used to characterize the obtained fibres as well as the produced CNF.

Carboxylic acid content

Conductometric titrations were used to determine the carboxylate content of the TEMPO-oxidized fibres (Besbes *et al.* 2011). To this purpose, TEMPO-oxidized fibres (0.5 g dry matter) were suspended in 10 mL of HCl (0.1 mol L⁻¹) in such a way that the pH

reached 3. Afterwards, the suspension was titrated with NaOH (0.01 mol L⁻¹). Titration curves were used to calculate the carboxyl content according to the following Eq. 1,

$$C(\text{COOH: (mmol g}^{-1}\text{)}) = \frac{C_{\text{NaOH}}*(V_2-V_1)}{w} \quad (1)$$

where C_{NaOH} is the concentration of NaOH (mmol L⁻¹), V_1 is the equivalent volume of NaOH solution added to titrate the strong acid corresponding to the excess of HCl (L), V_2 is the equivalent volume of NaOH solution added to titrate the weak acid corresponding to the carboxyl content (L), and w is the dry weight of sample (g).

Morphological properties

The morphological properties of the prepared samples were investigated. To determine the size distribution and fines content of the suspensions, a MorFi LB-01 fibre analyser (Techpap, Gières, France) was used. Suspensions were diluted to 0.3 g L⁻¹. The fibres/fines limit were set at 200 µm in length, and the analysis time was set at 5 min. The measurements were triplicated.

Optical microscopy was performed in transmission mode, using an Axio Imager M1 optical microscope (Carl Zeiss, Munich, Germany). For each prepared CNF gel-like suspension (at a concentration of 0.5 wt%) at least 10 images were taken.

Atomic force microscopy is a method for visualizing the topography of the sample surface or for characterizing the physical properties of materials with a resolution between 0.1 and 10 nm (under the best conditions for microscopic observation). Atomic force microscopic (DI, Veeco, Instrumentation Group, Plainview, NY, USA) observations were thus performed by using CNF gel-like suspensions diluted at 10⁻⁴% (w/w) with deionized water. A droplet of the prepared sample was deposited on a mica disc and then allowed to dry under ambient conditions for 24 h.

Transmission electron microscopic analysis was performed using a JEOL 200CX transmission electron microscope at 80 kV. The images were obtained after depositing approximately 0.5 µL of the diluted suspension onto a carbon-coated 300-mesh copper grid.

Fourier transform infrared spectroscopy (FTIR) and crystallinity index (CI)

Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) was used to characterize the prepared papers from refined pulp and TEMPO-oxidized pulp (prepared by filtration under vacuum: see material and method, section Quality index). The spectra were obtained using a spectrophotometer (Spectrum 65 FT-IR, Perkin-Elmer, Waltham, MA, USA). It covered the spectral range from 600 to 4000 cm⁻¹. The acquisition conditions of this analysis were 16 scans and 2 cm⁻¹ resolution.

The crystalline structure of the prepared samples was studied using an X-ray diffractometer (XRD) (D8-Advance, Bruker, Billerica, MA, USA) at room temperature with a monochromatic CuKα radiation source ($\lambda = 0.154$ nm). Experiments were performed in step-scan mode with a 2θ angle ranging from 5° to 60° with a step of 0.04 and a scanning time of 5.0 min. The CI was calculated using deconvolution method (amorphous subtraction method).

Quality index

A quality index (Desmaisons *et al.* 2017) was used to facilitate the monitoring of nanocellulose production at laboratory and industrial scales. The simplified quality index

(QI^*) was based on four parameters, namely nanosized fraction, turbidity, average microparticle size (μm^2) of CNF gel-like suspensions, and Young's modulus of nanopapers. The QI^* is expressed according to Eq. 2,

$$QI^* = 0.3 \times x_1 - 0.03 \times x_2 - 0.072 \times x_3^2 + 2.54 \times x_3 - 5.34 \ln(x_4) + 58.62 \quad (2)$$

where x_1 represents the nanosized fraction (%), x_2 is the turbidity (NTU), x_3 is the Young's modulus (GPa), and x_4 is the average microparticle size (μm^2).

The nanosized fraction in CNF gel-like suspensions was measured according to Naderi *et al.* (2015). This method allows determining the quantity of particles at the nanometric scale in the CNF gel-like suspensions by a gravimetric method. At first, the CNF gel-like suspensions were diluted to 0.02% (w/w) and centrifuged at 1000 g for 15 min using a centrifuge (Sigma 3-18 KS, Sigma Laborzentrifugen GmbH, Osterode am Harz, Germany). The concentrations in the supernatant before (C_{bc}) and after centrifugation (C_{ac}) were used to calculate the nanosized fraction according to Eq. 3,

$$\text{NF (\%)} = \frac{C_{ac}}{C_{bc}} \times 100 \quad (3)$$

where the concentration is expressed in percentage (w/w).

A turbidimeter (AL250T-IR, Aqualytic, Dortmund, Germany) was used to measure the turbidity of the diluted CNF suspensions (0.1% w/w). Turbidity is related to the size, number, and refractive index of the visible suspended particles. The presence of nanoparticles that are not detectable makes the value of the turbidity close to zero. The average of the turbidity values was obtained from the measurement of five different samples.

The CNF gel-like suspensions were diluted to 0.5 wt%, and the resulting samples were observed with an optical microscope (Axio Imager M1, Carl Zeiss, Munich, Germany). Images were taken at 20x magnification and analysed with Fiji software (ImageJ, Java 1.8.0_172, Madison, WI, USA). They were converted to 8-byte type and thresholded before assessing the average length of the visible particles remaining in the suspension after steam explosion or grinding. At least five pictures were taken, and the most representative ones were used.

Nanopapers with basis weight of approximately 60 g m^{-2} were finally produced using a standard sheet former (Rapid-Köthen, Noviprofibre, Eybens, France) as per ISO 5269-2 (2004) standard and equipped with a nylon sieve of $1 \mu\text{m}$ mesh size (Buisine, Clermont de l'Oise, France). The suspensions were filtered under vacuum, and the obtained wet mats were dried for 12 min at $90 \text{ }^\circ\text{C}$. For each CNF suspension, five nanopapers were produced and stored at $23 \text{ }^\circ\text{C}$ and 50% relative humidity (RH) for at least 48 h before further characterisation. The thickness of the nanopapers was measured using a micrometer (Adamel Lhomargy, Sms-Labo, Saint-Baldoph, France) at different positions on the nanopaper, and an average value was used for further calculation (m).

Finally, the Young's modulus of the nanopapers were measured. The samples (width of 15 mm and length between jaws of 100 mm) were tested using a vertical extensometer an (Instron 5965, Instron, Norwood, MA, USA) with a capacity of 5 kN and a tensile velocity of 10 mm/min.

RESULTS AND DISCUSSION

Fibre Properties

Carboxylic acid content

As previously explained, bleached and refined fibres, R-FOB and R-FOL, were submitted to a TEMPO-mediated oxidation (TEMPO/NaBr/NaClO).

Table 3. Carboxylic Content of Various Biomasses Treated under Different Conditions by TEMPO-mediated Oxidation

Biomass	Oxidation System (mmol g ⁻¹)	Carboxylic Content (mmol g ⁻¹)	Reference
Wood Sources			
Dried sulfite pulp	TEMPO/NaBr/NaClO: 0.08/1.21/6.3 ^c	1.52	Saito <i>et al.</i> (2006)
Hardwood bleached kraft pulp	TEMPO/NaBr/NaClO: 0.1/1/5 ^a	0.30	Fukuzumi <i>et al.</i> (2009)
Softwood kraft pulp	TEMPO/NaBr/NaClO: 0.08/1.21/10 ^c	1.70	Isogai <i>et al.</i> (2011)
Bisulfite softwood pulp	TEMPO/NaBr/NaClO: 0.1/1/5 ^a	1.75	Nechyporchuk <i>et al.</i> (2015)
<i>Pinus radiata</i> kraft pulp	TEMPO/NaBr/NaClO: 0.064/0.97/11.6 ^c	0.95	Valdebenito <i>et al.</i> (2017)
Never dried bleached <i>Eucalyptus globulus</i> kraft pulp	TEMPO/NaBr/NaClO: n.m/n.m/7 ^b	1.41	Alves <i>et al.</i> (2020)
Hardwood dissolving pulp	TEMPO/NaBr/NaClO: 0.08/1.21/5 ^c	1.32	Levanič <i>et al.</i> (2020)
Agricultural and Annual Plants			
Cotton	TEMPO/NaBr/NaClO: 0.1/0.97/10 ^c	1.36	Okita <i>et al.</i> (2010)
Kraft bleached bagasse pulp	TEMPO/NaBr/NaClO: 0.1/0.97/7.5 ^c	1.45	Puangsin <i>et al.</i> (2012)
Hemp bast holocellulose	TEMPO/NaBr/NaClO: 0.1/0.97/7.5 ^c	1.50	Puangsin <i>et al.</i> (2012)
Commercial bamboo	TEMPO/NaBr/NaClO: 0.1/0.97/7.5 ^c	1.70	Puangsin <i>et al.</i> (2012)
Rice straw	TEMPO/NaBr/NaClO: 0.1/0.97/5 ^c	1.32	Jiang and Hsieh (2013)
Bleached <i>Posidonia</i> leaves	TEMPO/NaBr/NaClO: 0.1/1/6.22 ^a	0.65	Bettaieb <i>et al.</i> (2015)
Bleached <i>Posidonia</i> balls	TEMPO/NaBr/NaClO: 0.1/1/6.22 ^a	0.70	Bettaieb <i>et al.</i> (2015)
Bamboo pulp	TEMPO/NaBr/NaClO: 0.1/1/10 ^a	1.88	Chen <i>et al.</i> (2017)
Oat hulls	TEMPO/NaBr/NaClO: 0.064/0.97/11.6 ^c	0.70	Valdebenito <i>et al.</i> (2017)
Bagasse	TEMPO/NaBr/NaClO: 0.1/1/8 ^c	1.29	Lu <i>et al.</i> (2018)

Biomass	Oxidation System (mmol g ⁻¹)	Carboxylic Content (mmol g ⁻¹)	Reference
F-POB: bleached fibres from <i>Posidonia</i> balls	TEMPO/NaBr/NaClO: 0.1/1/6.22	0.97	This work
F-POL: bleached fibres from <i>Posidonia</i> leaves	TEMPO/NaBr/NaClO: 0.1/1/6.22	1.07	This work

Note: ^a: through methods described in the article; ^b: given information does not allow for the calculation of a ratio per g or not mentioned; ^c: recalculated values are in the form of mmol g⁻¹ (without considering the purity of chemicals); n.m: not-mentioned

This reaction was intended to modify the cellulose and to convert the primary alcohols of carbons from glucose rings to carboxylic acid cycles. Under the tested conditions, the treated samples were produced with important carboxylic contents, which are 0.97 mmol g⁻¹ and 1.07 mmol g⁻¹ for T-RFOB and T-RFOL, respectively.

The present results agree with previous studies (Bettaieb *et al.* 2015). Table 3 summarizes the carboxylic acid content of various biomasses treated under different conditions by TEMPO-mediated oxidation, showing that the amounts of acidic groups ranged from 0.3 to 1.75 mmol g⁻¹. This range depended on many factors, especially the chemical composition of the starting materials, the different treatments undergone by the fibres during the bleaching step, and finally the quantities of TEMPO/NaBr/NaClO reagents. However, it is worth noting that 0.3 mmol g⁻¹ of ionic groups seemed to be a minimal level to perceive an effect on the microfibrillation behavior (Besbes *et al.* 2011).

Morphological properties

Fibre morphology was assessed for the unrefined and refined pulps as well as for TEMPO-oxidized fibres by using SEM analysis and MorFi Lab. From SEM observations, it appears that the length of *Posidonia oceanica* fibres was remarkably lower than that observed for other common plants. MorFi analysis confirmed this observation: the average length and width as well as the percentage in fine elements are summarized in Table 4.

Table 4. Morphological Properties and Crystallinity of Bleached F-POB and F-POL, Refined R-FOB and R-FOL, and TEMPO-oxidized Fibres T-RFOB and T-RFOL

	Mean Arithmetic Length (µm)	Mean Width (µm)	Fines Content (%)	Crystallinity Index by Deconvolution Method (%)
F-POB	406.0 ± 1.0	21.5 ± 0.8	20.7 ± 1.0	50
R-FOB	374.5 ± 3.5	20.6 ± 0.2	77.6 ± 0.1	68
T-RFOB	371.7 ± 0.6	18.3 ± 0.1	75.1 ± 0.1	73
F-POL	484.0 ± 1.0	24.2 ± 0.1	65.4 ± 0.1	47
R-FOL	381.7 ± 4.2	23.2 ± 0.1	91.2 ± 0.2	63
T-RFOL	319.5 ± 2.6	18.7 ± 0.2	95.7 ± 0.1	71

It is worth noting that the unrefined fibre length, whatever the starting raw material, was relatively low compared to other vegetal sources: *Amaranthus caudatus* L. (Fiserova *et al.* 2006), orache (Fiserova *et al.* 2006), Jerusalem artichoke (Fiserova *et al.* 2006), *Cannabis sativa* (Dutt *et al.* 2008), or *Cynara cardunculus* L. (Antunes *et al.* 2000; Abrantes *et al.* 2007). In contrast, the width seemed to be comparable with most wood, non-wood, as well as non-perennial fibres. It is also important to mention that the unrefined POL was characterized by a high content of fine elements (60% in length). Refining treatment induced variations of the fibre morphology, which were more noticeable for POL. Furthermore, the decrease of the fibre length was negligible for POB despite the large increase in fine element content. Contrastingly, for POL, the fibre length decreased from 484 to 381 μm . This behavior could be explained by the difference between the intrinsic strength of the fibres in the starting materials (Khiari *et al.* 2010; Bettaieb *et al.* 2015). TEMPO-oxidation importantly affected fibre morphology by weakening/fragilizing the cell wall structure. It was found in previous studies that TEMPO-mediated oxidation initiated microfibrillation of the fibres by the incorporation of carboxylic groups and fragilization of fibres by breaking of cellulose chains (Tarrés *et al.* 2017). These phenomena could explain the decrease of width and length of fibres after TEMPO-oxidation.

XRD and FTIR analysis

X-ray diffraction analysis was used to investigate the effect of the applied treatment on fibre crystallinity, namely the different polymorphic forms, the ratio of crystalline to amorphous regions, and the degree of crystallinity. The XRD patterns of all the tested samples are shown in Figs. 2a and 2b. It can be deduced from this figure that all the samples showed three main peaks at $2\theta = 16.5^\circ$, 22.6° , and 34.5° corresponding respectively to the (110), (200), and (004) crystalline diffraction planes of cellulose type I (Khiari *et al.* 2010; Khiari and Belgacem 2020). The appearance of the peaks at $2\theta = 16.5^\circ$ and $2\theta = 22.6^\circ$ in all XRD patterns confirmed that cellulose was of type I.

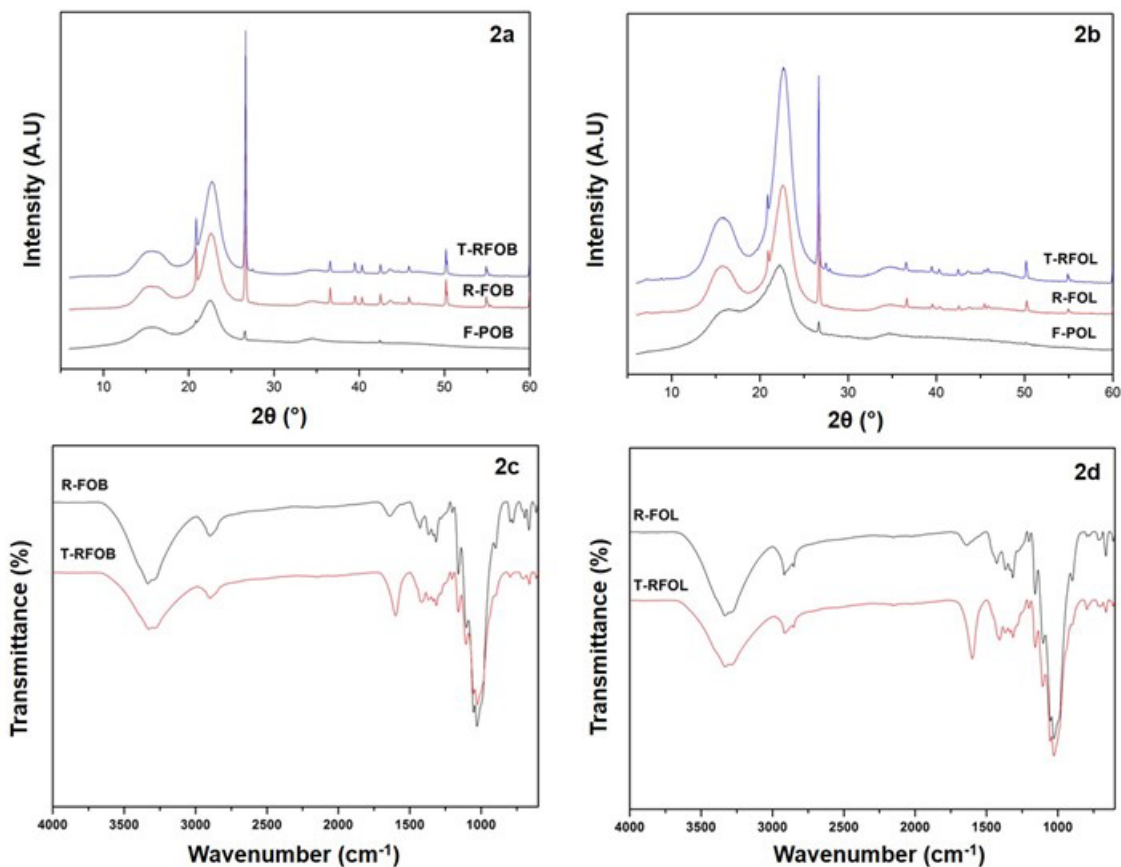


Fig. 2. (a) X-ray diffractogram of *Posidonia* balls; (b) X-ray diffractogram of *Posidonia* leaves; (c) ATR spectra of *Posidonia* balls; (d) ATR spectra of *Posidonia* leaves

Moreover, peaks around 25° reflect the presence of mineral compounds, namely quartz (SiO_2) and $\text{Ca}_{(1-x)}\text{Mg}_{(x)}\text{CO}_3$. The presence of these mineral salts is typical in sea biomass and related to the high ash content (Bettaieb *et al.* 2015). The results, reported in Table 4, show that the crystallinity index depended on the treatment conditions: CrI increased with refining and TEMPO-oxidation treatment. This increase reached 30% for the refining treatment. During this operation, which was performed in an intense way, the final Schopper Riegler degree reached approximately 80, a considerable fraction of hemicelluloses would thus have been released from the fibres. This would explain the increase in CrI.

The ATR analysis results of R-FOB, T-RFOB, R-FOL, and T-RFOL are shown in Figs. 2c and 2d. The spectra display characteristic cellulose absorption bands, such as the $-\text{CH}$ stretching bands at 2900 cm^{-1} and the $-\text{CH}_2$ band at 2950 cm^{-1} , as well as the broad peak between 3000 and 3650 cm^{-1} due to $-\text{OH}$ stretching. The band around 1645 cm^{-1} corresponds to the $-\text{OH}$ bending mode of adsorbed water. The presence of different peaks at 1602 and 1409 cm^{-1} can also be noticed in the figure, which correspond to the vibrations of carboxylate groups ($-\text{COO}^-$) (Saini *et al.* 2016; Michel *et al.* 2020), thus clearly confirming the oxidized state of the bleached fibres from *Posidonia* after TEMPO oxidation.

Characterisation of CNF

Non-oxidized fibres produced by steam explosion

Cellulose fibres produced by SE using non-oxidized *Posidonia* fibres are shown in Fig. 3. An important number of coarse elements was observed by optical microscopy (Fig. 3.1). Likewise, even if the transmission electron microscope (TEM) images (Fig. 3.2) showed some individualised nanofibrils of cellulose with a width between 2 and 8 nm for *Posidonia* balls (3 and 13 nm for *Posidonia* leaves), elements with a diameter of more than 100 nm were also present.

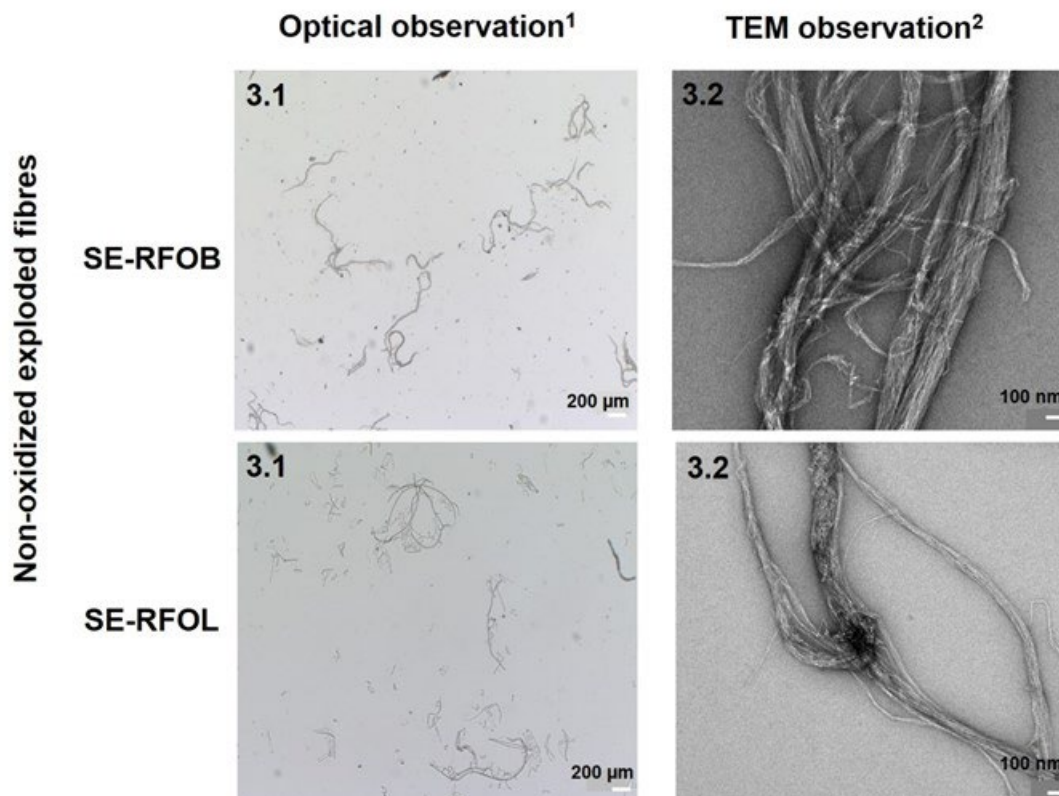


Fig. 3. Morphological aspect of non-oxidized nanofibres of cellulose produced using a steam explosion: 1) optical observations, 2) TEM observations

From this morphological analysis, it can then be concluded that coupling the two processes of refining and SE does not constitute an efficient way to produce CNF with a narrow width distribution.

Turbidity of the gel-like suspensions was equal to 402 and 388 NTU for *Posidonia* balls and leaves, respectively. These relatively high values confirmed the presence of large particles in the non-oxidized suspensions, as already discussed. Nevertheless, the suspensions presented a nanosized fraction of approximately 54%, which still reflected the presence of nanofibril of cellulose. Nanopapers exhibited Young's modulus of 3.0 and 5.8 GPa for *Posidonia* balls and leaves, respectively. Such a result was lower than the CNF obtained from the same raw material by grinding (Bettaieb *et al.* 2015). Consequently, it can be concluded that steam explosion without any chemical pretreatment did not allow an effective individualisation of the cellulose nanofibres.

Oxidized-CNF produced by grinding or steam explosion

The CNF gel-like suspensions obtained from oxidized pulps, ground or steam exploded, were fully characterized. Figure 4 shows the different CNF gel-like suspensions images obtained from optical microscopy, atomic force microscopy (AFM), and transmission electron microscopy (TEM). For CNF gel-like suspensions obtained by grinding, optical microscopy (Fig. 4a.1) revealed the presence of a reduced number of coarse particles compared to Fig. 3.1. This confirmed the effectiveness of microfibrillation for TEMPO-oxidized *Posidonia* fibres by grinding. The AFM and TEM images (Fig. 4a.2 and Fig. 4a.3) also show the production of cellulose nanofibrils. The mean width of the prepared nanofibrils, between 2 and 7 nm from balls (3 and 8 nm from leaves), was low and in agreement with the literature (Table 5).

Table 5. Width Values of CNF Produced *via* Masuko and TEMPO-mediated Oxidation and Steam Explosion Process

Raw Material	Width (nm)	Reference
Masuko and TEMPO-mediated Oxidation		
Bisulfite softwood pulp	4	Nechyporchuk <i>et al.</i> (2015)
Bleached <i>Posidonia</i> balls	5 to 21	Bettaieb <i>et al.</i> (2015)
Bleached <i>Posidonia</i> leaves	5 to 15	Bettaieb <i>et al.</i> (2015)
Bleached kraft eucalyptus	3 to 8	Qin <i>et al.</i> (2015)
Bleached bagasse pulp	3 to 10	Abouzeid <i>et al.</i> (2018)
Bleached softwood	20	Liu <i>et al.</i> (2019)
Bleached hardwood kraft pulp	5 to 10	Siqueira <i>et al.</i> (2019)
Bleached CTMP pulp	10 to 20	Bakkari <i>et al.</i> (2019)
Bleached bagasse pulp	10 to 20	Al-Ahmed <i>et al.</i> (2020)
Bleached cane pulp	4 to 10	Abou-Zeid <i>et al.</i> (2020)
Bleached <i>Posidonia</i> balls	2 to 7	This work
Bleached <i>Posidonia</i> leaves	3 to 9	This work
Steam Explosion		
Pineapple leaf fibres	5 to 60	Cherian <i>et al.</i> (2010)
Pineapple, banana, and jute fibres	5 to 40	Abraham <i>et al.</i> (2011)
Oil palm empty fruit brunch	23 to 78	Solikhin <i>et al.</i> (2019)
Bleached <i>Posidonia</i> balls (TEMPO-oxidation)	3 to 11	This work
Bleached <i>Posidonia</i> leaves (TEMPO-oxidation)	4 to 13	This work

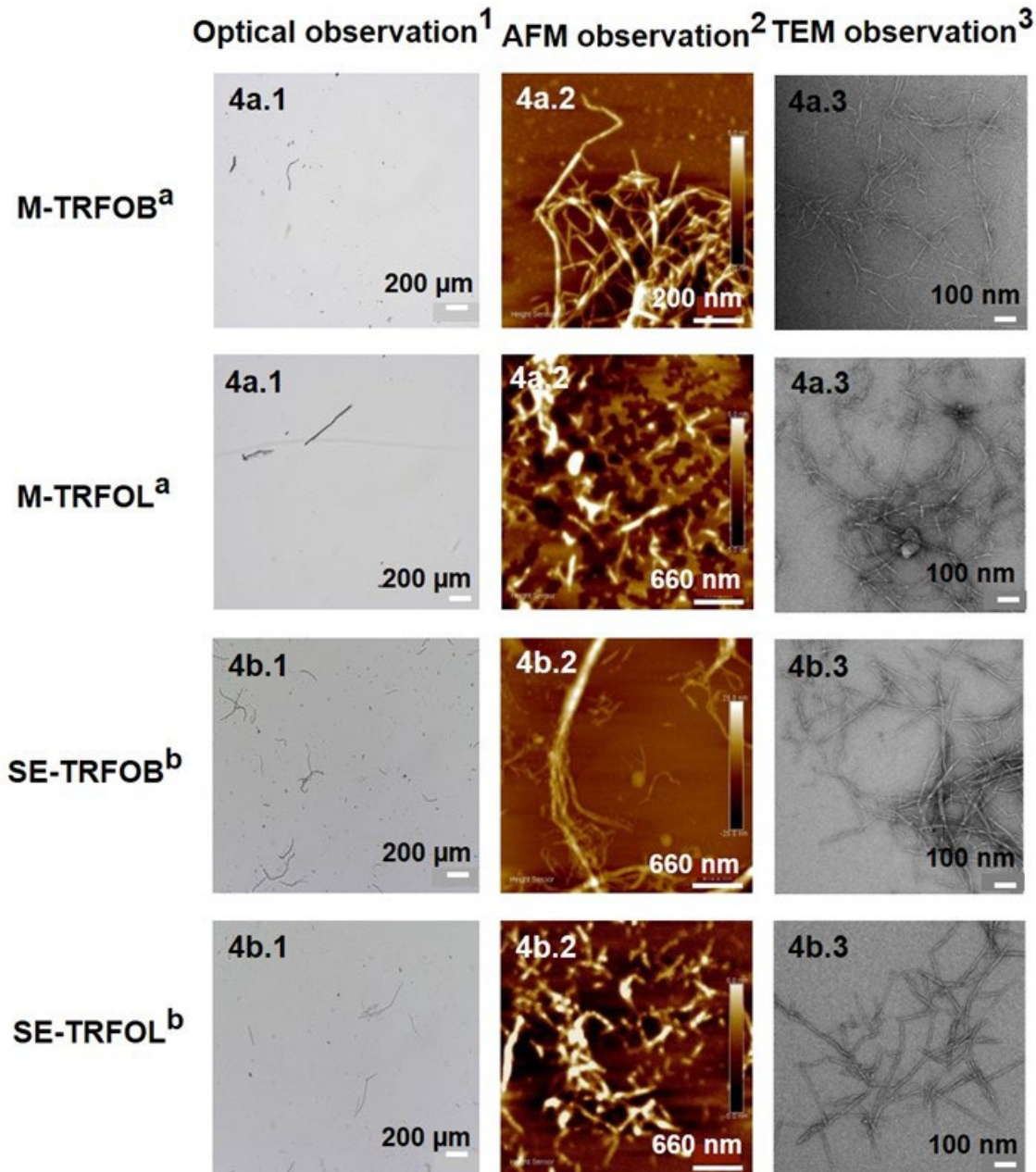


Fig. 4. 1) optical, 2) AFM, and 3) TEM observations of the oxidized CNF; a) for ground CNF and b) for steam exploded CNF

Regarding the now oxidized-CNF obtained by steam explosion, optical microscopy (Fig. 4b.1) showed a higher number of coarse particles compared to the ground gel-like suspension. The AFM images (Fig. 4b.2) highlight the presence of nanometric cellulose fibril, which is also revealed by TEM images (Fig. 4b.3) showing individualised nanofibrils together with some aggregates. The mean width of the observed nanofibrils of cellulose from *Posidonia* balls was between 3 and 11 nm (4 and 13 nm from *Posidonia* leaves). These results are comparable with previous studies results (see Table 5).

Table 6. Quality Indexes for the Different CNF Obtained in this Work Compared to Data from the Literature

	Process and Raw Material	Average Microparticle Size (μm^2)	Nanosized Fraction (%)	Turbidity (NTU)	Young's Modulus E (GPa) for nanopapers	<i>Qf</i>
This Work	<i>Posidonia</i> balls refined and steam exploded	40.7 \pm 7.9	54.0 \pm 2.0	402 \pm 17	3.0 \pm 0.2	52.3 \pm 2.4
	<i>Posidonia</i> leaves refined and steam exploded	35.6 \pm 18.2	49.0 \pm 2.1	388 \pm 16	5.8 \pm 0.3	57.2 \pm 3.7
	<i>Posidonia</i> balls refined, TEMPO and steam exploded	14.7 \pm 12.8	76.4 \pm 3.8	200 \pm 8	7.4 \pm 0.3	78.8 \pm 5.2
	<i>Posidonia</i> leaves refined, TEMPO and steam exploded	10.7 \pm 6.5	81.8 \pm 4.8	213 \pm 15	8.8 \pm 0.4	83.8 \pm 5.0
	<i>Posidonia</i> balls refined, TEMPO and ground by Masuko	24.2 \pm 16.9	85.4 \pm 3.5	249 \pm 10	12.0 \pm 0.5	80.0 \pm 4.6
	<i>Posidonia</i> leaves refined, TEMPO and ground by Masuko	16.2 \pm 9.5	85.6 \pm 4.3	235 \pm 5	11.5 \pm 0.5	85.1 \pm 4.1
Literature	Bleached eucalyptus enzymatic pretreatment + homogenizer (Rol <i>et al.</i> 2018)	20.3 \pm 0.6	63.2 \pm 5.3	67 \pm 3	15.2 \pm 0.5	81.5 \pm 2.1
	Bleached eucalyptus enzymatic pretreatment + homogenizer + twin-screw extruder (Rol <i>et al.</i> 2018)	30.0 \pm 3.8	86.4 \pm 11.1	91 \pm 11	15.4 \pm 0.3	85.7 \pm 3.4
	Bleached eucalyptus enzymatic pretreatment + twin screw extruder (Rol <i>et al.</i> 2019)	41 \pm 12	64.3 \pm 9.9	289 \pm 13	11.6 \pm 0.3	69.5 \pm 1.8
	Bleached eucalyptus cationic pretreatment + twin-screw extruder (Rol <i>et al.</i> 2019)	131 \pm 59	54.6 \pm 2.5	113 \pm 6	7.3 \pm 1.0	60.7 \pm 0.4

Quality index

To compare the produced CNF in a more thorough way, the quality indexes were assessed. The obtained data are summarized in Table 6, which also includes data from previously published works, for comparison purposes.

Table 6 shows that the quality indexes (QI^*) of the CNF produced in this work were in line with data from the literature, independently of the process and the applied pretreatment, except for non-oxidized steam exploded fibres, which presented, as expected, lower values of QI^* . For the latter, the quality indexes were comprised between 52 and 57 for *Posidonia* balls and leaves, respectively. These values were lower than that encountered for gel-like suspensions obtained by grinding of enzymatically treated fibres, generally higher than 65 (Desmaisons *et al.* 2017).

Compared to previous studies, the four measured parameters were close, apart from turbidity that seemed to be higher than that reported for TEMPO-oxidized wood, *i.e.*, 50 NTU. This can be explained by the heterogeneity of suspensions, due to the presence of residual fibres in a non-negligible quantity in the prepared samples, which may negatively impact the mechanical properties of nanopapers. Indeed, Young's moduli do not exceed 12 GPa, and it is important to mention that the values were relatively low for SE-TRFOB and SE-TRFOL (where steam explosion was used as the main microfibrillation process). Probably for exploded samples, some failure zones were created during nanopaper preparation, due to the presence of more residual fibres. Therefore, the existence of these coarse elements limits the mechanical properties, but it is still important.

In contrast, the microparticle sizes and nanosized fractions evolved in a classical way depending on the treatment intensity.

CONCLUSIONS

1. During this work, cellulose nanofibril (CNF) suspensions were successfully produced from a marine biomass waste, namely *Posidonia* balls and leaves, after delignification and bleaching, by combining TEMPO-oxidation as a chemical pretreatment with the ultra-fine grinding or steam explosion.
2. The obtained CNF was extensively characterized using complementary methods. Steam explosion made it possible to produce CNF with morphological and mechanical properties comparable to those conventionally produced by grinding.
3. Promising results were obtained when steam explosion was applied to non-oxidized fibres. Indeed, considering the mild conditions used in this work (170 °C, 30 s), an improvement in the quality of the gel-like suspensions may be expected by applying a more severe steam explosion.
4. Finally, this work confirms that *Posidonia oceanica* could be promising alternative biomass for manufacturing CNF products in the future.

ACKNOWLEDGMENTS

This work was financially supported by the “PHC Utique” program of the French Ministry of Foreign Affairs and Ministry of Higher Education, Research and Innovation

and the Tunisian Ministry of Higher Education and Scientific Research in the CMCU (project number TN 18G1132// FR 39316VF). The LGP2 is part of the LabEx Tec 21 (Investissements d'Avenir - grant agreement n°ANR-11-LABX-0030) and of PolyNat Carnot Institute (Investissements d'Avenir - grant agreement n° ANR-16-CARN-0025-01). This research was made possible thanks to the facilities of the TekLiCell platform funded by the Région Rhône-Alpes (ERDF: European Regional Development Fund).

In addition, the authors would like to thank Thierry Encinas from CMTC – Grenoble, France for the XRD analysis and Christine Lancelon-Pin from CERMAV Grenoble, France for the TEM observations.

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Article submitted: December 22, 2021; Peer review completed: March 20, 2022; Revised version received and accepted: March 29, 2022; Published: April 6, 2022.
DOI: 10.15376/biores.17.2.2933-2958