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UNCONVENTIONAL HEAT INSULATION BIRCH WOOD MATERIAL MADE THROUGH DELIGNIFICATION PROCEDURE

BY

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Abstract. Wood waste delignification is a process known and used for longer time in the pulp and paper industry, whose technological adoption is necessary for producing wood foam. The new cellular wood manufacturing procedure is attractive for the production of a cheap and effective insulating material for the building construction sector. Experimental testing of several tree wood types was initiated in recent decades and the team of the current paper authors was also recently involved in this research. The current paper aimed at testing the behaviour of birch wood in foaming made by delignification of waste in the form of sawdust. The work originality results from the premiere use of birch wood in the manufacturing process of a stable foam. Utilizing wood waste, nanoclay, an

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adequate surfactant, and distilled water as well as a heat treatment at 75ºC, products with peculiarities suitable to established objective were obtained.

Keywords: birch wood, lignin, delignification, sawdust, insulation properties.

1. Introduction

The orientation of construction researchers and manufacturers towards cheaper and more environmentally friendly materials has now become a global trend. The interesting regarding the wood as a natural, ecological, and renewable material is increasing (Farid *et al*., 2021). The structure of biomaterials, including also the wood, contains lignin, cellulose, and hemicellulose, which are natural polymers whose behaviour and properties are similar to those of man-made artificial polymers. Of the three components, lignin has a three-dimensional amorphous structure with a highly branched appearance (Börcsök and Pástory, 2021). The presence of lignin in combination with cellulose and hemicellulose in the wood structure represents a bottleneck for the bioconversion of wooden material into cellulose usable in papermaking or into biofuel suitable for thermal energy production (Tribot *et al*., 2019). Obtaining the cellulosic structure of wood by complete or partial removal of lignin without damaging its initial structure is a process known as wood delignification (Kumar *et al*., 2021). The high permeability and porosity of skeletons in the wood structure create adequate opportunities for the material infiltration with various fillers and thus its densification (Farid *et al*., 2021). The wood peculiarities in terms of structure and biocompatibility are associated with state-of-the-art processes for a wide range of applications. According to the work (Farid *et al*., 2021), the preparation of hydrogels, aerogels, membranes, and wood fibres through state-of-the-art physio-chemical modifications was investigated with future modern applications aiming biomedicine, energy storage, sensors, building insulation, etc.

The industrial manufacture of pulp and paper has long applied a well-known technique based on the removal of lignin from the wood used as raw material. In technological terms, this operation of lignin removal is of major importance for the production of pulp and paper (Kumar *et al*., 2021). Typical effluent treatment processes in the pulp and paper industry are chemical precipitation, covering, activated sludge and anaerobic treatment (Uğurlu *et al*., 2008).

Given its ability to capture CO_2, satisfactory mechanical strength and durability, as well as high availability, wood is considered a very interesting biomaterial (Ding *et al*., 2022). The sawdust used in the foaming process is a by-product of the mechanical processing of wood and is available in very large quantities, at a low price, and difficult to dispose of when not in use.

The production of building insulation materials using wooden waste in an environmentally friendly and high profitable conditions has become a special concern of researchers. Wood waste panels have been made by hot pressing and assembled with vinyl and floor adhesive (Merli *et al*., 2021). The wet spraying and bulk filling without binder technique has also been experimented for obtaining thermal insulation from wooden waste (Cetiner and Shea, 2018). By utilizing high ratios of binding materials as well as low pore size wood, heat conductivity values up to 0.084 $W \cdot m^{-1} \cdot K^{-1}$ have been reached. Diminishing the heat conductivity to much lower values (0.026 $W \cdot m^{-1} \cdot K^{-1}$) was tested applying the method of forming additional small voids through a chemical treatment in accordance with (Liu and Zhao, 2022).

The technical option presented in the paper of Siciliano *et al*. (2023) was based on the delignification procedure of wood chips used to reduce the density and respectively, the heat conductivity of the material as a viable alternative to the usual heat insulation. It is known that heat conductivity is the main type of heat transfer through the solid walls of wooden cells. The most suitable conditions for hindering the circulation of heat flow are ensured by removing lignin from the neighbouring channels of the wooden material. Delignified wood chips can be suitable for partial blocking the heat transfer. Additionally, densification creates a larger surface area of the cellulose favouring the production of a stable foam. The experiment presented in (Siciliano *et al*., 2023) utilized poplar wood chips, mixed with carboxymethyl cellulose (CMC) as an adhesive binder, and deionized water. The mixture was heated to 100-150 ℃ in a hot plate stirrer for 8 hours. The density value of delignified wood significantly decreased compared to lignin-containing wood (from 0.087 to 0.053 g·cm-3). Also, heat conductivity had a much lower value (0.038 $W \cdot m^{-1} \cdot K^{-1}$), while the compression strength reached an acceptable value (1.1 MPa).

Research conducted in Finland at Aalto University (Alava and Koivisto, 2021) allowed to obtain a wood foam using in the starting mixture lignin, wood fibres, and nanoclay, completely avoiding plastics. The raw wooden material was the durable Nordic wood (such as: European oak, pine, European larch, etc.). Wood fibres as cellulosic elements recovered in the form of wood chips from the tree mass and processed at high temperatures and pressures are applied to the manufacture of materials such as paper, cardboard, absorbent paper, etc. Nanoclay (montmorillonite) in the form of extremely thin plates (about 1 nm) forming multilayer stacks of about 10 μm, which is the most commonly used nanoclay, was utilized in the Finnish university experiment. The wood foam thus produced is cheap and biodegradable, being applied mainly as packaging, but also as insulation in building construction, due to its fireproof and waterproof abilities.

Authors of the current paper have recently been concerned with the valorisation of delignified wood for the production of wood foam. In the paper (Paunescu *et al*., 2023), oak wood in the form of sawdust recovered from a processing workshop was used. The chemical procedure for treating the wooden waste used an aqueous mixture including NaOH, $Ca(OH)_2$, and distilled water. The components were mixed until a suspension was formed, which was poured into a metal mould. This was introduced into an electric oven and heated to 260°C to decompose the lignin and then slowly cooled. The applied procedure allowed to obtain several delignified oak foam specimens, of which the optimal version had a density of 0.024 $g{\cdot}cm^{-3}$, porosity of 90.5%, heat conductivity of 0.031 $W{\cdot}m^{-1}{\cdot}K^{-1}$, and compression strength of 0.9 MPa. The water uptake was within the limits of 2.2-3.1%.

The same authors of the current work have designed an original technique for producing wood foam (Paunescu *et al.*, 2024) using oak and beech wood waste as raw material, ground together in pre-wetting conditions with distilled water. Bentonite clay as a nanomaterial and calcium lignosulphonate (LSC) as a surfactant, were the additional materials chosen by the authors. The formation of a slurry including about 20% solids and 80% distilled water through stirring at 8000 rpm for 10 min, then poured into a mould and subjected to heating in an electric oven at 75°C for 8 hours, allowed obtaining a hardened wood foam. The optimal version of the wood foam had the following properties: apparent density of 0.06 $g{\cdot}cm^{-3}$, porosity of 94.1%, heat conductivity of 0.027 $W{\cdot}m^{-1}{\cdot}K^{-1}$, and compression strength of 0.985 MPa.

German specialists from the Fraunhofer Institute for Wood Research WKI have designed and experimented a new foam type made from renewable resources, fully replacing foams usually manufactured based on petroleum oil (Wood foam, 2025). The novelty of this advanced material consisted in the use of finely ground beech wood fibres, which mixed with deionized water led to the formation of a slurry. This was expanded through chemical or physical techniques, without the use of adhesives and thus avoiding the typical hazardous emissions. The porous material resulting from these techniques had an open-pore structure and, implicitly, low density in the range of 0.04-0.25 $g{\cdot}cm^{-3}$. In the next stage, the material was processed in the form of hard porous panels or as elastic foam. Generally, recent wood foam insulation materials for building applications have the disadvantage of low dimensional stability. The new product designed by the Fraunhofer Institute has adequate compressive strength for a compression of 10 % within the limits of 20-600 kPa, depending on the density level. The rather high value of heat conductivity has been reduced to under 0.04 $W{\cdot}m^{-1}{\cdot}K^{-1}$, reaching the level of polystyrene boards commonly used for building insulation.

The removal of lignin from wood pulp is the main fractionation procedure previously adopted in the pulp and paper industry. Several delignification types are applicable: alkaline, acid, catalytic reductive fractionation, etc. Alkaline techniques require temperatures within the limits of 40-210°C, the chemical activator used mainly being NaOH. According to (Paunescu *et al.*, 2023), alkaline delignification is the most frequently applied method for the purpose of lignin removal. This delignification type favours increasing lignin solubility due to the alkaline environment, which facilitates the removal of phenolic OH^- groups as well as H^+. Also, the alkaline environment favours breaking the bonds between lignin and carbohydrates and thus, the increase of lignin fragmentation and degradation process.

The current work aimed at the use in the foaming process of a new type of tree wood (birch), whose application range generally includes other preferential areas (Wang *et al*., 2012). Birch, generally widespread in Europe and Asia (including Japan), occupies large areas of forest, especially in northern Europe, in the south of the continent being identified less frequently as a mountain tree. In Romania, birch trees are found relatively isolated in mountainous areas of Transylvania. The Scandinavian countries and Russia have areas rich in birch forests, its wood being commonly used for the manufacture of plywood. In terms of physical and mechanical properties, birch wood has an average density of around 0.65 $g \cdot cm^{-3}$ and acceptable strength. The wood of this tree is easily processable by turning, profiling, and sawing. The bending properties are satisfactory. Commercially, birch wood is available as lumber, plywood, and veneer (Denomme and Perone, 2024).

Utilizing the lignin removal technique to obtain a cellular wooden material with insulating properties suitable for its application in construction works, under conditions of choosing a type of tree not commonly used for preparing the birch foam.

2. Methods and Materials

The main technique applied for producing an advanced material with remarkable thermal insulation features (very low apparent density and reduced heat conductivity) through the delignification procedure of birch wood, tried for the first time, consists in the chemical treatment of this biomaterial in an aqueous environment.

Research carried out by (Askvik *et al*., 2001) has shown that the association between a lignosulfonate, which is a water-soluble anionic polyelectrolyte polymer, and a cationic surfactant has the ability to create a complex phase gel, in the case of concentrations close to charge equilibrium between lignosulfonate and surfactant, favouring dispersion from the aqueous phase into the oil phase. The type and concentration of surfactant have major importance by adsorption through hydrophobic interactions to the lignosulfonate-surfactant combination. The delignification process in sulfite pulping takes place through acid cleavage of several bonds that include components of lignin.

Sodium lignosulfonate $C_{20}H_{24}Na_2O_{10}S_2$ (Sodium lignosulfonate, 2025) as the chosen surfactant was added over the ground wood at particle size under 90 μm in very low ratios, reducing the surface tension of the slurry and promoting the froth formation. Also, bentonite clay as a nanoclay was introduced into the wet mixture containing distilled water. The solid components of the mixture were subjected to mechanical stirring for homogenization. The aqueous slurry concentration did not exceed 20%. Through stirring at 7500 rpm for 10 min in an electrically driven equipment, the obtained foam volumetrically increased. The slowing down and stopping this growing process indicated the need to stop the stirring operation. The stabilized wet foam was poured into a metal mould and the material was dried in a laboratory electric oven at 75°C for 8 hours. At the end of this process, the hardened wood foam was performed.

The materials chosen to compose the base mixture for the manufacture of wood foam were birch wood in the form of sawdust, bentonite clay as a nanoclay, and sodium lignosulfonate as a surfactant.

The sawdust birch waste was finely ground in a ball mill. The selection of the particle sizes was performed by sieving and their values were less than 90 μm.

Bentonite clay (Bentonite clay, 2023; Amankeldi *et al.*, 2022) is a natural clay material as a fine powder, containing natural minerals such as calcium, iron, and magnesium. Stabilization of aqueous foams with solid particles such as bentonite clay presents a special interest in some processes including the manufacture of wood foam. Reduction of coalescence and gas diffusion as well as improvement of foam lifetime are important effects of using bentonite clay.

Surfactants are well-known as substances with fluidizing role of the mix prepared for manufacturing foams. Their influence consists in realization of adsorption on the surface of fine solid particles, facilitating hydration, dispersion, and the formation of a structure with higher resistance. In industry, the most well-known and used surfactants are lignosulfonates coming from the recovery of by-products and therefore being quite cost-effective. Sodium lignosulfonate resulting as a secondary product of the cellulose manufacturing process and available on the market as a fine powder was chosen by authors for the current experiment (Ouyang *et al*., 2006).

Table 1
Composition of experimental versions

Composition	Version 1	Version 2	Version 3	Version 4
Sawdust birch wood (g) (wt. % in solids)	 48.73 97.45	 48.30 96.59	 47.86 95.72	 47.43 94.87
Bentonite clay (g) (wt. % in solids)	 1.20 2.40	 1.60 3.20	 2.00 4.00	 2.40 4.80
Sodium lignosulfonate (g) (wt. % in solids)	 0.074 0.15	0.105 0.21	 0.136 0.27	 0.167 0.33
Distilled water (g)	 200	 200	 200	 200
Wet slurry (g)	 250	 250	 250	 250

Four experimental variants for making the birch wood foam were tested using the composition mentioned above. The component amounts tried in each version are shown in Table 1. The weight proportion of birch wood waste varied in a very high value range (between 94.87-97.45%). Bentonite clay represented between 2.4-4.8 wt. %, while sodium lignosulfonate had very low weight percentages (between 0.15-0.33%). The weight ratio between added distilled water (kept constant at 200 g) and the wood waste varied in the limits 4.10-4.217.

The investigation methods for determining the specimen features were the following. Archimedes' principle was the procedure applied for measuring the apparent density of wood foam specimens. Using ASTM C642-97, the apparent porosity was identified by dividing the difference between wet and dry mass by the difference between wet mass and suspended mass of the sample. Heat conductivity was identified at room temperature using the HFM448 Lambda heat-flow-meter (SR EN 1946-3:2004). The measurement of compression strength was performed using a universal testing machine. The specimens were compressed at 1.3 $mm \cdot min^{-1}$ (ASTM D695) and the compression strength was determined at 10% compression (ASTM D1621-16). The microstructural appearance of foams could be analyzed with the Biological Microscope MT5000 model. Water uptake was measured by maintaining wood foam samples in a humidity room at 85% humidity for 30 days according to ASTM C272/C272M-18.

3. Results and Discussion

The solid material in the wet mixture was about 50 g in all four versions, representing 20%. The addition of distilled water constituted the remainder, i.e. 80%. Theoretically, the wet slurry resulting from mixing was approximately 250 g, in accordance with Table 1.

The main results of investigating features of the four wood foam specimens (shown in Fig. 1) are presented in Table 2.

According to experimental results shown in Table 2, with the increase of sodium lignosulfonate from 0.074 to 0.167 g, the thermal insulation properties of cellular wood specimens consistently improved. Apparent density decreased from 0.14 $g \cdot cm^{-3}$ (in the case of sample A) to 0.07 $g \cdot cm^{-3}$ (in the case of sample D), simultaneously with heat conductivity, whose value decreased from 0.040 $W \cdot m^{-1} \cdot K^{-1}$ (sample A) to 0.027 $W \cdot m^{-1} \cdot K^{-1}$ (sample D). Also, apparent porosity remained at extremely high values in all the tried tests (over 90%), increasing from 90.2% (sample A) to 94.0% (sample D). The compression strength values have indicated a normal low level considering the excessively low porosity of investigated specimens. The lowest value of the strength has corresponded to sample A (805 kPa) and the highest was reached in the case of sample D (922 kPa). Water uptake was within the usual value range of previously manufactured cellular woods (between 4-5.3 wt. %).

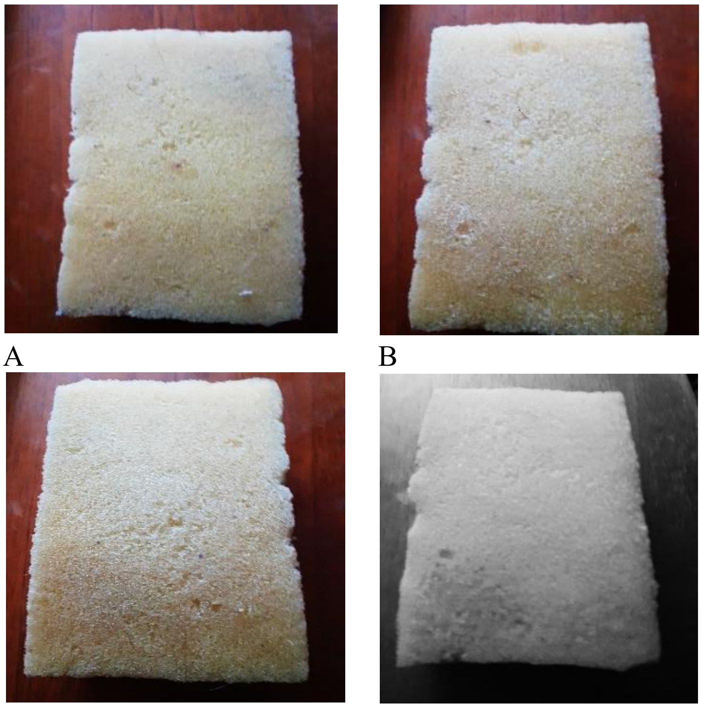

Fig. 1 – Surface images of wood foam specimens
A – version 1; B – version 2; C – version 3; D – version 4.

Table 2
Features of birch wood foam samples

Feature	Sample A	Sample B	Sample C	Sample D
Apparent density ($g \cdot cm^{-3}$)	0.14	0.10	0.08	0.07
Apparent porosity (%)	90.2	92.4	93.6	94.0
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.040	0.031	0.028	0.027
Compression strength (kPa)	805	818	895	922
Water uptake (wt. %)	5.3	4.8	4.0	4.3
Pore size (μm)	15-30	35-85	35-95	40-110

The investigation of the microstructural appearance of the wood foam specimens was carried out by analyzing the pictures (Fig. 2) corresponding to microstructures of experimental samples. The obtained cells are closed and uniformly distributed, especially in the case of the first two samples (A and B). The closed cell type is also preserved in the case of the following samples (C and D), only the uniformity of the microstructural network is very slightly affected, especially in the case of the last sample.

The cell sizes of the four specimens are indicated in Table 2, being determined the following value ranges: 15-30 μm (sample A), 35-85 μm (sample B), 35-95 μm (sample C), and 40-110 μm (sample D).

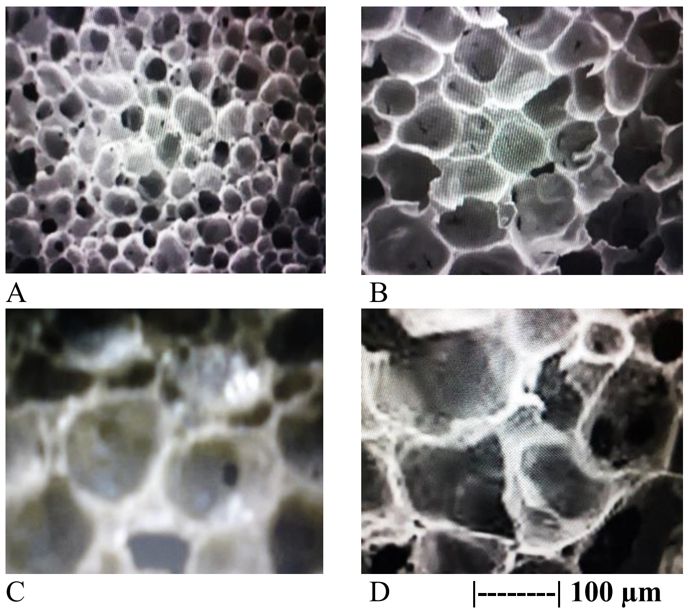

Fig. 2 – Microstructural appearance of wood foam specimens
A – version 1; B – version 2; C – version 3; D – version 4.

Although the technique of producing cellular wood by delignification of tree wood species has recently begun to attract the interest of researchers in insulating materials field for construction, the first results have already been reported in the literature. Several tree wood types have been experimentally tested: beech, poplar, oak, etc. The most famous centre for wood research, the German Fraunhofer Institute, used beech wood fibres without added adhesives and managed to obtain a high-performance insulating material with open-pore structure, lowering its minimum density to 0.04 g·cm^{-3}, under conditions of a compression strength of only 20 kPa. Of course, other specimens of beech foam with a semi-open or even closed pore structure have been produced, thus increasing the density (up to 0.25 g·cm^{-3}) and compressive strength (up to 600 kPa) of the wood foam. Research at the German institute is ongoing and new high-performance products are expected. On the other hand, other research teams in the world, including the authors of the current paper, are investigating various other types of wood subjected to foaming.

The optimal sample D made in experimental version 4 of the current work, prepared with highest proportions of sodium lignosulfonate as a surfactant and respectively, bentonite clay as a nanomaterial, led to very low values of apparent density (0.07 g·cm^{-3}) and heat conductivity (0.027 W·m^{-1}·K^{-1}), the compression strength reaching the acceptable value of 900 kPa. These results obtained using birch wood waste as a wooden material not utilized until now for producing wood foam are comparable with those made in the case of poplar wood (Siciliano *et al*., 2023), of oak wood foam (Paunescu *et al*., 2023), and of the foam produced using oak and beech wood combination (Paunescu *et al*., 2024).

4. Conclusions

The work aimed at the experimental production of a cellular wood by delignification of birch wood. This species of tree wood was chosen for the first time to test its behaviour in the making process of the naturally porous, environmentally friendly, and highly cost-effective material. The adopted recipe consisted of mixing at 7500 rpm wood waste in the form of finely ground sawdust, together with sodium lignosulfonate as a surfactant, and bentonite clay as a nanomaterial as well as distilled water. The mixing process was completed after the formation of a wet slurry, which was subsequently dried by heating at 75°C for 8 hours in an electric laboratory oven. The optimal product manufactured by adopting the solid mixture with the highest proportions of sodium lignosulfonate and bentonite clay was a porous material with 94% apparent porosity, apparent density of 0.07 g·cm^{-3}, heat conductivity of 0.027 W·m^{-1}·K^{-1}, and compression strength of 922 kPa.

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MATERIAL TERMOIZOLATOR NECONVENȚIONAL DIN LEMN DE MESTEACĂN, REALIZAT PRIN PROCEDEUL DELIGNIFICĂRII

(Rezumat)

Noul procedeu de fabricare a lemnului celular este atractiv pentru producerea unui material izolator ieftin și eficace pentru sectorul construcțiilor. Testarea experimentală a mai multor tipuri de lemn de copac a fost inițiată în ultimele decenii, iar echipa autorilor prezentei lucrări a fost și ea implicată recent în această cercetare. Prezenta lucrare a avut ca scop testarea comportamentului lemnului de mesteacăn la spumarea realizată prin delignificarea deşeurilor sub formă de rumeguş. Originalitatea lucrării rezultă din utilizarea în premieră a lemnului de mesteacăn în procesul de fabricare a unei spume stabile. Utilizând deșeu de lemn, nanoargilă, un surfactant potrivit și apă distilată, precum și un tratament termic la 75°C, s-au obținut produse cu particularități adecvate obiectivului stabilit.