



Pilot plant up-scaling of tannin foams



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ARTICLE INFO

Article history:

Received 20 July 2015

Received in revised form 2 November 2015

Accepted 3 November 2015

Available online 21 November 2015

Keywords:

Tannin foams

Natural materials

Lightweight panels

Pilot up-scale

Sandwich boards

ABSTRACT

In the last years many studies were led to characterise and optimise the tannin-furanic foams. In the present work the up-scaling of the production from laboratory to industrial scale is discussed. The tannin foams produced in an industrial pilot plant have shown comparable properties with the laboratory-produced foams. In particular the panels manufactured in the continuous press presented slightly higher bulk densities of around 176 kg/m^3 (+20%) which led to higher mechanical properties and thermal conductivities. The up-scaled tannin foams of around 150 kg/m^3 presented 20–25% enhanced compression resistances (28 MPa) and thermal conductivities ($55 \text{ mW/m} \times \text{K}$). Lightweight sandwich panels with various side layers were produced directly in the press, without the need of external adhesives. From the marketing viewing angle, the up-scaled tannin foams show higher densities than the other commercial synthetic insulation materials but the mechanical and insulating performances observed are promising and suggests interesting industrial developments for this innovative bio-based product.

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1. Introduction

Condensed tannins (also called polyflavonoids or proanthocyanidins) are hydroxy aromatic polymers which can be easily extracted in high percentages from plants like pine, spruce, mimosa or quebracho (Hagerman et al., 1998; Pizzi, 2008; Kemppainen et al., 2014; Arbenz and Avérous, 2015). These bio-resources have similar reactivity than phenol and hence they can produce stable polymers when combined with hardeners such as formaldehyde, glyoxal, hexamine and furfuryl alcohol (Coppens et al., 1980; Ballerini et al., 2005; Pichelin et al., 1999; Foo and Hemingway, 1985). Until now, adhesives, wood preservatives and varnishes were the most common traditional applications which exploited this polymerization reaction (Sowunmi et al., 1996; Abdullah et al., 2013; Thévenon et al., 2010). Recently this reaction was applied also for producing porous materials such as aerogels and foams (Amaral-Labat et al., 2013; Merkleheim and Pizzi, 1994).

In particular the tannin foams were considered as a very promising material already when discovered, even if only a few applications for this material were proposed. The presence of expensive organic catalysts and the significant amount of formaldehyde (6,5%) used for their production, were two reasons for classifying the first tannin foams only as a prototype product (Tondi

and Pizzi 2009). In the last years intensive studies were carried out on this subject and very valuable improvements were done. The group of Pizzi and Celzard did a broad research considering various aspects of tannin foams: Initially the characterization of the material (Pizzi et al., 2008; Tondi et al., 2008, 2009a,b; Zhao et al., 2010a,b) and its derivatisation (Tondi et al., 2009b, 2010) were prior. Successively the developments of foams (i) with other tannin resources (Lacoste et al., 2013a,b; De Yuso et al., 2014); and (ii) with tailored properties (Celzard et al., 2011; Li et al., 2012, 2013; Sanchez-Martin et al., 2013) were investigated. Recently, the productions of “green” foams at room temperature and in alkaline conditions were also achieved (Basso et al., 2013, 2014a).

In the last years, our group was investigating more applicative aspects of the foam in order to prepare this technology for large-scale production. The synthesis of foams without formaldehyde was the main focus and the solution we found consisted in increasing the input energy during production, catalysing the cross-linking capacity of furfuryl alcohol and tannin. Formaldehyde-free foams, indeed, were successfully obtained producing them in the hot-press (Link et al., 2011) or with alternative irradiative energy sources such as the microwaves and the infrared beams (Kolbitsch et al., 2012; Tondi et al., 2014a). Detailed vibrational investigations based on the chemistry of formaldehyde-free foams (Tondi et al., 2014b, 2015a; Reyer et al., 2015) have allowed to define the chemistry of these tannin-furanic polymers. A general overview concerning the development of 100% natural tannin-furanic foams and their

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possible applications were also recently presented (Tondi et al., 2015b; Link et al., 2015).

Sandwich panels with tannin foams as a core layer were successfully produced in lab-scale (Link et al., 2011; Zhou et al., 2013) and they showed improved properties which would allow them to be used for the production of green insulation materials.

Tannin-furanic-polyurethane foams were successfully produced also in an industrial scale (Basso et al., 2014b) but until now, completely natural, aldehyde-free foams and their related sandwich panels have not yet produced in industrial pilot plants.

In this paper the up-scaling process as well as a comparison between laboratory (short cycle press) and the pilot plant industrial (continuous isochoric press) produced foams are presented.

2. Material and method

2.1. Materials

For the production of formaldehyde-free tannin-based rigid foams, the industrially extracted tannin from Mimosa (*Acacia Mearnsii*) delivered by Silva Chimica was used as the major component. Furfuryl alcohol supplied by International Furan Chemicals BV was used as co-monomer. Diethyl ether and sulphuric acid (32 %) were delivered by C. Roth and used as solvent and catalyst, respectively. Charges were also included in the formulations to regulate the initial viscosity: recycled tannin foam powder ($\varphi = 125\text{--}500\ \mu\text{m}$) or fractioned wood chips ($\varphi = 500\text{--}1000\ \text{mm}$) were selected.

Various materials were used as side layers for the sandwich panels: white and recycled paper, melamine laminate, wood veneer, plywood, particleboards and HDF.

2.2. Methods

2.2.1. Preparation of the formulation

According to the volume of foam to be obtained the following materials were mixed: 50% of tannin was mechanically stirred with 12.5% of furfuryl alcohol, 12.5% of water, 5% of charges (foam powder or wood chips) and 3.3% of diethyl ether. When the solution was homogeneous 16.6% of acid catalyst was added to the batch and stirred until homogeneity. The so-prepared formulation was then ready to be spread in the mould for foaming.

2.2.2. Production of foams

The lab-scale foams were produced as follow: The formulations were homogeneously spread on the bottom-layer and bordered with a mobile metallic frame of $60 \times 60 \times 2.5\ \text{cm}$. The top layer panel was then laid on top of the metal frame. The complete system was then transferred in a Höfler (HLOP 280) hot-press with the temperature of the plates set at $120\ ^\circ\text{C}$. The side layers and

the metal frame were previously pre-heated for 2 min at the same temperature. The complete system was let react for 5 min until the tannin-furanic resin was completely set.

A similar process was applied for the production of the foams in the industrial continuous press. In this case a special frame of MDF of $150 \times 100 \times 2.5\ \text{cm}^3$ (internal area = $0.72\ \text{m}^2$) was used. An isochoric continuous press (Hymen) with a width of 1.3 m and a length of 6.0 m was applied. The temperature inside the press was set to $120\ ^\circ\text{C}$ and the conveyor speed was calculated for having 5 min heating time for the whole length of the panel in order to reproduce the more similar conditions to that of the lab.

The foams obtained were then stored in normative climate ($20\ ^\circ\text{C}/65\% \text{ m.c.}$) for at least 48 h before being cut to the required dimensions for normative testing.

2.2.3. Density and homogeneity

The bulk density was calculated registering weight, length, width and thickness according to the DIN EN 323 (1993):

$$\rho = \frac{m}{(l \times w \times h)} = \left[\frac{\text{kg}}{\text{m}^3} \right]$$

The homogeneities of the tannin foams were controlled by X-ray scanning of the produced panel. The measurements of the density profile were performed using a Dense-lab X from EWS.

2.2.4. Mechanical properties

All the mechanical tests were performed in a Zwick-Roell Z250 universal testing machine.

The compression resistance was evaluated to samples of $50 \times 50 \times 25\ \text{mm}^3$ of foam according to the standard DIN 52185 (1976) with a compression rate of 2 mm/min. The compressive strength of the samples was measured towards the direction of growth. The point of maximal resistance was taken at the end of the elastic region, when the first crack occurred.

The internal bond for the industrially produced foams was measured on sandwich boards with HDF as side layers of $50 \times 50 \times 31\ \text{mm}^3$ (25 mm of foam and 3 mm of HDF side panels) which were hot-melt glued to metallic supports. The samples were tested along the growing direction with an opening rate of 2 mm/min. Percentage of failure of the glue-line between the foam and the HDF side layers was also reported.

Samples of sandwich boards with HDF as side-layers ($500 \times 50 \times 31\ \text{mm}^3$) were tested towards their bending strength and their modulus of elasticity according to the standard EN 310 (1993).

The screw tear resistance of 100% natural tannin-furanic foams was evaluated according to the standard EN 13446 (2002) using M3 screws. The screws were fixed vertically to the tannin-foam surfaces and in the middle of samples with the size $50 \times 50 \times 25\ \text{mm}^3$. The screwing depth was 20 mm.

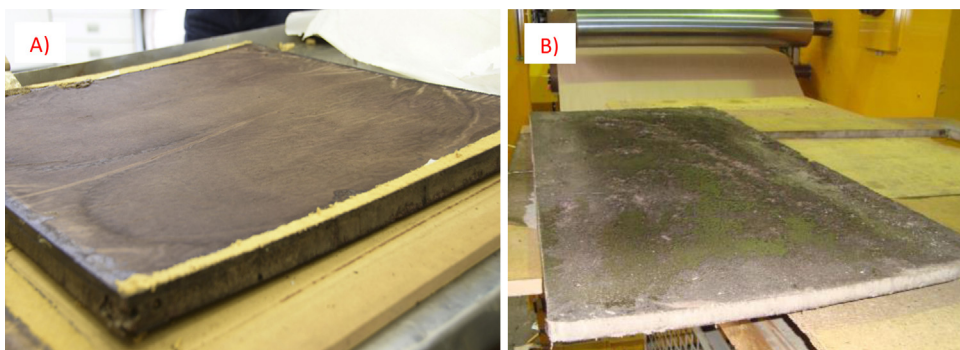


Fig. 1. Tannin-based foams produced in (a) lab-scale and (b) industrial scale.

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