Potential of shortening pressing time or reducing pressing temperature for plywood resinated with PF resin modified using alcohols and esters

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Abstract: The study was an analysis of the potential to shorten pressing time or reduce pressing temperature for plywood resinated with alcohol- and ester-modified PF resin. Birch and pine plywoods were produced using such modified resin at a temperature range of 135 to 115°C, at a constant pressing time (4 min) and at pressing times ranging from 240 to 150 s at a constant pressing temperature (135°C). Tests showed that the application of PF resin modified with polyols and esters makes it possible, under standard pressing conditions, i.e. 135°C and pressing time of 4 min, to produce plywood with improved properties in comparison to respective control plywoods. Moreover, it was shown that the used modifiers enhance resin reactivity, facilitating an adequate course of resin curing, resulting in the manufacture of plywoods with good properties even at a pressing temperature reduced by 20°C or pressing time shortened to 150 s.

Potential von mit Alkohol und Estern modifiziertem PF-Harz zur Reduzierung der Presszeit oder der Presstemperatur bei der Sperrholzherstellung

Zusammenfassung

In dieser Studie wurde untersucht, wie die Presszeit bzw. die Presstemperatur bei der Herstellung von Sperrholz durch Verwendung eines mit Alkohol- und Ester modifizierten PF-Harzes reduziert werden kann. Unter Verwendung solcher Harze wurde Birken- und Kiefernsperrholz hergestellt, und zwar bei einer Presstemperatur von 135 bis 115°C und einer konstanten Presszeit von 4 min bzw. bei Presszeiten von 240 bis 150 s und einer konstanten Presstemperatur von 135°C. Die Versuche zeigten, dass durch Verwendung von PF-Harz, das mit Polyolen und Estern modifiziert wurde, bei Standardbedingungen, d.h. bei 135°C und einer Presszeit von 4 min, Sperrholz hergestellt werden kann, das im Vergleich zu Kontrollproben verbesserte Eigenschaften aufweist. Des Weiteren wurde gezeigt, dass die Modifizierung die Harzreaktivität verstärkt und somit den Härungsverlauf verbessert, mit dem Ergebnis, dass auch bei einer um 20°C reduzierten Presstemperatur oder einer auf 150 s verkürzten Presszeit Sperrholz mit guten Eigenschaften hergestellt werden kann.

INTRODUCTION

Phenol-formaldehyde (PF) resins are predominant bonding agents in the production of water-resistant wood-based materials (Andersen and Troughton 1996), primarily construction plywood. The application of these resins, thanks to their good adhesion to veneer surface, makes it possible to obtain adhesive-bonded joints with good mechanical properties and high water resistance. Additionally, formed joints are relatively elastic, resistant to vibrations and thermally stable, which contributes to higher stability of wood-based materials resinated with PF resin (Irle and Bolton 1988). However, for curing they require considerable amounts of thermal energy, i.e. the application of high temperatures (135 - 150°C), or long pressing times (45 - 60 s/mm). For these reasons it has been attempted to increase reactivity of PF resin. In view of the fact that construction plywood produced with the use of PF resin is frequently more than 18 mm in thickness, a reduction of temperature and especially a shortening of pressing time would contribute to improved economic indexes of its manufacture processes. A method preferred by many researchers and producers of wood-based materials is modification of ready-to-use resins with additions of substances accelerating curing or reducing the temperature of this process, due to the considerable number of compounds facilitating this process and their easy application. It results from a review of literature that promising results have been obtained when using esters as compounds accelerating curing of PF resin. Reports on the activating action of esters on PF resin curing appeared as early as the 1950's (Orth 1957, 1958), while in the 1970's pioneering attempts were made to apply esters in curing phenolic resin under commercial-scale production conditions, although only in foundry practice (Westwood and Higgins 1985). In recent years the problem of enhancing reactivity of phenolic resins using esters has been focused on (Stephanou and Pizzi 1993, Pizzi and Stephanou 1994, Higuchi et al. 1994, Tohmura and Higuchi 1995, Pizzi et al. 1997, Park and Riedl 2000, Conner et al. 2002). To date the mechanism activating the action of esters as modifiers of PF resin polycondensation has not been fully clarified and the results of these studies have not been comprehensively evaluated (Lei et al. 2006).

Moreover, investigations on this subject undertaken by these authors showed that the application of esters as modifiers of PF resin makes it possible to manufacture particleboards and plywood with very good physical and mechanical properties (Łęcka et al. 2001a, Mirski et al. 2004, 2008c). Additionally, studies carried out by these authors proved that esters of diols exhibit the highest effectiveness in enhancing reactivity of PF resin (Mirski et al. 2008a), while the application of such modified resin makes it possible to manufacture

particleboards and plywood with good mechanical properties and high water resistance (Mirski et al. 2008b,c). When attempting to explain the mechanism of the action of esters in the process of enhancing the reactivity of PF resin these authors focused on the special role played in this process by alcohols, formed as a result of their alkaline hydrolysis. Studies showed that alcohols, particularly polyols, used as modifiers of PF resin, not only catalyze the reaction of polycondensation, but they are also embedded in its structure (Mirski et al. 2005, 2008a). In turn, particleboards and plywoods produced with the use of such modified resin are characterized by better physical and mechanical properties than materials resinated with pure PF resin (Łęcka et al. 2001b, Mirski et al. 2008c).

Thus, it was considered advisable to investigate the potential use of polyols and selected esters to manufacture exterior plywood at a shortened pressing time or reduced pressing temperature.

MATERIALS AND METHODS

In the analyses, the tested PF resin was the type commonly used in the manufacture of plywood with increased water resistance, with the following characteristics: dry matter – 51.37%, density – 1.215 g/cm³, viscosity – 305 mPa·s, gel time at 130°C – 246 s, pH – 11.08. The following compounds were used as modifiers of phenolic resin: propane-1,3-diol, propane-1,2,3-triol (glycerin), ethyl malonate and propylene diacetate. Applied modifiers were introduced to resin at 0.05 mol/100 g d.m. resin, with curing agent UT-10 used as a filler of the adhesive mixture. The type and amounts of modifiers were selected on the basis of preliminary testing (Mirski et al. 2008c).

Three-ply plywood was manufactured from birch and pine veneers in the 1.4×1.7×1.4 mm thickness system, applying the following pressing conditions:

- pressure - 1.6 N/mm² for birch plywood and 1.4 N/mm² for pine plywood,
- temperature - 115, 120, 125, 130 and 135°C,
- time - 240, 210, 180 and 150 s,
- amount of adhesive mixture applied on veneer - 160 g/m².

Thus manufactured plywoods were tested in terms of such properties as:

- modulus of rigidity MOR and modulus of elasticity MOE according to EN 310,
- bond quality in the shear test f_v according to EN 314-1, with samples subjected to pretreatments following points 5.1.1 (24 h soaking in cold water, tested wet) and 5.1.3 (4 hours boiling water + 16 hours 70°C drying + 4 hours boiling water, tested wet), according to the recommendations of standard EN 314-2 for exterior plywood.

For each combination (pressing time/temperature, type of modifier and species of veneer wood) three plywood boards were manufactured. A total of 12 samples were cut from each plywood sheet to be used in testing of bending strength and modulus of elasticity as well as bond quality after accelerated ageing treatments. Results of analyses of individual properties were thus a mean of 36 measurements. Results of these determinations were subjected to statistical analysis.

RESULTS AND DISCUSSION

Results of studies regarding the effect of PF resin modification with polyols and esters on properties of manufactured hardwood and softwood plywoods, depending on their pressing parameters, are presented in Tables 1 - 4. It results from conducted analyses that the application of such modified PF resin makes it possible under standard pressing conditions, i.e. temperature of 135°C and time of 4 min, to produce plywoods with better properties than respective control plywoods, at markedly superior properties found for plywood resinated with ester-modified resin. When the average increase in bond quality and bending strength of plywoods manufactured with the use of alcohol-modified resin, irrespective of wood species, direction of testing and the type of ageing-acceleration tests, was 8%, then in case of ester-modified resin it improved by as much as 20% (Tables 1, 2, 3 and 4). In contrast, no significant differences were observed in properties of plywoods depending on the type of applied alcohols or esters, since for both polyols and esters they were similar.

In turn, tests conducted on the potential shortening of plywood pressing time showed that plywoods manufactured at each pressing time (240, 210, 180 and 150 s), irrespective of the type of applied modifier of PF resin, exhibited better properties than respective control plywoods, and with its shortening only a slight reduction of properties of manufactured plywoods was found (Tables 1 and 2). The most effective modifier was ethyl malonate, since properties of plywood produced with such modified resin, even at the shortest pressing time of 37.5 s/mm, were comparable to those of plywood resinated with pure PF resin at a pressing time of 60 s/mm thickness, both in case of birch and pine plywood. Moreover, as it results from data presented in Table 1 birch plywoods manufactured at a pressing time of 150 s with the use of such modified resin exhibited bond quality after accelerated ageing tests, in relation to control plywood produced under identical conditions, higher by 13 and 28%, respectively, after soaking and boiling tests. Under these conditions such a considerable increase in bond quality proves an advantageous effect of the applied modification on PF resin curing, which determines the stability of produced adhesive-bonded joint. However, it needs to be assumed

that these reactions occur not only within resin, but also at the resin-wood interface, as it is indicated by the results of bending strength testing in both directions. An increase in the level of this property was recorded, amounting to 17 and 21%, respectively, as measured perpendicularly and parallel to grain in top layers (Table 2). Thus it may be assumed that the applied modifier on the one hand accelerates polycondensation processes of PF resin and on the other hand facilitates its copolymerization with functional groups of cellulose or lignin in wood. As a result this leads to increased chemical adhesion of resin to veneers and thanks to enhanced rigidity of the formed glue line an increase in bending strength is observed.

These assumptions are also confirmed by the results of tests on properties of plywoods manufactured at pressing temperature reduced by 20°C. They showed that strength determined in shear tests after accelerated ageing tests for plywoods manufactured under these conditions and resinated with resin modified using ethyl malonate was higher than that of respective control plywoods on average by 17 and 35% for birch and pine plywood, respectively (Table 3). In turn, bending strength of plywoods manufactured under these conditions, irrespective of the wood species and direction of testing, was on average by 17% higher than those of control plywoods (Table 4). Moreover, it needs to be stressed that even despite a reduction of pressing temperature to 115°C, plywoods produced using such modified resin exhibit properties comparable to those of plywood resinated with pure PF resin and pressed at 135°C.

Such trends in testing results of plywoods manufactured at a reduced pressing temperature most probably result from the fact that applied modifiers, particularly ethyl malonate, increase reactivity of resin, thus facilitating an adequate course of its curing, resulting in the manufacture of plywoods with good properties even at pressing temperature reduced by 20°C, as manifested by the considerable shortening of gel time (Mirski et al. 2008a)

Promising testing results both concerning the shortening of pressing time and reduction of pressing temperature, were the basis for the attempted manufacture of plywoods at pressing time shortened to 3 min and simultaneously pressing temperature reduced to 120°C. In this part of the study plywoods were manufactured, resinated with resin modified with propane-1,3-diol and ethyl malonate. However, it turned out that the use of such pressing conditions does not make it possible to produce plywoods resinated with resin modified with propane-1,3-diol, as they disintegrated during accelerated ageing tests. In turn, plywoods resinated with PF resin modified with ethyl malonate, produced under identical conditions, exhibited good properties (Table 5). It needs to be stressed here that strength of plywoods

manufactured at shortened pressing time and reduced pressing temperature, determined in shear testing after accelerated ageing tests, irrespective of wood species, was higher than that of respective control plywoods on average by 32%, while bending strength, irrespective of the direction of tests, on average by 19%.

Results of strength testing of plywoods resinated with resin modified with ethyl malonate and manufactured at the simultaneously lowered temperature and shortened pressing time seem to confirm the thesis on the double role of esters in PF resin curing. As it was shown by the tests conducted by the authors of this study, acids released as a result of the reaction of hydrolysis occurring in the alkaline medium of PF resin, act as catalysts and facilitate embedding of simultaneously formed alcohol into its structure (Mirski et al. 2008a), thanks to which it is possible to manufacture plywoods with good properties under these conditions.

CONCLUSION

1. The application of PF resin modified with alcohols and esters makes it possible, under standard pressing conditions, i.e. temperature of 135°C and time of 4 min, to manufacture plywoods with better properties than those of respective control plywoods, with markedly superior properties characterizing plywoods resinated with ester-modified resin.
2. Modifiers increase resin reactivity to such an extent that they facilitate an adequate course of its curing process, facilitating the manufacture of plywoods with good properties even at pressing temperature reduced by 20°C or pressing time shortened by 38%.
3. The most effective modifier of PF resin was ethyl malonate and the application of such modified resin made it possible to produce plywood with good mechanical properties and bond quality at a shortened pressing time and reduced pressing temperature.

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Tabelle 1

Scherfestigkeit von Sperrholz, das mit modifiziertem Pf-Harz hergestellt wurde, in Abhängigkeit der Presszeit

Tabelle 2

Biegefestigkeit und Biege-Elastizitätsmodul von Sperrholz, das mit modifiziertem PF-Harz hergestellt wurde, in Abhängigkeit von der Presszeit

Tabelle 3

Scherfestigkeit von Sperrholz, das mit modifiziertem Pf-Harz hergestellt wurde, in Abhängigkeit von der Presstemperatur

Tabelle 4

Biegefestigkeit und Biege-Elastizitätsmodul von Sperrholz, das mit modifiziertem PF-Harz hergestellt wurde, in Abhängigkeit von der Presstemperatur

Tabelle 5

Eigenschaften von Sperrholz, das mit Ethylmalonat modifiziertem PF-Harz hergestellt wurde (Presstemperatur 120°C, Presszeit 180 s)

Table 1

Bond quality of plywood manufactured with modified PF resin depending on pressing time

Kind of modifier	Pressing time (s)*	Shear strength f_v (N·mm ⁻²)							
		24 h cold water soaking				boiling water			
		Pine		Birch		Pine		Birch	
PF	240	1.29	0.21**	2.10	0.41	1.22	0.26	1.74	0.29
propane-1,2,3-triol		1.33	0.16	2.14	0.19	1.30	0.22	1.81	0.19
propane-1,3-diol		1.39	0.26	2.19	0.28	1.50	0.19	1.81	0.22
propylene diacetate		1.41	0.17	2.27	0.32	1.51	0.26	1.85	0.21
ethyl malonate		1.48	0.19	2.33	0.46	1.70	0.17	1.95	0.17
PF	210	1.23	0.22	1.92	0.31	1.16	0.19	1.65	0.23
propane-1,2,3-triol		1.25	0.2	1.95	0.29	1.17	0.22	1.78	0.19
propane-1,3-diol		1.26	0.19	2.05	0.33	1.18	0.29	1.79	0.19
propylene diacetate		1.27	0.21	2.07	0.42	1.25	0.21	1.81	0.22
ethyl malonate		1.47	0.14	2.12	0.36	1.34	0.24	1.89	0.32
PF	180	1.10	0.09	1.83	0.26	1.11	0.29	1.55	0.34
propane-1,2,3-triol		1.11	0.16	1.90	0.29	1.17	0.22	1.65	0.26
propane-1,3-diol		1.13	0.21	1.99	0.33	1.21	0.19	1.69	0.19
propylene diacetate		1.20	0.29	2.03	0.42	1.27	0.17	1.74	0.23
ethyl malonate		1.35	0.18	2.08	0.25	1.27	0.21	1.77	0.36
PF	150	1.07	0.18	1.76	0.22	0.98	0.21	1.32	0.21
propane-1,2,3-triol		1.09	0.22	1.86	0.35	1.12	0.11	1.47	0.19
propane-1,3-diol		1.10	0.26	1.91	0.32	1.12	0.19	1.64	0.17
propylene diacetate		1.37	0.21	1.98	0.21	1.14	0.16	1.67	0.3
ethyl malonate		1.47	0.21	1.99	0.41	1.16	0.18	1.69	0.24

* pressing temperature - 135°C

** standard deviation

Table 2

Bending strength and modulus of elasticity in bending of plywood manufactured with modified PF resin depending on pressing time

Kind of modifier	Pressing time (s)*	MOR				MOE			
		N·mm ⁻²							
		Pine		Birch		Pine		Birch	
		II	⊥	II	⊥	II	⊥	II	⊥
PF	240	112	22.2	135	35.9	12000	890	13100	1690
		19	2.3**	16	2.9	1950	110	2340	210
propane-1,2,3-triol		116	25.6	140	37.8	12300	1250	14200	1790
		22	1.9	17	3.2	2010	80	2460	170
propane-1,3-diol		124	26.6	145	38.3	12600	1260	14600	1800
		17	2.4	19	2.9	2630	150	2190	150
propylene diacetate		135	32.3	150	38.8	12600	1440	14700	1810
		16	1.8	21	2.6	1750	90	1870	230
ethyl malonate		138	32.3	159	45.1	14100	1570	14800	1950
		12	4.2	22	4.1	1980	210	2240	190
PF	150	102	23.0	113	33.1	9060	990	11800	1460
		9	2.9	19	2.6	1730	130	2420	130
propane-1,2,3-triol		105	23.2	114	35.0	10700	1000	11900	1470
		16	3.2	26	4.8	1390	140	2190	410
propane-1,3-diol		104	23.5	119	35.4	11200	1010	12100	1560
		17	2.6	21	4.9	2600	145	2190	210
propylene diacetate		106	24.9	129	37.3	11200	1020	12700	1580
		12	3.4	19	5.1	1380	160	1980	180
ethyl malonate		108	26.2	137	38.7	11400	1090	13600	1740
		21	4.3	24	5.5	1860	170	2000	250

* pressing temperature - 135°C

** standard deviation

Table 3

Bond quality of plywood manufactured with modified PF resin depending on pressing temperature

Kind of modifier	Pressing temperature (°C)*	Shear strength f_v (N·mm ⁻²)					
		24 h cold water soaking			boiling water		
		Pine		Birch	Pine		Birch
PF	135	1.49	0.12**	1.58	0.23	1.24	0.19
propane-1,2,3-triol		1.59	0.26	1.89	0.33	1.39	0.23
propane-1,3-diol		1.59	0.31	1.97	0.25	1.43	0.18
propylene diacetate		1.66	0.22	2.00	0.36	1.43	0.22
ethyl malonate		1.68	0.26	2.14	0.27	1.52	0.27
PF	130	1.38	0.13	1.60	0.19	1.03	0.12
propane-1,2,3-triol		1.40	0.23	1.79	0.22	1.12	0.16
propane-1,3-diol		1.42	0.29	1.82	0.26	1.27	0.21
propylene diacetate		1.42	0.24	1.94	0.21	1.53	0.23
ethyl malonate		1.68	0.26	2.05	0.28	1.61	0.29
PF	125	1.19	0.16	1.75	0.22	1.16	0.23
propane-1,2,3-triol		1.33	0.13	1.91	0.21	1.23	0.26
propane-1,3-diol		1.49	0.11	1.95	0.19	1.37	0.17
propylene diacetate		1.50	0.17	1.95	0.23	1.39	0.19
ethyl malonate		1.64	0.22	1.96	0.28	1.43	0.2
PF	120	1.24	0.23	1.47	0.29	1.12	0.19
propane-1,2,3-triol		1.44	0.21	1.48	0.23	1.28	0.17
propane-1,3-diol		1.54	0.18	1.57	0.21	1.30	0.21
propylene diacetate		1.54	0.11	1.63	0.19	1.30	0.22
ethyl malonate		1.64	0.25	1.75	0.31	1.47	0.26
PF	115	1.06	0.31	1.79	0.32	1.03	0.22
propane-1,2,3-triol		1.20	0.11	1.79	0.28	1.04	0.17
propane-1,3-diol		1.38	0.26	1.83	0.21	1.31	0.22
propylene diacetate		1.44	0.22	1.86	0.22	1.35	0.21
ethyl malonate		1.47	0.23	1.87	0.24	1.36	0.19

* pressing time - 240s

** standard deviation

Table 4

Bending strength and modulus of elasticity in bending of plywood manufactured with modified PF resin depending on pressing temperature

Kind of modifier	Pressing temperature (°C)*	MOR				MOE			
		N·mm ⁻²							
		Pine		Birch		Pine		Birch	
		II	⊥	II	⊥	II	⊥	II	⊥
PF	135	92.7	27.9	104	37.6	9080	1200	10200	1630
		12	4.3**	14	4.7	1450	130	1480	210
propane-1,2,3-triol		98.0	29.7	118	38.1	10100	1250	10400	1640
		17	4.8	19	5.7	1780	190	1870	240
propane-1,3-diol		99.9	31.3	119	40.3	10100	1350	10500	1710
		11	3.8	14	4.7	1450	210	1630	210
propylene diacetate		101	32.7	120	42.3	11500	1580	11000	1970
		18	4.9	17	5.3	1800	310	1450	260
ethyl malonate		105	35.6	131	42.9	11800	1720	12300	1990
		16	5.1	11	5.3	1680	240	1780	250
PF	120	79.2	24.4	97	31.5	8750	1190	9440	1460
		16	4.1	12	4.9	1350	200	1470	230
propane-1,2,3-triol		80.0	27.2	101	31.5	9790	1240	10400	1470
		11	4.7	16	5.6	1470	230	1510	190
propane-1,3-diol		87.0	27.7	103	34.8	9860	1300	11200	1490
		15	4.5	18	5.2	1380	220	1650	240
propylene diacetate		88.1	28.5	105	37.7	10280	1340	11200	1490
		14	4.8	21	5.6	1560	190	1580	280
ethyl malonate		90.6	29.9	108	38.0	10400	1470	11400	1490
		18	4.2	15	4.8	1440	240	1880	210

* pressing time - 240s

** standard deviation

Table 5

Properties of plywood manufactured with ethyl malonate modified PF resin (pressing temperature 120°C, pressing time 180 s)

Wood species	MOR		MOE		Shear strength f_v	
	II	$\perp$	II	$\perp$	cold water	boiling water
	N/mm ⁻²					
pine	80.2* 12**	23.4 2.1	8380 1560	1060 150	0.78 0.08	0.55 0.09
	101 11	28.3 3.2	10600 1890	1210 180	0.89 0.07	0.79 0.13
birch	102* 12	31.3 3.6	10400 2100	1500 130	0.77 0.11	0.68 0.15
	118 9	34.6 4.2	12900 1220	1670 210	1.06 0.19	0.89 0.12

* control plywood

** standard deviation