



Bark based porous materials obtained with a simple mechanical foaming procedure

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Abstract

Tree bark is a by-product of the wood industry and has currently only little use as raw material. In this study, spruce bark disintegrated into three different types of particles was used to obtain porous structures with a wide range of properties. The manufacturing process includes a simple mechanical foaming method, using bark particles, a common wood adhesive, a surfactant and water. Physical and mechanical characterization of the materials was carried out in terms of density, thermal conductivity, water uptake, compression resistance and microscopy observation. All materials produced presented a heterogeneous open porous structure. Thermal conductivity values range between 0.075 and 0.125 W m⁻¹ K⁻¹, while the density values range between 100 and 650 kg/m³. Water uptake percentage varies between groups but is stabilized after 24 h of immersion, and in some cases, the water uptake reaches up to 450%. Regarding the mechanical properties, they vary greatly showing a tendency of decreasing when adding higher amounts of surfactant.

1 Introduction

Bark is the outer layer of trees and corresponds to between 9 and 24% of the tree stem's volume depending on the tree species (Smith and Kozak 1967). It is mainly responsible for the transport of nutrients, but also for ensuring the protection of the cambium from any outer threats as fungi, extreme temperatures and mechanical impacts (Fengel and Wegener 1989). Despite its relevance for the plant, bark is still treated as a low value by-product of the timber industry and accumulates in rough quantities (Wagner et al. 2019). As a result, further valorization of bark pieces, which are detached from the tree when cut, is needed. Every year,

millions of cubic meters are predominately burned (Feng et al. 2013), which creates emissions that are declared as CO₂ neutral, as the released carbon dioxide was captured during the tree's growth. From this point of view, biomass that is directly fueled after harvesting does not contribute to reduce the overall CO₂ concentration in the atmosphere. Hence, the nature inspired use of renewable resources in materials that also contribute to a reduction in CO₂ emissions through their function seems to be a better approach than instantly fueling the raw materials.

Currently households, manufacturing, electricity supply, agriculture, transportation and storage are quite resource-consuming sectors and major contributors to CO₂ emissions in the European Union (European Commission 2018). For this reason, developing materials inspired by nature could both enhance the life cycle of bark in the wood industry and help the current environmental situation by designing new materials for the packaging or building construction industries.

Regarding bark, one of its main drawbacks is its chemical heterogeneity (Kuo et al. 2018; Wenig et al. 2021), which makes it difficult to introduce it in large industries. This is why studying bark in granular form can be interesting and

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a helpful strategy. Bark has been investigated for years to be used for panel production or as part of wood composites. In addition, one of the most studied bonding agents has been urea formaldehyde resin (UF), investigated since the 1970s (Feng et al. 2013; Muszynski and McNatt 1984) examined the gradual addition (from 0 to 100%) of bark in wood panels using urea formaldehyde resin as the adhesive. The strength properties decrease with the addition of bark, but even with a 30% addition of bark the mechanical properties were appropriate for furniture production (Muszynski and McNatt 1984). Similar results were obtained by Xing et al. (2006) who produced medium density fiber boards with refined bark fibers of several species varying its content from 0 to 40% and urea formaldehyde resin. No negative effect on thickness swelling was reported here (Xing et al. 2006). Nemli and Colakoglu (2005) proved that an addition of more than 12.5% of bark reduces significantly the mechanical properties and is therefore not recommended, even though the formaldehyde emissions are diminished. Pressing temperature and particle size of bark particles have also been the subject of other studies, concluding that they have a great influence on the final density and properties. Mechanical and physical properties are shown to improve with the pressing temperature and the use of fine particles, of approximately 1 mm, compared to bigger or mixed particle sizes (Gupta et al. 2011). Kain et al. (2012, 2018) produced different thermal insulation panels (200 and 500 kg/m³) using bark particles and urea formaldehyde as a binder. Additionally, a resin formulation based on tannin was used for bark based insulation panels, that acquired excellent thermal conductivity values of 69 mW/(mK) with densities of 250 kg/m³ (Kain et al. 2016). Pásztor et al. (2019) also reported data for bark insulating panels made out of different species and particle sizes. Boards made from particles with sizes between 1 and 8 mm showed a thermal conductivity of 64.5 mW/mK, and lower formaldehyde emission than boards produced with bigger particle sizes (Pásztor et al. 2017). The same group also investigated the addition of fiber glass and paper sheets to bark panels with urea formaldehyde in order to improve their properties and develop a thermal insulation material. Thermal conductivity values ranged between 67 and 74 mW/mK (Tsalagkas et al. 2019).

Besides investigations as potential raw material for the wood panel production, tree bark was also examined as a source to obtain chemicals and produce adhesives, mainly with its main extractive: tannins (Ogunwusi 2013). Studies on tannin-based resins have been performed since the 1950s and condensed tannins are preferred compared to hydrolysable tannins for this specific application (Feng et al. 2013). In addition, tannin foams are another product that is being studied. Tannin is a polyol both with aliphatic and aromatic hydroxyl groups. As a consequence it can be used

to produce tannin-based polyurethane foams, tannin rigid foams and tannin formaldehyde furfuryl foams (Tondi and Pizzi 2009; Feng et al. 2013). There are several production methods using different formulations that create different types of porosity and therefore properties. One simple method to obtain foams is mechanical frothing. It has been used both for tannin-based foams (Szczurek et al. 2014) and with other by-products of the wood and pulp industry like black liquor (Jalalian et al. 2018). Industrially, mechanical foaming is not considered a major technology to produce polymeric foams (Lee et al. 1998). Other methods like soluble or reactive foaming are more common and well-developed techniques. However, mechanical foaming is still used for some batch process, like in the production of latex foams (Eaves 2004). Additionally, it has the advantage of being an inexpensive and simple manufacturing technique. Another important parameter to take into account is the type of bark used. In this study spruce bark was selected. Spruce is a coniferous tree whose bark is formed by sieve cells, the walls of which are made up of small pores, making the bark a porous structure itself (Fengel and Wegener 1989). This could be beneficial since small pores within the structure can have an impact on lowering the thermal conductivity of the material (Kain et al. 2013).

In the present study, a mechanical foaming method was applied to obtain several types of open porous materials, using bark as the main component. In addition, PVAc-D3, a common adhesive in the wood industry, and a surfactant (sodium dodecyl sulfate) were added to maintain the porous structure. PVAc is a thermoplastic used as an emulsion. In its structure, there are free hydroxyl groups that can cross-link with other compounds (Conner 2001). Other common adhesives like urea-formaldehyde have been studied to produce foams using the multifold reactions of urea and formaldehyde. Unfortunately, these materials tend to be brittle and highly negatively influenced by water absorption (Liu et al. 2019; Wu et al. 2020).

It is the aim of the present study to investigate the influence of different types of particle sizes and morphologies of bark as well as different amounts of surfactant on the properties of the materials produced. All the materials obtained showed an open macroporous and heterogeneous structure. Properties like density, thermal conductivity, water immersion and mechanical properties were studied, and some of them varied greatly as a function of the surfactant amount and the type of particles used.

2 Materials and methods

For the experiments, spruce bark (*Picea abies*) with a moisture content of approx. 60% was obtained directly from the sawmill debarking process (Stora Enso, Ypps a.d.Donau, Austria). An adhesive, PVAc-D3, (Rakoll GXL 3, H.B.Fuller) with a solid content of 52%, in combination with the anionic surfactant sodium laurylsulfate (SDS, >99%, Carl Roth GmbH & Co.KG, Karlsruhe, Germany) with a molar ratio of 288.38 g/mol and distilled water were used to build the bark foam matrix.

2.1 Milling and fibrillation of spruce bark

Three different particle sizes were produced to obtain the maximum possible variability of the raw material and to investigate its full potential. Therefore, two different disintegration procedures were used, a dry process with a hammer mill disintegration and a wet process using a single disc laboratory refiner. For the hammer mill disintegration, the bark was dried at 60 °C for 14 days to a moisture content of 5%.

The dry milling was conducted by two devices. First, a Retsch SM1 (Retsch GmbH, Haan, Germany) was used with a sieve screen of 5 mm and afterwards the material was milled with a Retsch ZM200 using sieves of 2 mm and 0.2 mm, to obtain two different particle types. They are called in this study, milled particles – 2 and milled particles – 0.2, respectively.

For the wet refining process, bark pieces were pre-chopped into squares of 20 mm side length and then treated with water vapour at 140 °C for 15 min before milling. The refining process was performed in a 12 " single-disc laboratory-refiner (Andritz, Graz, Austria), where two commercially available refiner discs for wooden fibreboard production were used. The milling gap of the discs was set to 0.1 mm. After the wet disintegration, the refined particles were kiln dried at 60 °C for 14 days to a moisture content of approx. 5%.

2.2 Investigation of morphological properties

Particle size distribution of each fraction was measured in accordance with EN 17827 (ISO 17827-2:2016 2016) using a vertical sieve shaker (AS 200, Retsch GmbH, Germany). The mesh sizes were: 0.2, 0.4, 0.6, 0.8, 1, 1.4, 2 and 3.15 mm. The measurement procedure was performed with an amplitude of 2 mm at 1 Hz for 30 min. Additionally, five repetitions were executed for each particle fraction. An optical morphological analysis of the different particle fractions was conducted using a digital microscope (DSX1000, Olympus).

Table 1 Specific surfactant contents in mmol/L added to 120 ml of water

Water ml	Surfactant mmol/l
120	0.0
	2.0
	4.0
	8.0
	10.0

2.3 Mechanical foaming

All samples were prepared by means of mechanical foaming, using a planetary mixer (CTTC Clatronic KM 3632 1200 W). For the foaming procedure, the PVAc, SDS and distilled water were whipped using the planetary mixer at maximum mixing speed until a stable foam structure was built up (approx. 60 s). If all the whipped material remained at the bottom of the mixing cup when it was turned over, a stable foam structure had been achieved. Subsequently, the bark material was added slowly to the foam and stirred in for another minute. The whipping time and the output power of the mixer were kept constant over all whipping steps and foams produced.

For the experiment, the particle type of bark and the molar concentration of the SDS solution were the studied variables. Adhesive and bark, with respect to their dry mass, and water were mixed in the same amounts for all foams produced, following a ratio of 1:1:2 respectively. The surfactant was added in low but specific portions to the water volume as shown in Table 1. For every foam sample, a dry mass of 60 g adhesive and bark were used respectively. The wet porous emulsion was cast in aluminium containers of size 140×115×40 mm³ that were covered with baking paper and put in a kiln at 60 °C for 24 h to harden. After the drying step, the samples were entirely removed from the containers and stored at 20 °C/65% RH until equilibrium mass (mass change < 0.1%/12 h) was reached.

2.4 Physical and mechanical characterization

Firstly, samples were cut into blocks (25×25×12.5 mm³) and conditioned (20 °C/65% RH). The density of twelve samples per variant was measured in accordance with ÖNORM ISO 3131:1996 (Austrian Standards Institute (ASI) 1975). Afterwards, thermal conductivity was evaluated using a miniature heat flow meter (Mini-HFM), described in detail in a previous work (Vay et al. 2021). Two samples were placed together forming a cube of 25 mm in height and packed into polyethylene foil to prevent biasing of occurring temperature or humidity gradients to the surrounding. Six repetitions were performed. The samples were placed into an ideally fitting polyethylene foam frame

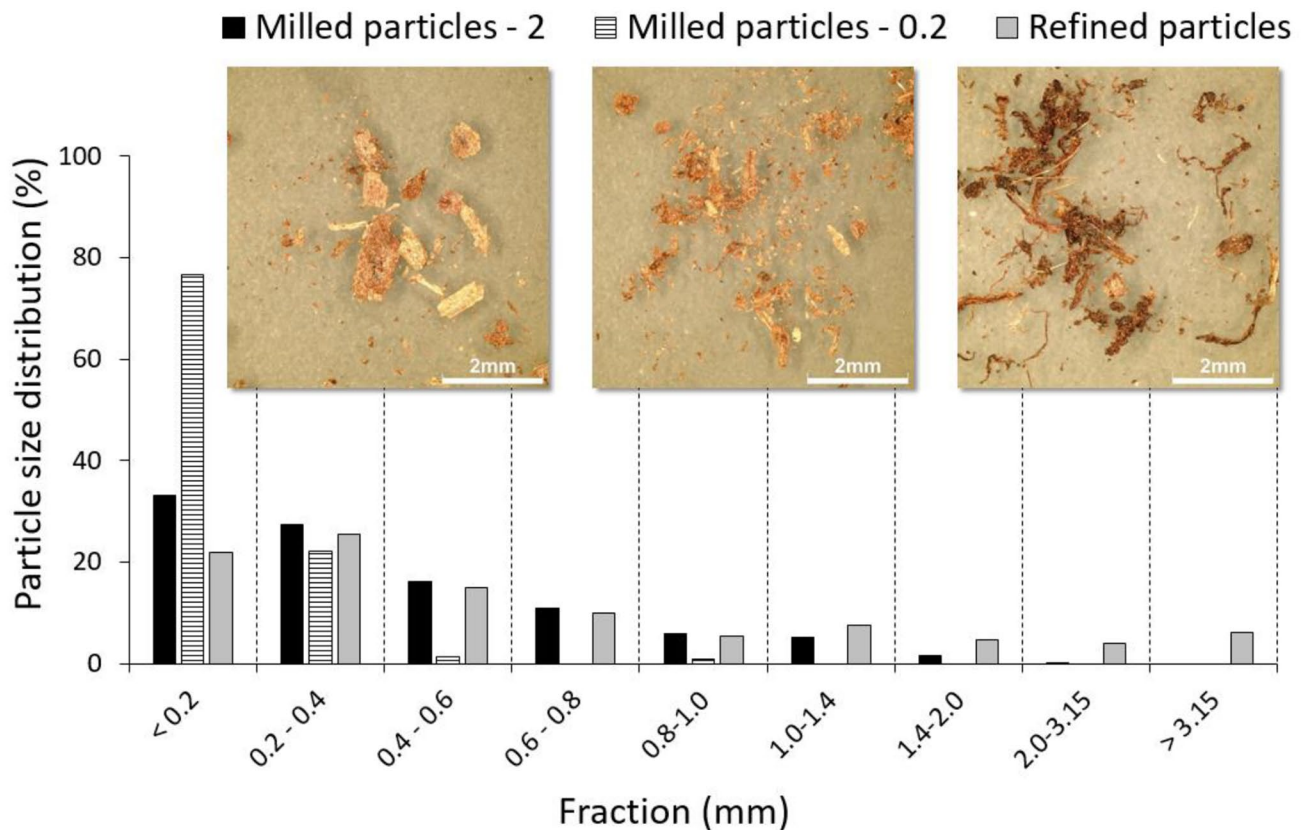


Fig. 1 Frequency results and morphology of the particles used in the study, obtained by a vibrating screen method using sieves and light microscopy, respectively

(Plastazote LD 29, Zotefoam, England) and positioned between the flux sensor and the heating plate. The temperature difference was constantly 20 K for all measurements. For both thermal conductivity and density results a statistical one-way ANOVA analysis with a post-hoc Scheffé test was performed to compare mean pairs ($p=0.05$).

The morphology of the materials and particles produced was observed using an Olympus DSX 1000 digital microscope in incident light mode.

To evaluate the mechanical stability of the foam structures, a compression resistance test was performed in accordance with EN 826:2013 by means of a universal testing machine (Zwick/Roell Z100, Germany). Samples cut with dimensions of $50 \times 50 \times 20 \text{ mm}^3$ were compressed with a constant displacement rate of 2 mm/min by 20% of their initial height at a preload of 250 Pa. The maximum stress at 20% strain (σ_{20}) was tested for five replicates per sample type.

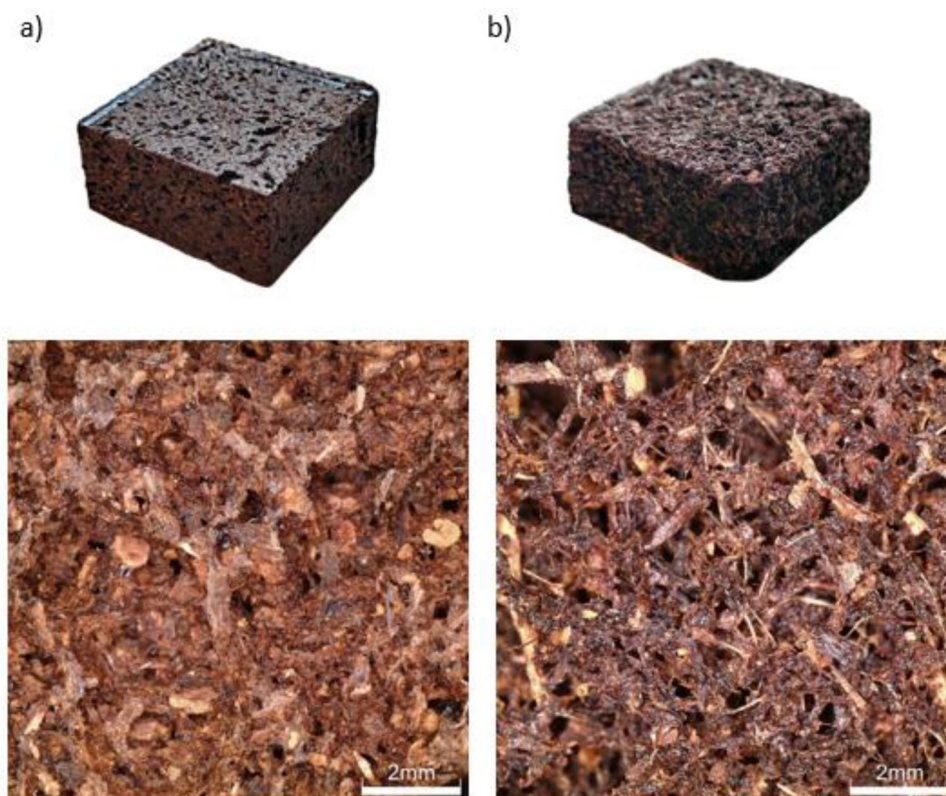
To analyze moisture sorption behavior of bark foam cubes ($5 \times 5 \times 5 \text{ mm}^3$) in terms of mass change over time, a gravimetric dynamic vapor sorption device (DVS Advantage 1, Surface Measurement Systems Ltd., London) with a resolution of 0.1 μg was used. The samples were exposed to

the following order of 0, 60, 95, 60 and 0% relative humidity inside a sealed and dry nitrogen-controlled testing chamber.

Additionally, the water uptake of bark samples was analyzed, by immersing the probes ($25 \times 25 \times 12.5 \text{ mm}^3$) in 100 ml of distilled water with 0.005% of sodium azide to avoid microbial growth. The samples were kept submerged at room temperature for seven days. The water uptake (W) was calculated by the difference of mass at a certain time m_t and the initial mass m_0 divided by the initial mass (Eq. 1). Before measuring the samples, they were drained for 30 min. Three repetitions were performed per sample type.

$$W = \frac{m_t - m_0}{m_0} \quad (1)$$

Fig. 2 Images of the materials produced with 8 mmol/l of surfactant: (a) milled particles –0.2 and (b) refined particles



3 Results

3.1 Particle characterization

As shown by light microscopy and particle size distribution analysis, the three types of particles used in the study exhibit significant differences in terms of size and morphology. Figure 1 displays the particle size distribution and the morphology of the particles, demonstrating that different milling procedures lead to different particle types.

Dry milled particles independent of the mesh size have very different shapes, whereas the majority of the refined material is of a fibrous nature with higher length to thickness ratios. For this reason, this type of particles is highly entangled, and conglomerates can be produced. The particle size distribution of the batch with smallest particles (milled particles –0.2) is narrow and concentrated. Additionally, despite of using the 0.2 mm sieve to produce this batch, in the particle size distribution results bigger particles can be observed. The bigger set of milled particles (milled particles –2) and the refined particles present similar and broader distributions, especially when analyzing the fraction smaller than 2 mm. Although both distributions are very similar, when observing the morphology of the particles with light microscopy, some differences in terms of length-to-diameter ratio can be noticed.

3.2 Material characterization

The foams produced show very different structures depending on the different amounts of surfactant and particles used. All materials had an open and heterogeneous porous structure. Generally, the more surfactant and the larger the particle size, the more pores are formed. Figure 2 shows an example where the same amount of surfactant is used (8 mmol/l) but different particles, milled particles –0.2 and refined particles. Both a macroscopic and microscopic image are shown. In the case of the refined particles, it is seen that the fibres enhance the production of a stable porous material. On the other hand having a set of particles with high dust contents, as in the case of the dry milled particles, produces agglomeration and as a result, denser foams, even with high levels of surfactant.

As depicted in Fig. 3, density and thermal conductivity results seem to decrease as the surfactant content increases. Density results are widely distributed depending on the particle type and the surfactant concentration used. The material produced with the refined particles present the lowest density values. Density results vary between 0.15 g cm^{-3} for refined particles and the highest amount of surfactant to 0.54 g cm^{-3} for materials produced with the smallest particles and no surfactant. Regarding the thermal conductivity results, there is no value lower than $0.06 \text{ W m}^{-1} \text{ K}^{-1}$. A relation between density and thermal conductivity can be

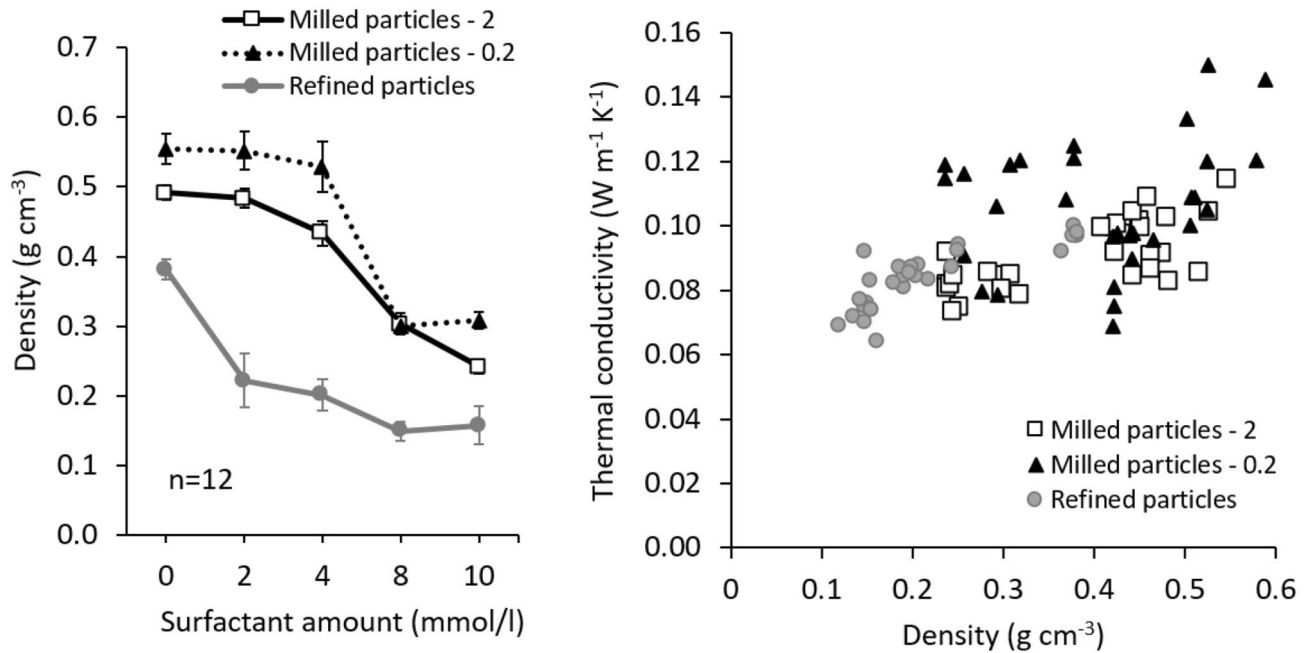


Fig. 3 Density and thermal conductivity results depending on the type of particles used and surfactant amount

observed. Furthermore, it can also be observed that when analyzing materials with the same density but produced with different particle types, those made with smaller particles (milled particles – 0.2) have higher thermal conductivity values. Additionally, the materials formed with the refined particles present the lowest results for both parameters.

The ANOVA result shows that there are statistically significant differences (sig. < 0.05) when using some specific particles and surfactant amounts. When analysing density, the medium particles – 1, there were significant differences when using higher amounts of surfactants (above 2 mmol/l), whereas when using refined particles these differences were not significant. Thermal conductivity results were statistically significant when comparing the lowest and highest amount of surfactant, but similar amounts, for example, 0 and 2 mmol/l and 8 and 10 mmol/l showed to be not statistically significant.

Regarding the mechanical properties, there is a relationship between the amount of surfactant and the compression resistance of the material, independent of the particle type used. For all types of bark, the higher the surfactant concentration the more fragile materials are produced. Denser and harder materials are obtained when using lower amount of surfactant and sets of particles with higher amounts of dust. Figure 4 illustrates the maximum compression stress of the materials in relation to its density. There is a direct relation between density and stress. In addition, all particles show a drastic decrease in stress at a specific density. For the smallest particles (milled particles – 0.2), this decrease appears

at density values of 0.4 g cm^{-3} , whereas for the refined particles this decrease is shown at 0.25 g cm^{-3} .

Water vapour sorption showed that at 95% relative humidity, the materials took up to 18.5% moisture, being the maximum values obtained with the refined particles and the milled particles – 2 using the higher surfactant concentration. In all three types of particles, changing the amount of surfactant does not seem to have a noticeable influence on the water vapour sorption of the materials produced. Figure 5 shows a representative isotherm of the samples produced with 8 mmol/l and the three different particle types.

Figure 6 depicts the immersion test results. Initially, for all particle types and surfactant amounts, water is absorbed until the first 24 h. From then on, the water uptake is not as noticeable, except for the materials produced with refined particles and no surfactant added, which has an approximately 75% increase from 24 to 144 h. In all materials studied, the higher the amount of surfactant the higher the mass change when immersed in water. Additionally, there is a clear difference in materials produced with surfactant amounts lower than 8 mmol/l for milled particles – 0.2 and refined particles. This difference is found with lower amounts of surfactant (2 mmol/l) when using bigger dry milled particles, i.e. milled particles – 2. Regarding materials with no surfactant added, the differences between all particle types are not as wide as when adding a surfactant. However, when surfactant is added, even in low concentrations (2 mmol/l) the water uptake increases, especially when using refined particles until up to 350% with this amount of

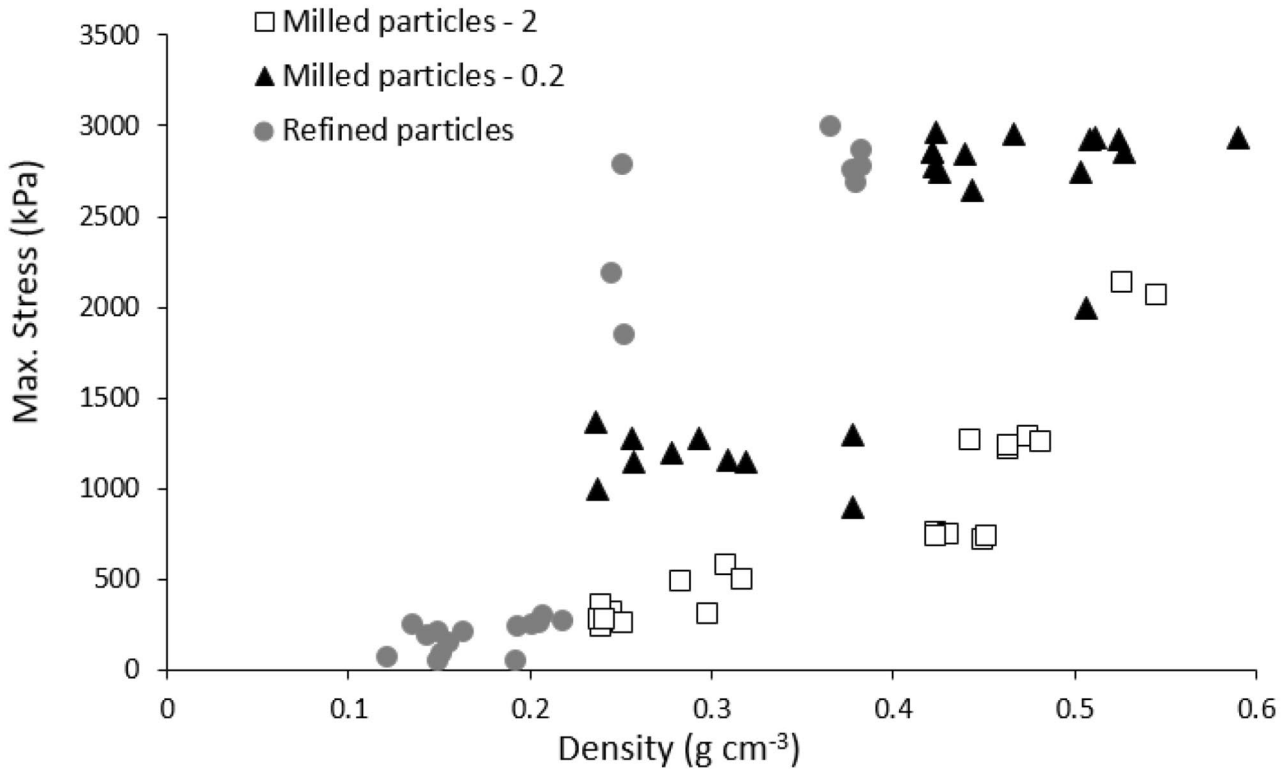
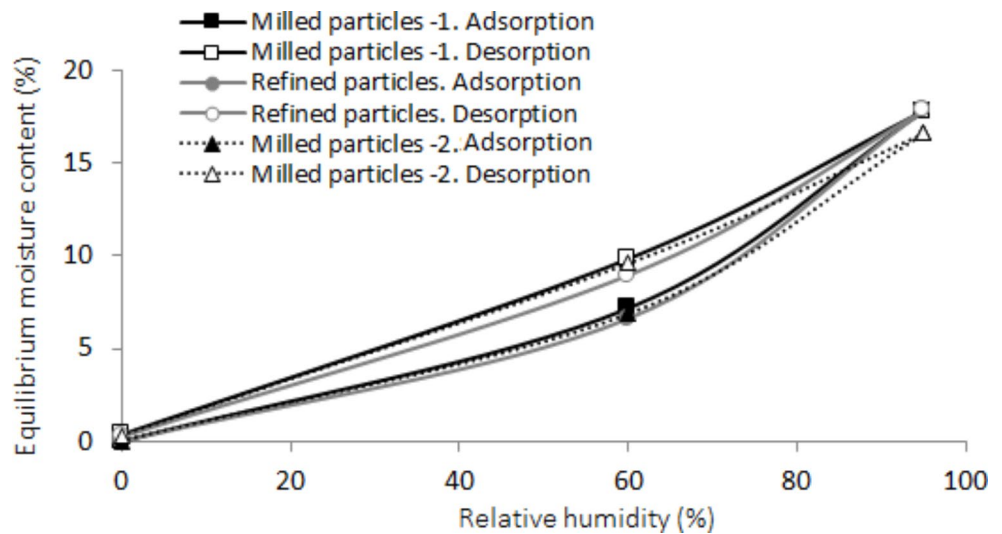


Fig. 4 Maximum stress obtained in the compression tests with regards to particle type and surfactant amount

Fig. 5 Equilibrium moisture content at 20 °C of materials produced with 8 mmol/l surfactant content and different particle types, at 20 °C



surfactant. Materials produced with refined particles present a higher mass change than materials produced with other particles, up to 455% with 10 mmol/l, whereas with the materials made with the dry milled particles the maximum water uptake reported was around 300%.

4 Discussion

Porous materials obtained with a foaming procedure and granulated bark as a raw material were successfully produced and studied.

The thermal conductivity and density results indicate a general trend of decreasing with the increase in surfactant. Correlation between porous characteristics and surfactant concentration has been described in other studies, where

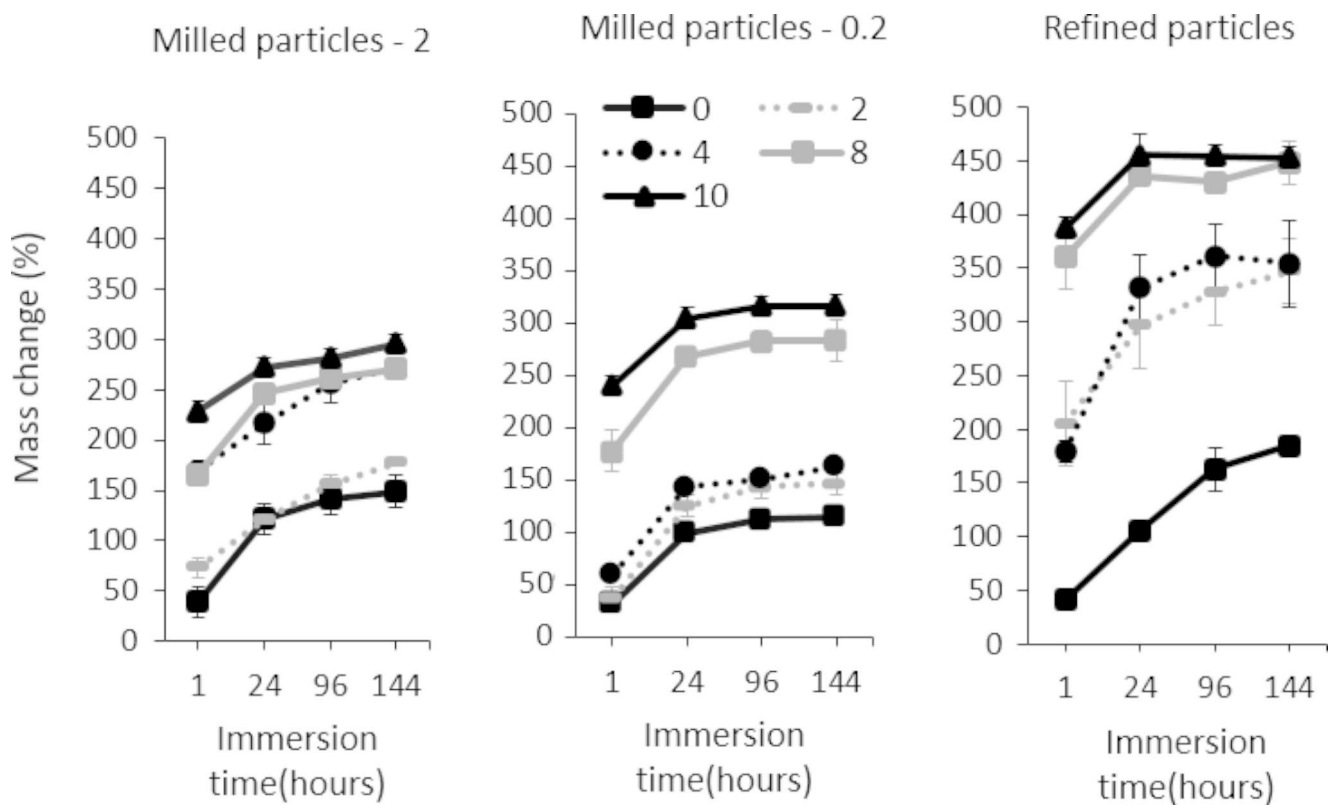


Fig. 6 Mass change after water immersion from 1 to 144 h for different particle types and surfactant contents

optimum pore characteristics were obtained with surfactant concentration close to the critical micellar concentration (Mohammad et al. 2018). Critical micellar concentration (cmc) is often used as a concentration criterion for foamability (Saint-Jalmes et al. 2005). SDS, the surfactant used in this study, has a defined cmc of 8.1–8.2 mmol/l at 25 °C. (Al-Soufi et al. 2012; Zhang and Meng 2014).

This matches well with the findings in this study, where the critical micelle concentration corresponds to the surfactant amount of 8 mmol/l. If higher values are employed, the results of density, thermal conductivity, maximum stress and change in mass due to immersion of the samples in water remain relatively constant. Lowest thermal conductivity recorded was approximately 0.06 W/mK for samples made with the refined particles and a density of around 0.2 g/cm³. Other thermal insulation materials made with bark constituent, like lignin, present similar results. Tondi et al. (2016) produced lignin-based foams, which had densities of 0.185 to 0.407 g cm⁻³ as well as thermal conductivities between 0.075 and 0.095 W m⁻¹ K⁻¹. Panels made directly with bark particles also present similar results. As an example, Kain et al. (2016) produced panels with thermal conductivities of 0.069 m⁻¹ K⁻¹ and densities of 0.25 g/cm³.

Mechanically disintegrating the raw material with the dry milling method using an ultracentrifuge (milled particles -2 and -0.2) produced particles with higher amounts of

dust, particles smaller than 0.2 mm, as well as less variation in particle size range than with the wet refining procedure studied. As a result, when producing the materials viscosity is increased and agglomerations can be formed. This has a direct impact on several properties like density, thermal conductivity and compression resistance. Higher amount of bark dust enhances the particles interaction and produces agglomeration, increasing viscosity and therefore producing denser materials. Similar behavior is observed with other lignocellulosic materials, like wood (Yang et al. 2010). In regards to the refined particles, the amount of dust is considerably reduced and even if the particle size distribution is very similar to the milled particles -2, their length to thickness ratio is higher. This particle's characteristic produces bundles of elongated particles and is something that needs to be considered when analyzing the raw material (Stark and Rowlands 2003). Because of this, particle size distribution analysis needs to be studied with other characterization methods like, for example, microscopy. Aspect ratio is an important parameter that influenced the final properties, being in some studies even more critical than particle size distribution (Stark and Rowlands 2003). However, regardless of which disintegration procedure was used, all particles and fibres show irregularity in particle shapes, which is considered a positive influence on foam and emulsion stability (Lam et al. 2014). Actually, even if these two types, i.e. the

refined and milled particles –2, have similar particle size distribution, the results of density and thermal conductivity are different, demonstrating once again that not only the bark particle size distribution influences the final materials but also their morphology, and implementing a fibrous raw material shows an advantage in relation to the characterization performed. Applying wet thermo-mechanical disintegrating procedures to lignocellulosic materials is believed to enhance the surface morphology and chemistry of the resulting fibres, and consequently the adhesion and reaction activity to form materials is improved (Gao et al. 2011), which may be the reason why the porous materials obtained with the refined fibres show better results in comparison to the other particles studied.

The stress-strain curves exhibit a linear region when using low amount of surfactant (none at all or 2 mmol/l), and in these cases, the curves present the typical shape of cellular materials, and moduli values between 10 and 30 MPa, which are comparable to other foam structures (Szczyrek et al. 2015). On the other hand, samples produced with a surfactant content higher than 2 mmol/l seem to produce too fragile materials. Even if their density and thermal conductivity are favourably lower, the mechanical properties are not comparable to other available materials. It seems that there is a turning point at which the mechanical properties start to decrease drastically, and this varies depending on the type of particle used. In the case of small particles, the limit is between 4 and 8 mmol/l and in the case of refined and medium particles, it is between 2 and 4 mmol/l. From this point where the mechanical properties decrease drastically, it may be because the surfactant content is too high, which creates a very porous, fragile and mechanically less stable structure (Gibson and Ashby 1999).

In other studies, tannin-based foams showed higher vapour sorption values, up to 41% at 90% moisture content (Jalalian et al. 2018) or in the case of tannin foams, water uptake values go up to 23% (Delgado-Sánchez et al. 2016). Bark contains a higher amount of fatty acids, compared with wood, like for example suberin, which is expected to limit moisture uptake (Heinämäki et al. 2015). This can contribute to the reduced results when comparing other foams or wood plastic composites. Regarding the biggest set of particles (milled particles –2), they show moisture uptakes similar to non-treated spruce bark (Holmberg et al. 2016), which can be explained by the big size of the particles which is up to 3.15 mm. Additionally water absorption seems to be slightly increased with the increase in the surfactant amount. This result is in accordance with the study of Tondi and Pizzi (2009), where they studied the water absorption of tannin foams and concluded that increasing the surfactant concentration can cause an increase in water absorption.

Regarding the water uptake, samples with higher porosity, like the ones produced with the refined particles and high amounts of surfactant show higher water uptake values. However, it must be taken into account that when the samples are immersed in water, a certain amount may be trapped in the open porous structure, and as a consequence give higher water uptake values. Reduction was attempted after removing the samples and draining them for 30 min, after removing them from the water and before weighing them.

5 Conclusion

In the present work, four different batches of spruce bark were used to create different porous structures. All materials were produced by a simple mechanical foaming method using SDS as a surfactant and PVAc as an adhesive. Properties of the materials vary greatly, due to the differences in characteristics, especially particle morphology and size, of the initial bark particles and fibres, which can be an advantage if the objective is to tailor natural materials.

The materials present an open heterogeneous porosity with density results that go from 0.15 g/cm³ to up to 0.54 g/cm³ and thermal conductivity values of minimum 0.06 W/mK. All materials are stable when immersed in water for longer periods, absorbing up to 300% of their mass. Compared with results reported in the literature, the mechanical properties are similar to other natural and foamy structures; however, only when using low amounts of surfactant. On the other hand, using concentrations of surfactant similar to the critical micelle concentration presented the best results in terms of density and thermal conductivity.

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Declarations

Competing interests On behalf of all authors, the corresponding author states that there is no conflict of interest.

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