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The potential of utilisation of birch bark suberinic acid residues as a component of the adhesive mass for bonding of wood composites

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Abstract: *The potential of utilisation of birch bark suberinic acid residues as a component of the adhesive mass for bonding of wood composites*. The aim of the investigation was to determine the effect of the different amounts of suberinic acid residues (SAR) (2%, 5%, 10%, 15%, 20%, 50%) introduced as a hardener to the adhesive mass based on urea-formaldehyde (UF) resin on the mechanical and physical properties of manufactured solid birch wood bonded samples. The produced samples were compared to samples bonded with industrial UF resin without the addition of SAR. The scope of the work was to determine the shear strength, the share of in-wood damage, the contact angle and the surface free energy (SFE) for wood and suberinic acid (SAR). The results show that increasing from 2-5% of SAR to 20% of SAR in the adhesive mass is not justified as there are no statistically significant differences between the mean values of shear strength for these four variants (2SAR, 5SAR, 10SAR, 20SAR).

Keywords: bark, suberinic acid, residue, solid wood, birch, shear strength, contact angle, surface free energy

INTRODUCTION

In recent years, thanks to the popularisation of ecological awareness and environmental regulations, there has been a growing interest in wood-based composites produced with the use of alternative lignocellulosic materials and natural adhesives as binders. Recyclable composites based on raw materials of natural origin are produced from both renewable and waste sources.

One of the alternatives is the use of by-products from the agricultural industry, such as sugarcane bagasse, kenaf stalks (Juliana et al., 2012), cotton stalks (Zhou et al., 2010; Fahmy and Mobarak 2013), sunflower residue (de Carvalho et al., 2020; Mahieu et al., 2019), rice husk (César et al., 2017), rice straws and coir fibres (Zhang and Hu, 2014). Agricultural residues are largely sourced and processed where they are locally available.

The second alternative is forest by-products – achievable in millions of tonnes per year – such as cones, tree bark and branches, which are mainly treated as waste from production in sawmills, paper and the wood-based panels industry (Aydin et al., 2017). Biomass residues from poplar plywood production make up over 54% of the raw material (Ferretti, 2021). Depending on where the given post-process residue comes from, it has some characteristic features such as moisture content, chemical composition, calorific value and price. The above features decide and set the direction of subsequent use, increasing the efficiency of the system. Production residues can be burned to generate thermal energy, sold on the market or returned to the production process. The efficient management of tree bark is one of the important problems the wood industry faces. It is estimated that 10-15% of the volume of each log consists of bark, depending on the species of wood from which it was obtained (Nemli et al., 2006; Busquets-Ferrer et al., 2021). In 2019, the potential total annual amount of tree bark was approximately 397,000,000 m^3 (FAO, 2021). The bark, since it is the outer layer of tree trunks, in the case of its chemical composition, is similar to wood (lignin, cellulose and hemicellulose). It protects the wood against biotic and abiotic factors (rain, snow, hail, heat, frost, ultraviolet radiation [UV], insects, fungi, bacteria, parasitic plants, animals, water loss and mechanical damage) (Giannotas et al., 2021). The bark consists of two layers: the outer, so-called "cork necrosis" that gives a protective and insulating function; and the inner layer, living, responsible for transporting nutrients. The tree bark contains, among others, ash and phenolic substances and is rich in extractive substances that cause a dark colour; therefore, this is an aesthetically undesirable feature for the pulp and paper industries. Extraction of biologically active compounds from tree bark can be advantageous for producing a natural binder based on tannin (Hoong et al., 2011; Da Silva Araujo et al., 2021) and based on suberinic acids (Tupciauskas et al., 2019). The outer birch bark (*Betula pendula*) contains up to 45% (dry weight) of the suberin (Tupciauskas et al., 2019).

Many researchers have confirmed the possibility of using bark in composites made of bark particles or adding bark particles to the wood particle, producing – among others – insulation panels and particleboards (Pedieu et al., 2008; Efe, 2022; Kain et al., 2020; Casas-Ledón et al., 2020; Mirski et al., 2022). Currently, the well-known uses for tree bark are: energetic and biogas (Janzon et al., 2014; Rasaeian et al., 2022), extraction of compost and soil mulching; sorbent (e.g. petroleum product sorbent, odour sorbent) (Jansone et al., 2017). Chen and Yan (2018) conducted research on the use of the Western red cedar (*Thuja plicata*) tree bark as a functional filler in pMDI wood adhesives.

This study was designed to determine the effect of the different amounts of SAR (ranging from 2% to 50% by weight) introduced as a hardener to the adhesive mass based on UF resin on the selected mechanical and physical properties of manufactured solid birch wood bonded samples. The produced bonded samples were compared to those bonded with industrial UF resin without adding SAR. The scope of the work was to determine the shear strength, the share of in-wood damage, the contact angle and the surface free energy (SFE) for wood and suberinic acid (SAR).

MATERIALS AND METHODS

Materials Characterisation

The bonded samples were produced under laboratory conditions from air-dry birch (*Betula pendula*) lamellas of the dimension of 110 x 22 x 7 mm^3. Industrial UF resin (Silekol S-123, Silekol Sp. z o.o., Kędzierzyn-Koźle, Poland) was used as a binder for all manufactured samples. For the production of the bonded samples were used the birch (Betula spp.) outer bark extraction residues, tissues containing suberin (hereinafter referred to as SAR – suberin acid residues). The dry mass content in SAR was around 25% (according to EN 827:2005). The SAR acidic paste-like was used as a hardener for urea-formaldehyde resin (UF). Birch bark suberinic acid residues (SAR) were obtained from the Latvian State Institute of Wood Chemistry (LSIWC) (Riga, Latvia).

Preparation of adhesive masses

Five variants of adhesive mass were produced with different SAR content (0% - reference, 2%, 5%, 10%, 20%, 50%; hereinafter referred to as UF, 2SAR, 5SAR, 10SAR, 20SAR, 50SAR). Industrial UF resin (Silekol S-123) was used as a binder base for all manufactured bonded samples. The hardener of the UF adhesive mass was a 10% aqueous solution of ammonium sulphate $(NH_4)_2SO_4$ in a weight ratio of 50: 15: 1.5 (resin: water: hardener water solution).

Bonding of the lamellas

All variants of the produced adhesives were applied in excess to two entire wide surfaces of the birch lamellas. Then, the two lamellas were manually connected and placed between the steel plates and then put into the press without any spacer bars. Under the influence of the pressure exerted, the excess adhesives were removed, resulting in a thin bonding line. The pressing process was carried out on a single-shelf press at a temperature of 180°C, under 1 MPa unit pressure, and with a pressing time of 10 min.

Shear strength testing and in-wood damage evaluation

The shear strength of the manufactured samples was measured on a standard universal testing machine Heckert FP 10, where the samples were loaded by tension to be broken within 60±30 s, and the maximum load [N] was registered. The samples were cut before testing (according to EN 205:2016 standard) to reach the nominal loaded bonded area of about 220 mm^2. Before the loading, the real dimensions of the bonding line were measured by a digital calliper with a precision of 0.01 mm. The shear strength was calculated as a maximum load [N], referred to as the loaded bonding line area [mm^2]. After the break of the sample, every destroyed zone was analysed if the break occurred the in-wood structure, and the area of in-wood destruction was estimated in an organoleptic way (in % of total loaded bonding line area) with an accuracy of ±10%. For the tests, no less than 20 samples of the bonded sample of each binder variant were used.

Contact angle and surface free energy (SFE)

Contact angle measurements were made using the contact angle analyser PHOENIX 300 (SEO – Surface & Electro Optics Co. Ltd., Korea) equipment using the distilled water sessile drop method. The contact angle was measured for the dry SAR surface, *Betula pendula* solid wood and cured UF resin.

The SFE of solid wood and SAR was determined by the surface energy calculator available in the contact angle analyser by the Owens Wendt method, which is based on the results of the contact angle after 60 s using two liquids with known surface tension values (Żenkiewicz, 2000, Rogowska, 2013, Xu et al., 2022). The components of polar and dispersive measuring liquids are summarised in

Table 1. The contact angle of the surface of the wood, UF and SAR was determined in three repetitions for both measuring liquids.

Table 1. Values of free surface energy and its components for measurement liquids used in the Owens-Wendt method taken from literature

Liquid	Surface free energy (mJ/m^2)	Dispersive component (mJ/m^2)	Polar component (mJ/m^2)
Distilled water	72.8	21.8	51
Diiodomethane	50.8	50.8	0

All of the tested samples were conditioned before the tests in 20°C/65% relative humidity (RH) to achieve constant weight.

Based on the above-mentioned measurements, the average values (mean) of tested parameters and the standard deviation (SD) of these were calculated and displayed on graphs. The ANOVA analyses were completed to confirm the presence of statistically significant differences between the mean values of achieved results (wherever available).

RESULTS and DISCUSSION

Shear strength testing and in-wood damage evaluation

The results of the shear strength measurement of birch lamellas bonded with the use of UF resin and the addition of SAR as a component of adhesive mass are presented in Figure 1.

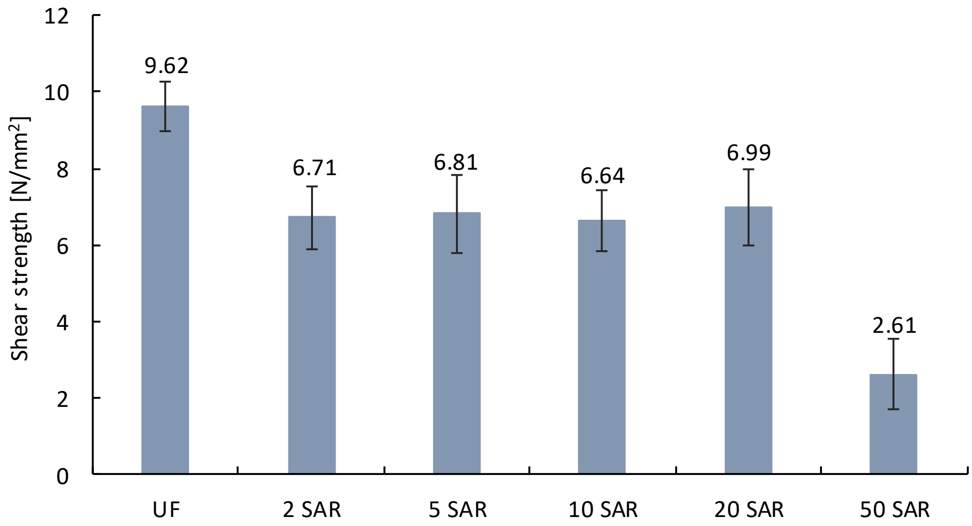

Figure 1. Shear strength of tested samples

The highest average value of shear strength is 9.62 N/mm^2 for reference samples, while the lowest strength value is 2.61 N/mm^2 – obtained for samples with 50% SAR as a hardener. Considering the values of standard deviations, it can be noted that there are statistically significant differences between the average values of the shear strength for reference samples and the samples with SAR addition (from 2% to 20% in the adhesive mass and 50SAR samples). The mean values of the shear strength for the 2SAR, 5SAR, 10SAR and 20SAR variants are comparable. There are no statistically significant differences between them, but the addition of 50% SAR causes a significant decrease in strength compared to the 20SAR samples (63%). The results show that increasing from 2-5% SAR to 20% SAR in the adhesive mass is not justified. A similar dependence is the case of a hydrophobic agent added in wood-based materials. Xu et al. (2009) manufactured bagasse particleboards (BPBs) with polymeric methylene diphenyl diisocyanate (pMDI) resin as a binder and wax as a dimension stabiliser. The use of wax significantly reduced the water absorption (WA) and thickness swelling (TS) after 24 hours of soaking in water compared to the wax-free reference panels. They proved that the wax amount has a less significant effect on the WA and TS than pMDI resin. On the other hand, some positive effects on the Modulus of Rupture (MOR) were observed after introducing 1% wax, and the obtained MOR value increased by 60%. However, no statistically significant differences were observed with a further increase in the amount of wax. The investigations described here confirmed the possibility of obtaining a bonding line with favourable results of the shear strength value with the utilisation of suberinic acid from outer birch bark as a component of the adhesive mass.

The above statement confirms the results of the in-wood damage evaluation, which are presented in pictures in

Figure *2*.

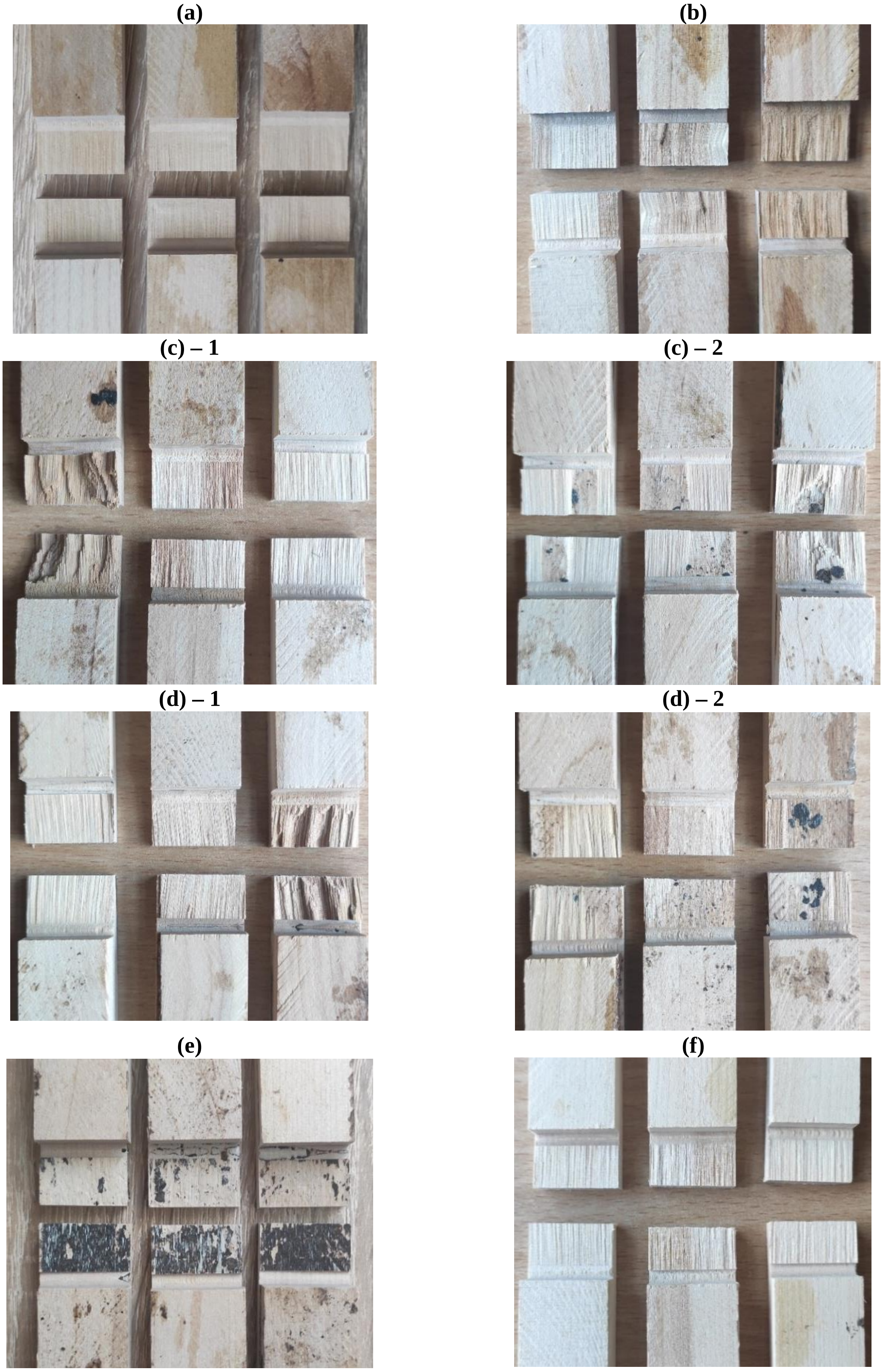

Figure 2. Examples of break zone of tested samples bonded with 2SAR (a), 5SAR (b), 10SAR (c – 1,2), 20SAR (d – 1,2), 50SAR (e) and UF (f).

In-wood damage evaluation results for 2SAR, 5SAR and UF samples were 100%, meaning there was interfacial adhesion between the bonding material and the wood substrate. Increasing the amount of SAR in the UF resin resulted in a lower share of damage in wood.

Figure **2** shows, among others, 10SAR samples (c – 1) and 20SAR samples (d – 1) where in-wood damage is 100%; but for this, both variants were first noted as partially damaged in the bonding line (c – 2 and d – 2). Partial damage in wood occurred for 20% of all tested samples. For the weakest variant of the samples, 50SAR, the damage in wood was 0-10%; some samples fell apart during the test.

Contact angle and SFE

Figure 3 presents the results of the contact angle for solid wood, SAR and UF resin. The contact angle method provides information about SFE in terms of the capacity of the lignocellulose material to absorb liquids or act as a barrier for liquids. The highest contact angle measured with water was recorded for the SAR surface 82° (1 s) and 64° (60 s), the lowest for reference bonding mass (UF resin-based) 58° (1 s) and wood surface 29° (60 s). It is worth adding that after 60 s of the water droplets remaining on the tested surface, the contact angle decreased. These changes were greatest for the birch wood surface (61% reduction) and SAR surface (by 22%). The smallest changes in contact angle after 60 s were found for UF resin (by 5%). The results for the surface of birch and SAR measured with diiodomethane show the same dependence – the greatest changes in the contact angle after 60 s were found for the birch wood surface and the smallest for UF-resin.

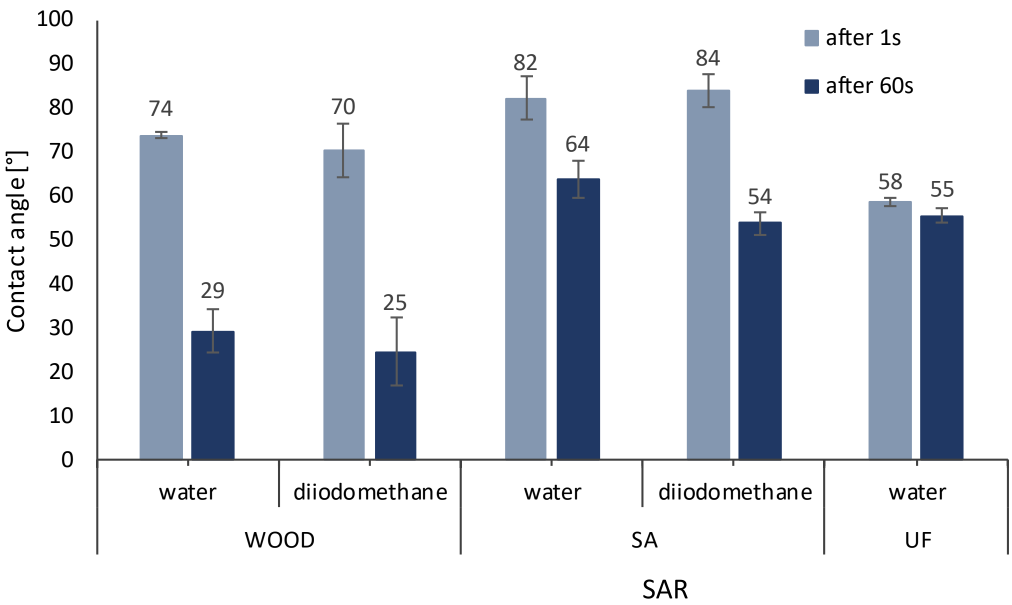

Figure 3. The contact angle of the tested surfaces

The values of the contact angle and the SFE of the given materials allow us to determine their wettability. SFE is a physical phenomenon caused by intermolecular interactions at the interface. A higher value of SFE means a surface of better wettability, and a lower value to a non-wettable surface (Jirkovec et al., 2021, Jothi Prakash and Prasanth, 2021). Table 2 summarises the values calculated based on the measurements of the contact angles after 60 s of the SFE according to the Owens-Wendt method.

The birch wood surface (72.1 mJ/m^2) has a higher SFE than SAR (45.7 mJ/m^2). The share of the dispersion component in the total SFE value is greater for both the tested surfaces.

Table 2. The surface free energy calculated according to contact angles (water and diiodomethane) after 60 s for the surface of wood and SAR

Samples	Surface free energy (mJ/m^2)		Dispersive component (mJ/m^2)	Polar component (mJ/m^2)
	Average	Standard deviation		
WOOD	72.1	2.27	46.1	26
SAR	45.7	2.79	32.2	13.5

Increasing the SAR content in the adhesive mass resulted in a weakening of the bond line and a reduction in the share of in-wood damage, shown in the photos of samples after the shear strength test

Figure 2. The solid content in SAR is 25%. Increasing the addition of SAR in the adhesive mass, the amount of water and compounds that evaporate was also increased. During the pressing of bonded samples, compounds in the adhesive mass evaporate or partially penetrate the lamellas, increasing their humidity simultaneously. Benkreif et al. (2021) investigated the effect of moisture content on the contact angle and SFE on the surface of birch wood (*Betula pendula*). The SFE gives information about the wettability of the surface and the adhesion of adhesives or coatings, for example. The researchers mentioned above proved that with increasing moisture content, the SFE of the birch wood surface decreases. Decreasing the SFE value means decreasing the wettability of the surface, too. Therefore, it can be concluded that adding more SAR to the adhesive mass contributes to an increase in the moisture content of the birch lamellas, which translates into a weaker bonding line. The moisture (water) added with SAR to create a bonding line can act as a moisturising agent for wood surfaces covered by glue mass. However, after conditioning the samples when cured, the moisture content of the samples decreases but, during the first period (i.e., after spreading the glue mass over the wood samples), the additional moisture can decrease the final bonding quality.

CONCLUSIONS

According to the conducted research and the analysis of the achieved results, the following conclusions and remarks can be drawn:

1. The highest average value of shear strength was for reference samples bonded UF resin without the addition of SAR, while the lowest strength value was obtained for samples with 50% of SAR – taking into account all variants of produced birch lamellas.
2. The share of in-wood damage for UF, 2SAR and 5SAR samples was 100%, whereas for at least 80% of the 10SAR and 20SAR samples, it was 100%.
3. There is small or no in-wood damage of samples bonded with UF with 50% of SAR, which can be the reason for the lowest bonding line strength among all variants.
4. Concerning the achieved results of shear strength and in-wood damage evaluation, it can be concluded that the increase of SAR content from 2-5% to 20% seems unjustifiable.

5. Increasing the SAR in the adhesive mass contributes to an increase in the moisture content of the birch lamellas, which can translate into a weaker bonding line.
6. Changes in contact angle after 60 s were highest for the birch wood surface, whereas the smallest changes were found for UF resin.

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Streszczenie: *Możliwości wykorzystania pozostałości kwasu suberynowego z kory brzozowej jako składnika masy adhezyjnej do łączenia kompozytów drzewnych*. Celem badań było określenie wpływu różnej ilości pozostałości poekstrakcyjnych kwasu suberynowego (SAR – Suberinic Acid Residues) (2%, 5%, 10%, 15%, 20%, 50%)

wprowadzanej, jako utwardzacz do masy klejowej na bazie żywicy UF, na wybrane właściwości mechaniczne i fizyczne wytwarzanych sklejonych próbek z litego drewna brzozowego. Wytworzone próbki porównano z próbkami łączonymi przemysłową żywicą UF bez dodatku SAR. Zakres badań obejmował wyznaczenie wartości wytrzymałości na ścinanie, określenie udziału uszkodzeń w drewnie, pomiar kąta zwilżania i swobodnej energii powierzchniowej dla drewna i SAR. Badania wykazały, że zwiększanie ilości SAR w masie klejowej z 2-5% do 20% nie jest uzasadnione, ponieważ nie zaobserwowano statystycznie istotnych różnic pomiędzy średnimi wartościami wytrzymałości na ścinanie dla tych czterech wariantów (2SAR, 5SAR, 10SAR, 20SAR). Udział SAR 50% powoduje istotny spadek wytrzymałości spoiny na ścinanie przez rozciąganie.

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