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THE EFFECT OF A PHENOL-FORMALDEHYDE ADHESIVE REINFORCEMENT WITH NANOCELLULOSE ON THE PRESSING PARAMETERS OF PLYWOOD

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ABSTRACT

Research on improving the reactivity of phenol-formaldehyde (PF) resin and the possibility of lowering the pressing parameters of wood-based materials manufactured with its participation are still progressing. Due to a number of favorable properties, nanocellulose (NCC) is gaining more and more popularity as a modifier of wood adhesives. Therefore, the objective of the study was to assess the possible reduction of plywood pressing parameters due to the reinforcement of PF resin with NCC. Based on the bonding quality results it was found that there is a possibility to reduce pressing time by 25% and pressing temperature by 7%. Moreover, the outcomes of mechanical properties (modulus of elasticity and bending strength) of manufactured plywood indicate that theoretically it could be possible to decrease the pressing parameters even more. However, the shear strength of the glue joints was considered to be a limiting factor for further reduction. The results of delamination test show that plywood bonded with phenolic resin have no tendency to delaminate. Thus, it can be concluded that NCC can be used as a modifier for PF resin which can contribute to the reduction of pressing time and pressing temperature during the plywood manufacturing process.

KEYWORDS: Nanocellulose, plywood, pressing time, pressing temperature, phenol-formaldehyde resin.

INTRODUCTION

Plywood is a valuable wood-based material which has been developed as an alternative for solid wood products. There are many features of manufactured veneer-based panels which make their industrial production one the fastest growing among all the available wood-based materials (particleboard, fiberboard and oriented strand boards) (Bekhta et al. 2018, 2020a). The high mechanical strength, the increased dimensional stability and the availability in larger sizes are listed as the examples among their favorable properties and they are the actual reason for plywood's wide range of applications. Some of the industry branches such as the structural materials or garden furniture assume the use in outdoor conditions which requires a proper binding agent (Kawalerczyk et al. 2019a). In this case, the phenol-formaldehyde resins (PF) receive much attention. They are thermosetting resins which have been used in wood gluing for over 100 years. The wide range of favorable properties, such as chemical, heat and water resistance, dimensional stability, durability and the ability to infiltrate the wood cell walls, make the PF adhesives particularly popular for plywood manufacturing (Atta-Obeng et al. 2013, El Mansouri et al. 2018, Liang et al. 2011). However, the studies aimed to improve the properties of PF adhesives are still progressing.

The possibility of lowering the pressing parameters in the production of wood-based materials is the interesting trend in research regarding the modification of PF resins. Both the pressing time and pressing temperature are of great importance due to the crucial effect on the properties of manufactured panels. Moreover, the hot pressing process is also important from the economic standpoint since it is one of the most energy-consuming operation in the whole plywood manufacturing process (Bekhta et al. 2020b). The effect of steam injection during pressing on the potential increase in the rate of plywood heat transfer was investigated. However, the major increase in the core layer temperature during steam-assisted pressing which was previously noted for particleboard, was not observed in the plywood manufacturing (Jokerst and Geimer 1994). What is interesting, studies have also shown that modification of veneers can significantly influence the pressing parameters. The engaging concept of veneer incising before entering the dryer has been developed in 1986 by Forintek Canada Corp (Dai et al. 2003). On the basis of the outcomes it can be concluded that incising the veneer was effective in case of plywood and led to the reduction of pressing time by up to 13% (Kurowska et al. 2011). However, no positive effect of incisions on the laminated veneer lumber (LVL) pressing parameters was determined (Wang et al. 2003). Furthermore, wood densification has been used for years to improve its properties such as mechanical strength, surface quality, hardness, durability and dimensional stability (Bekhta et al. 2017, 2014). Research have shown that implementing veneer densification prior to plywood pressing can increase the heat transfer and consequently reduce the total pressing time by 12-25% (Bekhta and Salca 2018, Kurowska et al. 2010). There are also studies which show that the modification of adhesives may have a positive effect on the plywood pressing parameters. Sedliačik et al. (2010) investigated the influence of PF resin reinforcement with various chemicals (melamine, resorcinol, urea, hydrogen peroxide, para-formaldehyde, dichromate ammonium, potassium and sodium). It was found that the addition of these substances allowed to significantly increase the reactivity of adhesives and reduce the pressing temperature from 120-130°C to 100°C. Furthermore, Mirski et al. (2011) applied the alcohol- and ester-modified PF resins in plywood production and investigated the effect on pressing parameters. It was shown that the introduced modifiers enhanced resin curing and allowed to significantly decrease both pressing temperature and pressing time. Plywood production process requires a precise control of resin viscosity. It can be achieved by adding a proper amount of fillers which, as shown by the outcomes, can also contribute to the reduction in pressing parameters. For example, the replacement of technical flour with the ground bark particles resulted in the acceleration of both resin curing and heat transfer which in consequence led to reduction in pressing time by up to 27% (Mirski et al. 2020, Réh et al. 2019, Ružiak et al. 2017).

Due to the increasing environmental awareness, the nanocellulose (NCC) has attracted the attention of scientists in recent years. It is the most abundant polymer occurring in the biosphere which is characterized by the unique properties, such as low density, high energy conversion capacity, exceptional mechanical strength, tremendous surface area and chemical compatibility (Abitbol et al. 2016, Kawalerczyk et al. 2021, Sokołowska et al. 2008, Zimmermann et al. 2004). NCC was also investigated as the additive for PF resin in plywood manufacturing. Studies have shown that the incorporation of nano-sized cellulose led to the shortening of adhesive curing, the improvement in morphology of bond lines and the increase in mechanical properties of the resultant plywood (Kawalerczyk et al. 2020, Lengowski et al. 2020). Therefore, taking into account the reinforcing nature of nanocellulose, the aim of presented study was to determine the possibility to reduce plywood pressing parameters by the introduction of NCC to the PF adhesive.

MATERIALS AND METHODS

Materials

In order to perform the experiments, the commercially available PF resin was purchased from the market (Silekol, Poland). It was characterized by the following properties summarized in Tab. 1. The experimental formulations assumed the incorporation of two types of fillers in different proportions. The first one was the nanocellulose (NCC) obtained from Nanografi Laboratory (Ankara, Turkey) with a trade name NG01NC0101-1000 having the average particle wide of 10-20 nm and length of 300-900 nm as declared by the producer. The second one was added in order to adjust the viscosity and it was the commercially applied tannin filler for phenolic resins labeled as UT-10, containing mimosa tannins and chalk. The plywood panels were produced using the rotary cut birch veneer with the dimensions of 320 × 320 mm, thickness of 1.3 ± 0.2 mm and moisture content (MC) of 5%.

Tab. 1: Properties of PF resin.

Property	Unit	Value
Viscosity	$mPa \cdot s$	933
Density	$g \cdot cm^{-3}$	1.213
Solids content	%	52
Gel time at 130°C	s	193

Preparation of adhesive mixtures and gel time measurements

The quantity of each components was based on the outcomes of previous study (Kawalerczyk et al. 2020). The nanocellulose-reinforced variants were compared to the reference one which was prepared according to industrial formulation. The composition of applied mixtures are shown in Tab. 2. The adhesives filled with a proper amounts of additives were mixed with the CAT-500 homogenizer for 90 seconds at 1000 rpm until the assumed homogenization level was achieved. The measurements of gel time at 130°C were carried out in triplicates following the guidelines of the relevant Polish standard PN-C-98352-3 (1996).

Tab. 2: Compositions of the adhesive mixtures.

Variant label	**Quantity (pbw per 100 g of PF resin solids)**	
	UT-10	**NCC**
REF	20	-
NC3	15	3
NC5	15	5

Note: pbw - parts by weight

Plywood manufacturing and testing

The prepared adhesives were applied on the surfaces of outer veneers in the amount of 160 $g \cdot m^{-2}$. In order to assess the possibility to decrease the pressing time and temperature, the methodology proposed by Mirski et al. (2011) was implemented. Pressing process was conducted with the unit pressure of 1.4 $N \cdot mm^{-2}$. The following pressing conditions were applied: temperature of 115, 120, 125, 130, 135 and 140°C and the time of 150, 180, 210, 240 s. For each variant four three-layered plywood panels were manufactured. Then, they were conditioned prior to testing for 7 days at 65% relative humidity and 20°C. After conditioning, the obtained panels were tested in terms of bonding quality. It was investigated in accordance with EN 314-1 (2004) both after soaking in water at 20°C for 24 h and after pre-treatment consisting of: boiling in water for 4 h, followed by drying in laboratory oven at 60°C for 18 h, boiling in water again for 4h and cooling in water at 20°C for 1 h. In addition, the extreme variants (temperature of 115/135°C and time of 150/240 s) were tested in terms of their mechanical properties such as modulus of elasticity and bending strength according to EN 310 (1993) perpendicularly to the direction of fibers in the outer veneer layers. Moreover, their delamination was also determined according to ANSI/HPVA HP-1 (2004) provided by The Hardwood Plywood and Veneer Association. The detailed description of the applied method can be found in the recent work of Taghiyari et al. (2020) evaluating the properties of wollastonite-treated plywood.

In order to analyse the results, the multivariate statistical analysis ANOVA was performed. Furthermore, in order to distinguish the homogeneous groups, the Tukey test on the significance level of $\alpha = 0.05$ was carried out with the use of Statistica 13.0 software.

RESULTS AND DISCUSSION

Gel time of the adhesive mixture is one of the most important parameter investigated in order to control the quality of adhesive in the industrial conditions. Furthermore, it is a common technical indicator of curing process and the phase transition from the liquid to solid state when the resin is no longer able to be molded (Gonçalves et al. 2018, Laza et al. 2008, Srebrenkoska et al. 2009). The results of gel time measurements are shown in Tab. 3.

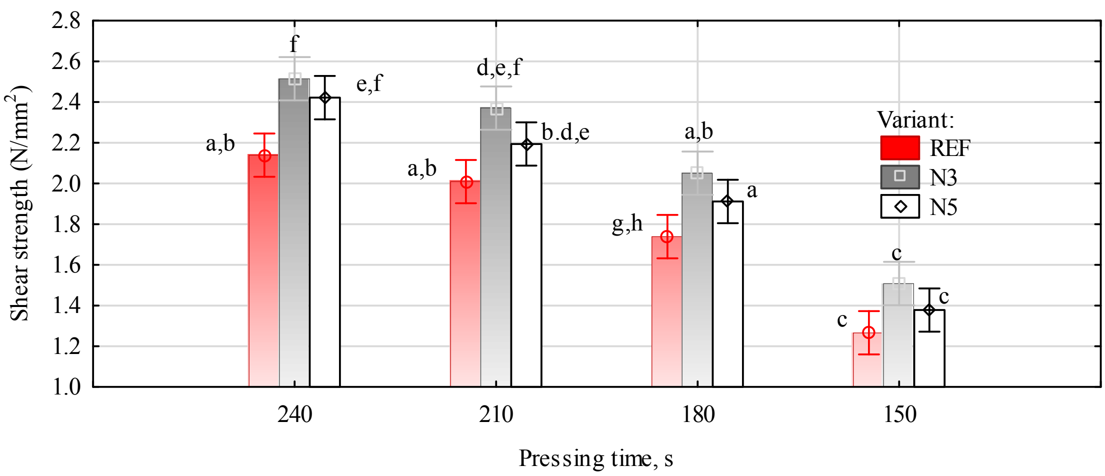
Tab. 3: The results of gel time measurements.

Variant label	REF	NC3	NC5
Gel time (s)	216 ± 3[a]	202 ± 2[b]	194 ± 4[b]

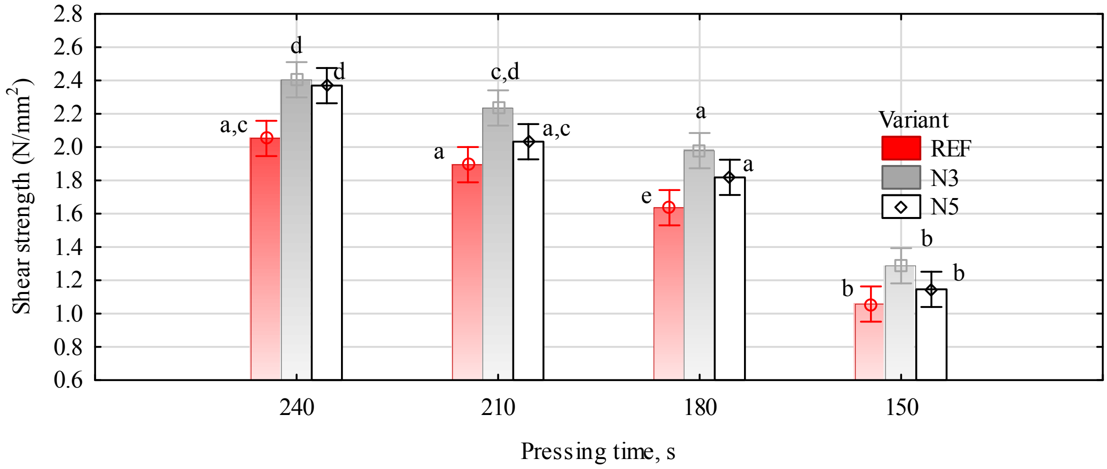
Note: mean value ± standard deviation; a,b letters indicate the homogeneous groups distinguished with Tukey test.

The outcomes revealed that the addition of nanocellulose led to acceleration of resin curing time. The differences between the NCC-reinforced variants and reference one were statistically significant. The increase in the amount of introduced nano-sized cellulose led to a slight reduction in curing time, however, the difference was not significant from the statistical standpoint. The accelerated curing can result from the highly reactive and hydrophilic nature of nanocellulose as indicated by the results of previously investigated viscosity changes (Kawalerczyk et al. 2020). The obtained outcomes are consistent with the observations of Lengowski et al. (2020) which concluded that NCC-enhanced mixture was characterized by curing time reduced by up to 33%.

According to Hong and Park (2017) the process-related parameters such as pressing time and temperature strongly influence bonding strength of plywood. Thus, in order to investigate the effect of decrease in pressing time and temperature on the properties of plywood, the shear strength test was conducted. It is a fundamental indicator of the adhesive performance in the resultant plywood (Bekhta et al. 2016, Kawalerczyk et al. 2019b). The results are presented in Fig. 1.



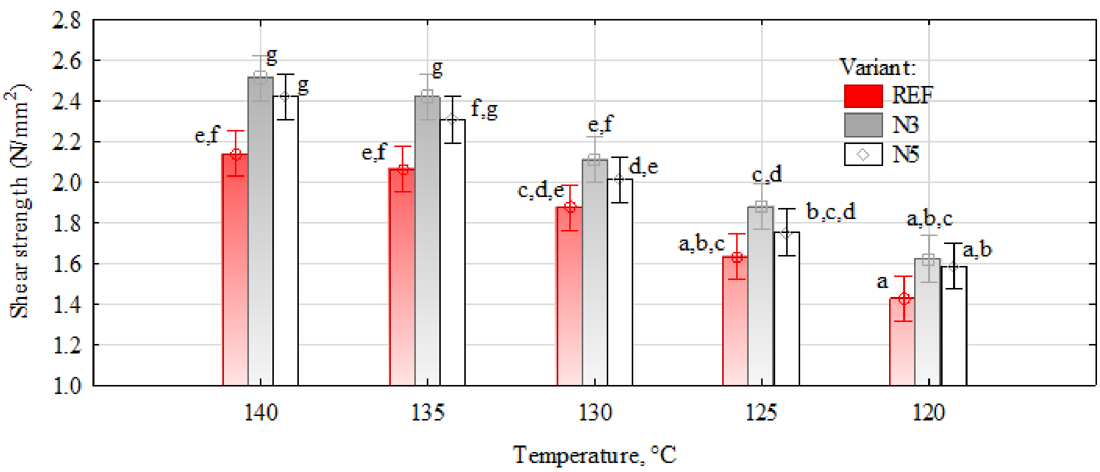
a)



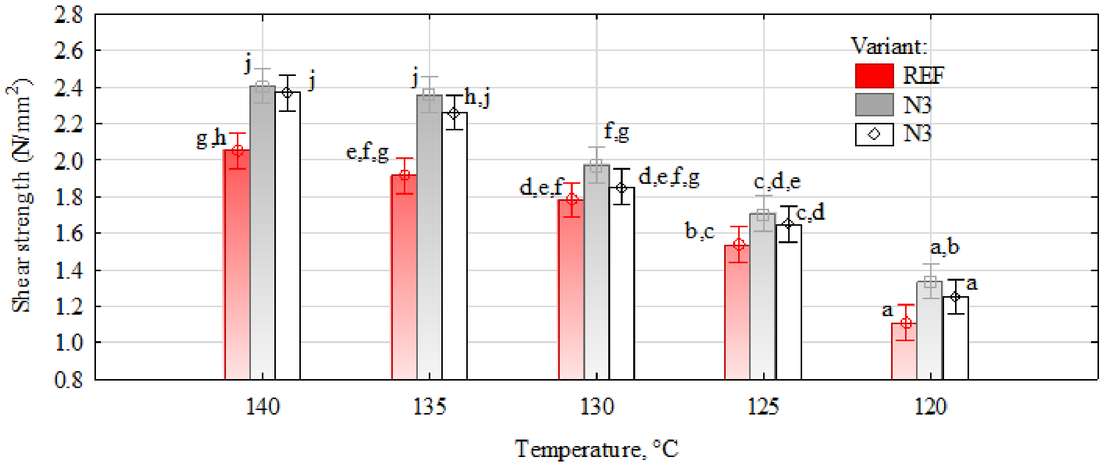
b)

Fig. 1: The results of bonding quality depending on the pressing time: a) after soaking; b) after boiling (letters indicate the homogeneous groups distinguished with Tukey test).

The results have shown that the addition of nanocellulose led to the statistically significant improvement in bonding quality of plywood which confirmed the previous observations (Kawalerczyk et al. 2020). However, no significant differences were observed as the amount of NCC increased contrary to the previous studies. The analysis of homogeneous groups indicates that the NCC-reinforced plywood panels hot-pressed for 180 s were characterized by the same bonding quality as the reference variant pressed for 240 s both after soaking and after boiling. Thus, the addition of nanocellulose allowed the reduction of pressing time by 25%. The further reduction in pressing time to 150 s led to a major decrease in bonding quality regardless of the variant. Moreover, the effect of pressing temperature on the shear strength of plywood is shown in Fig. 2.



a)



b)

Fig. 2: The results of bonding quality depending on the pressing temperature: a) after soaking; b) after boiling (letters indicate the homogeneous groups distinguished with Tukey test).

The presented results confirmed the positive influence of NCC introduction on the bonding quality of plywood when compared with the reference variant. Furthermore, the results also show that the addition of nanocellulose allowed to decrease pressing temperature by 10°C which accounted for approx. 7% of the total pressing time. The decrease in pressing temperature had more notable effect on the bonding quality of plywood than the time. The proper temperature is especially important for phenolic resins since they require a considerable amount of thermal energy in order to complete the polycondensation i.e. the application of high temperature ranging from 135 to 150°C (Mirski et al. 2011). Therefore, the drop of temperature below 130°C resulted in major decrease in bonding quality of both non-modified and modified plywood.

Both bending strength and modulus of elasticity are of great importance for the overall performance of wood-based materials. Their values can be crucial for determining the potential usability of the obtained panels. According to Ayrilmis et al. (2010) these are the features especially important for the structural applications exposed to varying environmental conditions which suits the uses for which PF resin-bonded plywood is intended for. Thus, the results of plywood mechanical properties are summarized in Fig. 3.

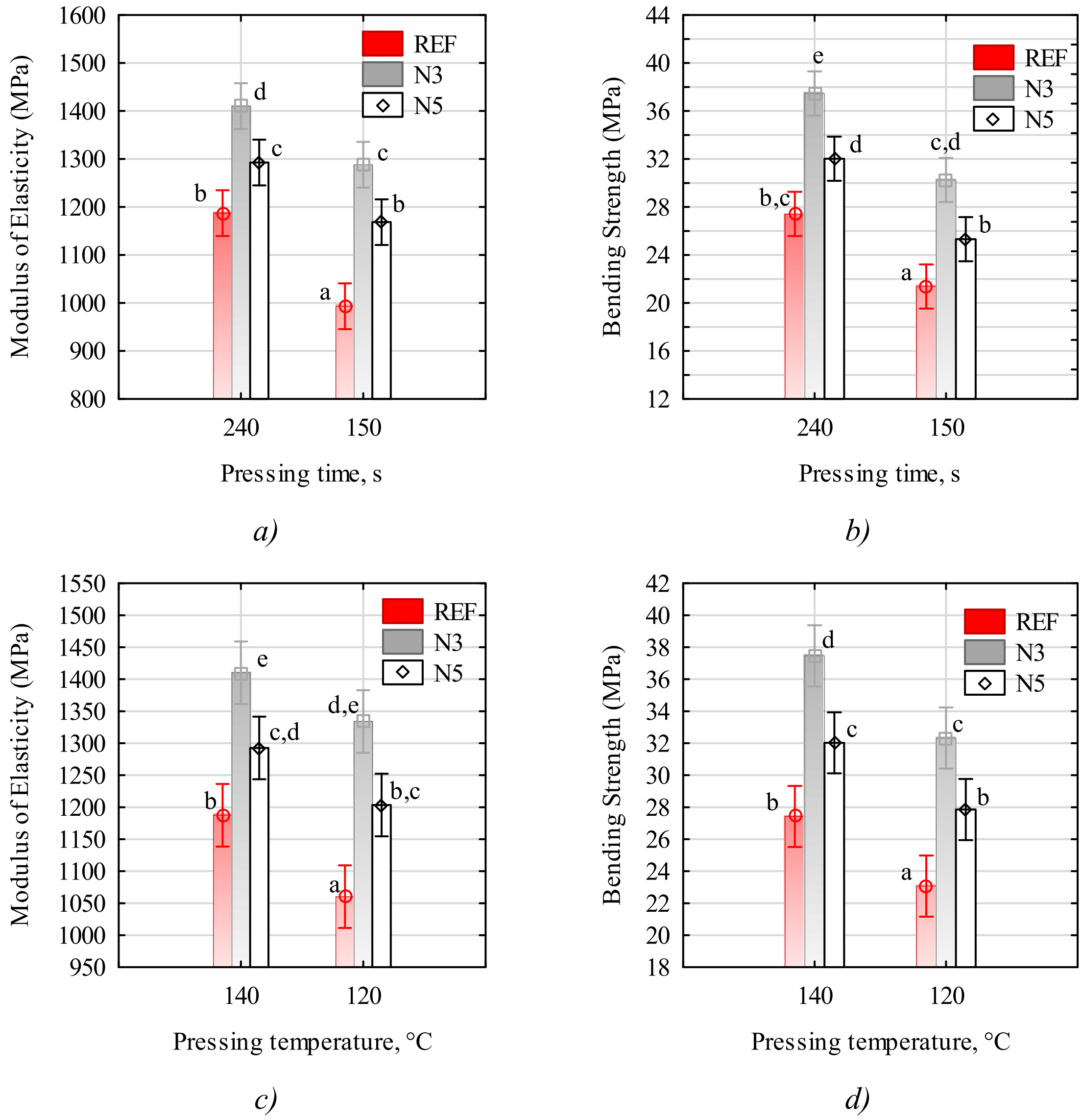


Fig. 3: The effect of pressing parameters on: a) and c) modulus of elasticity; b) and d) bending strength of plywood (letters indicate the homogeneous groups distinguished with Tukey test).

Contrary to the results of bonding quality, the amount of added NCC had a significant effect on the investigated characteristics of plywood. The best outcomes can be noted for N3 variant which confirms the previous observations (Kawalerczyk et al. 2020). The results show that in terms of mechanical properties it is possible to reduce the pressing time by 38% due to the use of NCC-enhanced PF resin. Moreover, the results of modulus of elasticity indicate that in case of N3 variant it could be possible to decrease the pressing time even more. In case of NCC-reinforced variants it was also possible to reduce the pressing temperature by 14% (from 140 to 120°C). Similarly as in the case of pressing time, it could potentially be possible to lower the temperature even further by introduction of N3 variant based only on the results of mechanical tests. However, taking into account the major decrease in bonding quality of plywood pressed with the use of temperature lower than 130°C these variants were omitted.

The veneer-based materials used in exterior environment tend to be delaminated over the time and it causes the significant loss of rigidity and strength of the applied panels. Because of the quick fall in the mechanical properties that accompanies the process of delamination, it is a reliable predictor of plywood behavior in varying conditions (El Moustaphaoui et al. 2021, Koch 1967). The results of performed investigations are shown in Tab. 4.

Tab. 4: The results of delamination test.

Adhesive variant	Pressing time (s)	Pressing temperature (°C)	First round*	Third round**	Result***
REF	240	140	0/20	0/20	P
N3	240	140	0/20	0/20	P
N5	240	140	0/20	0/20	P
REF	150	140	0/20	0/20	P
N3	150	140	0/20	0/20	P
N5	150	140	0/20	0/20	P
REF	240	120	0/20	1/20	P
N3	240	120	0/20	0/20	P
N5	240	120	0/20	1/20	P

Note: *number of delaminated samples after the first round of soaking/drying cycle; **number of delaminated samples after the third round of soaking/drying cycle; ***P means testing passed, F means testing failed.

It turned out that regardless of the pressing parameters and adhesive compositions, all of the investigated plywood samples passed the delamination test which means that they are suitable for both the indoor and exterior applications. Despite the reduction of pressing parameters, the delamination did not occur after first round of soaking and drying. The decrease in pressing temperature caused the delamination of one in twenty samples representing REF and N5 variants tested after 5 cycles of pre-treatment. However, the overall result is still positive and it meets the requirements for materials used outdoors. The high resistance to delamination probably results from the fact that the bond lines made of PF resin do not hydrolyze in the presence of water which makes them highly water-resistant.

The possibility to reduce the pressing parameters which was created due to the nanocellulose-reinforcement probably resulted from both the enhancement in resin curing and the improvement in bond line strength. The accelerated polycondensation which was observed during gel time measurements means that less time is needed for the PF adhesive to cure completely and consequently to provide the glue joints with the assumed strength. The significant improvement in plywood mechanical properties can be also observed for particleboards, oriented strand boards (OSB) or solid wood bonding. It was previously stated that the addition of NCC increases the specific fracture energy of wood adhesive bonds (Veigel et al. 2011, 2012). Moreover, the morphology of NCC-enriched UF and PF resins was determined with the scanning electron microscope (SEM). The results have shown that the structure of modified adhesive was more compact, solid and significantly less porous. Furthermore, in comparison with the reference, non-modified resin it was characterized by the limited occurrence of microcracks and cavities (Kawalerczyk et al. 2020, 2021). In addition, according to Lengowski et al. (2019) the presence of cellulose nanocrystals increases the wood-adhesive-wood bonding and interaction. Overall, all of the above mentioned factors show that the application of nanocellulose as a modifier of PF resin for wood-based composites has a great potential.

CONCLUSIONS

Based on conducted study it was found that the addition of nanocellulose to PF resin caused a significant enhancement in resin curing time which is a particularly beneficial effect considering the investigated influence on pressing parameters. Moreover, the reinforcing effect of NCC introduction on the properties of plywood was confirmed. The outcomes of bonding quality allow to conclude that there is a possibility to reduce pressing time by 25% and pressing temperature by 7% due to the use of NCC-enriched resin. Furthermore, the results of mechanical properties such as modulus of elasticity and bending strength of the resultant panels indicate that there is a possibility to decrease the pressing time by 38% and pressing temperature by 14%. However, bonding quality is a fundamental indicator for adhesive performance and in the case of resin modification it seems to be a more reliable parameter. Regardless of the pressing parameters and the adhesive composition, the PF resin bond lines remain resistant to delamination. Taking into account how energy-consuming the pressing process is, that major reduction could be very beneficial for the production of plywood.

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