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CHEMICAL METHOD OF BIOMASS DELIGNIFICATION AS AN ADVANCED TECHNIQUE FOR PREPARING WOOD FOAM WITH INSULATING PROPERTIES

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Abstract. The paper refers to a method of lignin removing in the biomass structure to obtain a wooden material with excellent heat-insulating properties suitable for use as an insulating material in construction. In principle, the method is almost similar to that applied in the pulp and paper industry, but with a different technological purpose. The chemical activation of maple wood waste was obtained through contact with an aqueous alkaline environment containing NaOH and Ca(OH)₂ the effect being significant reduction of thermal conductivity compared to non-chemically treated wood. The originality is the use for the first time of maple wood as recycled sawdust. The process temperature was 90°C for 8 hours. Results were remarkable, the optimal product having density of 0.21 g·cm⁻³ and heat conductivity of 0.029 W·m⁻¹·K⁻¹. In quality terms, the delignified wood features were almost similar with other foamed wooden products.

Keywords: maple wood, removing lignin, biomass, alkaline environment, thermal conductivity.

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1. Introduction

As a result of activities in the fields of agriculture, forestry, aquaculture, fishing, and also storing biodegradable waste (coming from industry and municipal activities), the conditions for the formation of biomass were naturally created (Diaz-Montaño, 2022). Usually, biomass has a bad influence on the environment health (especially soil and water). However, due to its properties, it is considered as a potential source for the production of materials with newly created value, interesting for various industrial activities. This interest exhibited a sharp increase in the last decades. On the other hand, the material capitalization of biomass has a major contribution to lowering the greenhouse gases (CO₂) emission into the atmosphere by limiting landfills for their storage (Tripathi *et al.*, 2019). The wide availability of biomass in nature is also remarkable.

An important amount of biomass originates from the forestry industry. The waste resulting from forestry treatment enters the first biomass group, while the residues resulting from the timber industry are considered in the literature (Diaz-Montaño, 2022) as part of the secondary biomass group. Both groups including this waste provide residual biomass. As a particular case, the sawdust coming from the timber industry constituted the field analyzed in this paper.

According to data from the literature (Wood composition, 2022), the wood structure includes cellulose, lignin, hemicellulose as well as very low amounts of organic impurities and water. Cellulose is a polymer that can crystallize forming high strength fibers. Lignin is also a polymer, but in an amorphous state and plays the role of binder or matrix. It is characterized by a highly branched three-dimensional structure. Hemicellulose is a partially crystalline polymer, that also has the ability to act as a binder or matrix. According to (Börcsök and Pásztor, 2021), compared to the other two main components of wood, lignin has a special behaviour by softening as a result of moistening and heating around 100°C, that leads to deforming the molecules in the cell walls of the wood.

The wood microstructure includes long cells (in the axial direction) and small cells (in the radial direction). The formations of cellulosic chains (known as microfibrils), initially covered by hemicellulose and then by lignin, represent the structural components initially discovered by the research teams. The long cells of wood are made up of several layers of differently oriented microfibrils. This multi-directional orientation of the layers contributes to the improvement of the wood resistance in several directions, keeping the anisotropic character.

In the last decades, it was found that lignin, like other derivatives containing lignin, constitute a very valuable source of new products as well as bioenergy, lignin being considered as the second most abundant polymer in the world (Yadav *et al.*, 2022). The pulp and paper industry constitutes a special source of by-products (such as aromatic compounds, resin acids, fatty acids, carboxylic acids, chlorinated compounds, etc.), that for their own technological

reasons must be removed. Different physical, chemical, thermal, and biological techniques were used following this goal.

The process of removing the lignin without affecting the wood structure is known as wood delignification. According to (Kumar *et al.*, 2021), numerous wood types were tested in this technological process: balsa, bamboo, basswood, birch, Chinese fir, Norway spruce, hybrid poplar, poplar wood, poplar wood flour.

Among the known techniques capable of removing lignin and improving the heat-insulating properties of biomass, the authors' interest was focused on chemical methods. According to the literature (Diaz-Montaña, 2022), the most effective processes of this type are alkaline hydrolysis and acid hydrolysis, the alkaline hydrolysis being adopted for this experiment. The usable agents in this hydrolysis type have proven to be sodium hydroxide (NaOH), ammonia (NH₃), calcium oxide (CaO), and calcium hydroxide (Ca(OH)₂), while the optimal process temperature range is between 50-90°C.

The originality of the current work was due to the choice of maple wood as the wood material subjected to the lignin removal tests. This tree has a good spreading in Europe over large areas between Greece and the Ural Mountains in Russia, including also Romania and is suitable for large-scale using as wooden furniture.

The literature presents several recent works related to the production of materials with heat-insulating properties by removing lignin. (Liu and Zhao, 2022) obtained a wooden product with very small pores, whose heat conductivity reached the minimum value of 0.026 W·m⁻¹·K⁻¹.

Starting from the idea that heat conductivity is the main way of thermal transfer through the solid walls of wood, the method of removing lignin from its common structure is the optimal solution for obtaining suitable porous features for significantly improving the thermal insulation properties of wood. According to (Siciliano *et al.*, 2022), by applying the delignification process, additionally, the extension of cellulose surface in the wood mass is obtained, leading to the formation of a stable foam. The authors of the mentioned paper found a significant decrease of the wood density subjected to delignification up to 0.053 g·cm⁻³ compared to the density value of the reference sample (0.087 g·cm⁻³). Also, heat conductivity had low values (about 0.038 W·m⁻¹·K⁻¹). The compressive strength decreased accordingly, but the value reached remained at a satisfactory level for a heat-insulating product applicable in construction. The raw wood material used by Siciliano's team was poplar wood chips, with the addition of carboxymethyl cellulose (CMC) as an adhesive binder, and distilled water. The temperature adopted for the heat treatment of the mixture was in the range of 100-150°C for 8 hours.

The German Fraunhofer Institute recently presented in the literature (Wood foam, 2021) a high-performance foamed wooden material. The technique for making it involved the use of a mixture of finely ground beech wood and

distilled water in the form of suspension. The procedure required chemical and physical methods, without adding adhesives. A porous material with open pore structure was obtained, the density dropping to 0.04-0.25 g·cm⁻³. Next, the material was processed, resulting in hard expanded panels or elastic froth. The physical stability of the final products was significantly increased compared to common wooden heat-insulating materials. Heat conductivity had values below 0.04 W·m⁻¹·K⁻¹, while compression resistance was within the limits of 20-600 kPa. Product features were thus comparable in value with those of polystyrene insulation panels existing on the market.

Improving the feasibility of wood as a thermal insulation material was achieved by treating some wood species with low density to obtain wood foams and was hydrophobically modified with polylactic acid (Jin *et al.*, 2023). Only a delignification treatment without hemicellulose removal ensured a better and more economical thermal insulation effect. After delignification, the thermal conductivity of balsa wood decreased from 0.068 W·m⁻¹·K⁻¹ to 0.052 W·m⁻¹·K⁻¹, while paulownia wood decreased from 0.092 W·m⁻¹·K⁻¹ to 0.066 W·m⁻¹·K⁻¹. By using the polylactic acid film on the surface of the delignified wood specimens, the hydrophobicity of the sample was improved and the water absorption was greatly reduced.

As mentioned above, the alkaline hydrolysis processes used in delignification are the most often applied to remove lignin from biomass. Alkaline environment has the ability to greatly increase the solubility of lignin due to the removal of a proton (e.g. H⁺) or phenolic OH groups. In this way, in the alkaline environment, lignin/carbohydrate bonds are broken, contributing to the fragmentation, destruction, and elimination of lignin.

The current paper adopted the wood treatment method in alkaline environment already applied in the industrial making process of pulp and paper. The mix was prepared from NaOH, Ca(OH)₂, and distilled water, while the wood species chosen for the first time was maple wood. A recent previous experiment carried out by the authors' team of this work (Paunescu *et al.*, 2023) used oak wood in the form of sawdust, recycled from the processing of wooden furniture and subjected to the delignification process in alkaline environment consisting of the same mentioned components in close proportions. The characteristics of the new product in the optimal version were excellent for a heat-insulating material (density of 0.024 g·cm⁻³, heat conductivity of 0.031 W·m⁻¹·K⁻¹, porosity of 90.5%, and compression strength of 0.9 MPa).

2. Materials and Methods

As mentioned above, maple wood was chosen as the wooden material in this experiment. The cellular wall structure and the composition are the main peculiarities of wood that influence its physical and chemical properties. According to (Novaes *et al.*, 2010), usually 25% lignin and 70% cellulosic

carbohydrates (including 45% cellulose and 25% hemicellulose) Romanian wood-working workshop. The recycled waste was finely ground in a ball mill to particle size less than 85 μm .

The alkaline environment was created by mixing commercially available NaOH in the form of solid pellets soluble in water and $\text{Ca}(\text{OH})_2$ in the form of fine powder also soluble in water. By mixing the two solid products in distilled water, a suspension is obtained that constitutes the required alkaline environment. The NaOH solution concentration was experimentally adopted at the value of $88 \text{ g}\cdot\text{L}^{-1}$. Theoretically, $\text{Ca}(\text{OH})_2$ has the role of forming more homogeneous and form the wood structure. The recycled wood was recovered in the form of sawdust from small finer porous structures.

The experiment was performed by testing three experimental variants (noted V1, V2, and V3) of producing porous wood material by applying the delignification process, compared with the reference variant of non-treated wood (noted R).

The composition of experimental variants adopted for this test is presented in Table 1.

Table 1
Composition of experimental variants

Composition	V1 (g/wt. %)	V2 (g/wt. %)	V3 (g/wt. %)
Maple wood	238/66.1	224/62.2	210/58.3
NaOH	88/24.4	88/24.4	88/24.4
$\text{Ca}(\text{OH})_2$	34/9.4	48/13.3	62/17.2
Total	360/100	360/100	360/100
Water addition	1000/-	1000/-	1000/-

According to the data in Table 1, it was started from the experimentally adopted value of the NaOH solution concentration of 88 g per 1000 g of distilled water, maintaining constant the total solid mixture at 360 g, and varying the amount of $\text{Ca}(\text{OH})_2$ between 34–62 g. Thus, the amount of maple wood was reduced between V1-V3 variants from 238 to 210 g.

The method of making a porous material with remarkable heat-insulating properties (extremely low values of density and heat conductivity and very high porosity) through the contact of a recycled biomass with an alkaline environment is basically a method recently applied in general in manufacturing processes of new materials from recycled waste, the alkaline environment acting as an activator of chemical processes. Probably, the most eloquent example in this direction is the method for obtaining geopolymers, developed by the French inventor J. Davidovits at the end of the last century and in the first decades of the new millennium (Davidovits *et al.*, 1994).

Of course, the method of biomass delignification has its specific peculiarity, but the effect of alkaline environment in the structural transformations of wood waste is largely similar.

The well-known using the delignification method in the pulp and paper industry has a different technological purpose than that of forming a cellular structure suitable for thermal insulation aimed in the current work and in other similar works in the literature. However, both purposes refer to the removal of lignin from the biomass structure.

Therefore, the chemical method of biomass delignification is not in itself an original method, but is a typical case of taking over a technique successfully used in a certain activity field, in another relatively related field.

Preparing the mixture including biomass and alkaline environment respects the basic rules of this preparation type. Mixing the liquid materials (NaOH, Ca(OH)₂, and distilled water) is separately done in a glass vessel and the finely ground biomass is also separately homogenized. Then, the liquid mixture is poured over the solid and mixing by mechanical stirring is carried out until the suspension is formed. This is loaded into a rectangular mould (80 x 120 mm) with a wall height of 60 mm. The adopted thermal treatment temperature of the fresh material, obtained into a laboratory electric oven (Fig. 1), was 90°C (Law *et al.*, 2022) for 8 hours.



Fig. 1 – Image of the laboratory electric oven.

The method of removing lignin from biomass favours the creation of a porous structure characterized by numerous small pores and implicitly, the increase of the total volume of voids in the wood structure. This structural change allows the density and the heat conductivity to drop greatly, because the air inside the voids is a poor conductor of heat flow.

According to (Kumar *et al.*, 2021), lignin is naturally generated through deposition of free internal secretions into the spaces existing between cellulose microfibrils in cellular walls of the living plant. Forming process of lignin leads thus to heterogeneous macromolecules generation.

The methods for investigating the characteristics of samples experimentally made were those shown below. Based on the literature recommendation (Density, 2014), Archimedes' method (ASTM D792-20) was adopted for measuring the density and porosity. The heat-flow method (ASTM E1225-04) (Yüksel, 2016) was utilized to determine the heat conductivity values and the compression strength level was identified with the TA.XTplus C Texture analyzer. The water-absorbing of specimens was determined through measuring the water absorbed in 24 hours by the wooden material immersed under water (ASTM D7433-19). The use of Biological Microscope MT5000 model, 1000 x magnification, allowed the investigation of the microstructural peculiarities of specimens.

3. Results and Discussion

The investigating methods of physical, thermal, and mechanical characteristics of expanded delignified maple specimens (V1-V3) mentioned above as well as of the reference non-treated maple sample (R) allowed obtained the results shown in Table 2.

Table 2
Characteristics of foamed delignified maple and reference wood sample

Characteristic	R	V1	V2	V3
Density ($\text{g}\cdot\text{cm}^{-3}$)	0.731	0.090	0.052	0.021
Porosity (%)	15.0	81.8	87.7	91.2
Heat conductivity ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$)	0.302	0.046	0.036	0.029
Compression strength (MPa)	3.8	1.9	1.7	1.6
Water-absorbing (vol. %)	10.2	2.9	2.3	1.5

Result presented in Table 2 show remarkable heat-insulating properties of delignified maple wood in experimental variants V1-V3, compared to those of the same wood material type non-treated for lignin removal (noted R). Thus, compared to the density level of the reference material ($0.731 \text{ g}\cdot\text{cm}^{-3}$), the chemical treatment applied to the delignified wood variants allowed a significant decrease in density to $0.090 \text{ g}\cdot\text{cm}^{-3}$ (V1), $0.052 \text{ g}\cdot\text{cm}^{-3}$ (V2), and $0.021 \text{ g}\cdot\text{cm}^{-3}$ (V3). The density values of delignified products are generally lower than those required in applications with traditional heat-insulating materials in building construction. The density of polyurethane insulation as rigid expanded board is in the range of $0.03\text{-}0.08 \text{ g}\cdot\text{cm}^{-3}$ (Anh and Pásztor 2021). The heat conductivity values obtained in case of delignified specimens are very low (between $0.029\text{-}0.046 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) and ensure an excellent thermal transfer compared to the heat conductivity value of the same non-treated wood sample ($0.302 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$). Obviously, there is a huge difference between porosity values of delignified wood

specimens (81.8-91.2%) and the reference value of porosity (15%). As expected, the strength of the wood material without lignin content in its structure significantly decreased from 3.8 MPa (in the case of non-treated wood) to 1.6-1.9 MPa (in the case of the experimental specimens), but these compression resistance values are satisfactory for a thermal insulation material intended for building construction. Instead, the water absorption capacity greatly decreased from 10.2 vol. % to only 1.5-2.9 vol. %.

The physical appearance of delignified maple wood specimens (V1-V3 experimental variants) compared to the appearance of the reference specimen of non-treated wood (R specimen) are shown in Fig. 2.

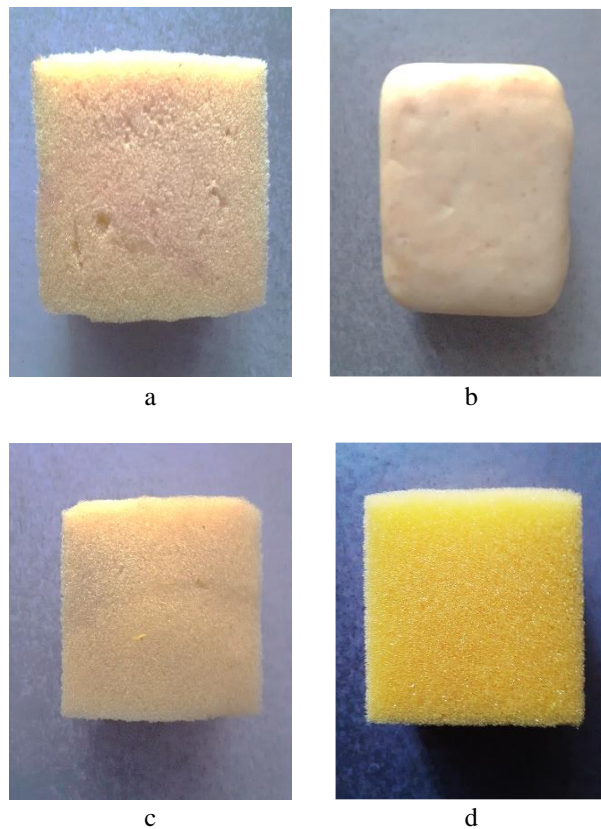


Fig. 2 – Physical aspect of delignified maple wood specimens (V1-V3) compared to the aspect of non-treated wood
a – reference specimen R; b – V1 specimen; c – V2 specimen;
d – V3 specimen.

According to images in Fig. 2, the surface of the cross sections of maple wood samples subjected to delignification (V1-V3) is much finer and more homogeneous compared to the surface of the non-treated reference sample (R).

The change in the microstructural appearance of specimens is much more visible in the pictures in Fig. 3, representing the microstructural investigation of the four samples including also the reference sample.

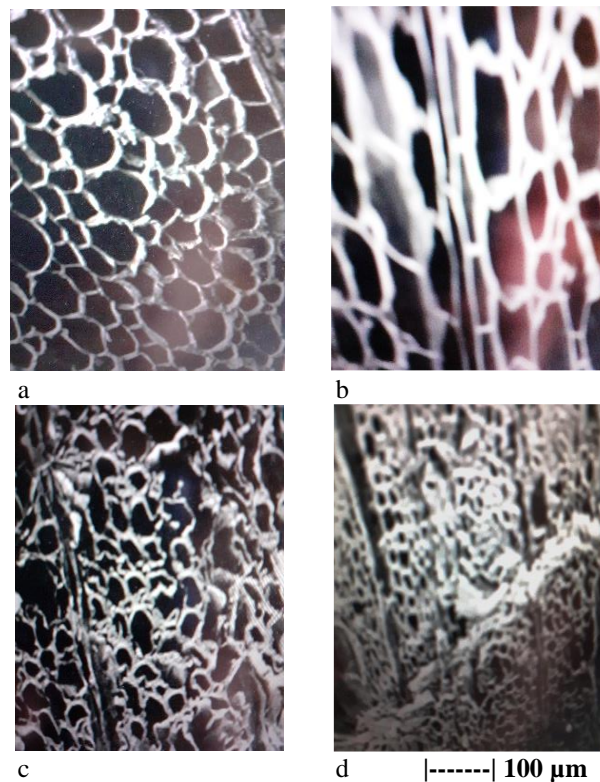


Fig. 3 – Microstructural appearance of specimens
a – reference specimen R; b – V1 specimen; c – V2 specimen;
d – V3 specimen.

The proportion of the alkaline environment components (NaOH and $\text{Ca}(\text{OH})_2$) used in the experiment for the chemical treatment of the structure of maple wood specimens played a major role in the removing process of lignin and forming the fine cellular structure of the wood. The decrease of the cell size and the increase of their degree of crowding between the vertical channels through which water and nutrients flow are more and more evident starting from the V1 specimen (b) to the V3 specimen (d).

The theme developed in this work can generate discussions. In the current global conditions in which energy consumption has become a major problem, the thermal insulation of the building is an important factor in the energy saving trend. The conservation of primary energy and its consumption reduction have focused the concern of researchers on new energy sources, but also on new efficient heat-insulating material types. This paper aims to research obtaining a material with high thermal insulation properties from a biomass through the procedure of removing the lignin from its structure in order to favour the significant decrease of the thermal conductivity through the new structural organization of the used wood.

The work is focused on a wood type not used until now in this delignification process. Although the biomass delignification is a technological procedure known and applied in the pulp and paper industry, but for other purposes than that targeted in this case, its utilization for the structural modification of wood to obtain fine porosity and very low thermal conductivity structure constitutes only an expansion of the basic principle of delignification technique, the whole process being original. Particularly, the originality of the current work is the choice for the first time of the type of used wood (maple).

According to (Jin *et al.*, 2023), in the case where lignin and hemicellulose are selectively removed from the wood structure, an expanded wood prepared in this way has an anisotropic structure, self-supporting resistance, excellent heat-insulating properties, and low costs. The expanded material has heat-insulating of $0.03 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ in the cross direction and $0.06 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ along the cellulose direction.

Among the three experimental tested variants, in which the majority proportion of maple wood waste was contained within the limits of 58.3-66.1 wt. % according to Table 1. The components of the alkaline environment (NaOH and $\text{Ca}(\text{OH})_2$) had their ratio values between 1.42-2.60, the highest value corresponding to the V1 variant. The temperature of the chemical treatment was 90°C for 8 hours. Under these conditions, the characteristics of all delignified maple wood samples in terms of heat-insulating properties were remarkable compared to the non-treated reference sample. The density had values in the range of $0.021\text{-}0.090 \text{ g}\cdot\text{cm}^{-3}$ compared to $0.731 \text{ g}\cdot\text{cm}^{-3}$ corresponding to the reference variant, heat conductivity was between $0.029\text{-}0.046 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ compared to $0.302 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, and porosity was within the limits of 81.8-91.2 % compared to 15.0%. The superiority of chemically treated wood sample properties than those of the non-treated reference sample is as obvious as possible. The optimal specimen of this experiment was considered that obtained through V3 variant, having the following features: density of $0.21 \text{ g}\cdot\text{cm}^{-3}$, heat conductivity of $0.029 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, porosity of 91.2%, compression strength of 1.6 MPa, and water-absorbing of 1.5 vol. %.

The features comparison of delignified maple wood with other recently reported performance results (between 2021-2023) showed that the level reached

by the optimal product presented in this article is relatively similar in terms of quality to those in the international literature.

High performances were reported by Fraunhofer Institute (Wood foam, 2021), the heat conductivity being of $0.04 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and the wood foam density range between $0.04\text{-}0.25 \text{ g}\cdot\text{cm}^{-3}$. Normally, the compression strength value was low ($0.02\text{-}0.6 \text{ MPa}$), even extremely low (0.02 MPa or 20 kPa) corresponding to the minimal density of the product. However, such a material with exceptional thermal insulation properties is only suitable for very lightweight thermal insulation.

Results of making a wooden material intended for thermal insulation in construction using poplar wood chips in a chemical treatment in alkaline environment at $100\text{-}150^\circ\text{C}$ is reported by (Siciliano *et al.*, 2022), having the density of $0.053 \text{ g}\cdot\text{cm}^{-3}$ and heat conductivity of $0.038 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$.

We mentioned above excellent thermal conductivity performances obtained by (Jin *et al.*, 2023) in the case of delignification processes of balsa wood ($0.052 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) and paulownia wood ($0.066 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$).

Remarkable performances were obtained by the authors' team of the present paper (Paunescu *et al.*, 2023) using oak wood waste as sawdust and the chemical treatment in aqueous alkaline environment (NaOH and $\text{Ca}(\text{OH})_2$) at a temperature of 260°C recommended by (Le Floch *et al.*, 2015): density of $0.024 \text{ g}\cdot\text{cm}^{-3}$, heat conductivity of $0.031 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, porosity of 90.5% , and compression strength of 0.9 MPa . Also, in the paper (Paunescu *et al.*, 2024) in process of publication, the same type of wooden material was tested for the production of a heat-insulating material for construction. The objective of the research was to remove plastics from the composition of the manufacturing mixture by preparing the wet suspension using ground wood, SDS surfactant and distilled water, the foaming of the suspension being obtained by stirring followed by drying at 80°C . The foam density was extremely low and the thermal conductivity lowered up to $0.035 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$.

4. Conclusions

The objective of the present work is to test applying the delignification process of a biomass by chemical treatment in aqueous alkaline environment of maple wood waste for the first time. Biomass delignification refers to the normal structural reorganization of wood through the formation of a woody mass with very small pores crowded between the channels through which water and wood nutrients flow. In this way, a homogeneous porous structure is generated, that contributes to reducing the thermal conductivity as well as the density of the new material, which becomes suitable for use as a thermal insulator in building construction. The delignification process, already applied in the pulp and paper industry, but for other technological purposes, has recently been taken over in processes of transforming wood rich in lignin into a product with excellent

thermal insulation properties. Several types of wood have been successfully tested in the world. Maple wood waste as recycled sawdust was the wooden material subjected to delignification in this work. The alkaline environment used by the authors' team was composed of NaOH, Ca(OH)₂, and water, i.e. materials usually utilized in the delignification process, but in slightly modified weight proportions. The process temperature was 90°C. Results were excellent, the optimal product having the following properties: density of 0.21 g·cm⁻³, heat conductivity of 0.029 W·m⁻¹·K⁻¹, porosity of 91.2%, compression strength of 1.6 MPa, and water-absorbing of 1.5 vol. %. The features comparison of delignified maple wood with other recently reported performance results showed that the level reached by the optimal product presented in this article is relatively similar in terms of quality to those in the world literature.

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METODĂ CHIMICĂ DE DELIGNIFICARE A BIOMASEI CA O TEHNICĂ
AVANSATĂ PENTRU PRODUCEREA SPUMEI DE LEMN CU PROPRIETĂȚI
IZOLATOARE

(Rezumat)

Lucrarea se referă la o metodă de îndepărtare a ligninei din structura biomasei pentru a obține un material lemnos cu proprietăți excelente de izolare termică, adecvat pentru utilizarea ca material izolator în construcții. În principiu, metoda este aproape

similară cu aceea aplicată în industria celulozei și hârtiei, dar cu un scop tehnologic diferit. Activarea chimică a deșeurilor de lemn de arțar a fost obținută prin contactul cu un mediu apos alcalin conținând NaOH și Ca(OH)₂, efectul fiind reducerea semnificativă a conductivității termice comparativ cu lemnul netratat chimic. Originalitatea este utilizarea pentru prima dată a lemnului de arțar sub formă de rumeguș reciclat. Temperatura procesului a fost de 90° timp de 8 ore. Rezultatele au fost remarcabile, produsul optim având o densitate de 0,21 g·cm⁻³ și o conductivitate termică de 0,029 W·m⁻¹·K⁻¹. Din punct de vedere calitativ, caracteristicile lemnului delignificat au fost aproape similare cu alte produse lemnoase spumate.