

# Synthesis of Cardanol-Containing Resols for Producing Phenolic Films: Protective Coatings for Wood Composites

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**Abstract**—This paper investigates synthesis of impregnating phenol–formaldehyde resins in which up to 30 wt % formulation phenol is replaced by cardanol, a plant product. The resulting resins were used to make impregnated paper for laminating the plywood and particle boards. The synthesis and structure of cardanol-containing water-soluble resols were studied by IR spectroscopy and <sup>1</sup>H NMR, and the reactivity of phenol cardanol formaldehyde resins at 80–150°C was studied by differential scanning calorimetry. The optimal amount of cardanol in relation to phenol in the resin structure was determined. It is shown that the introduction of cardanol into the composition of phenol–formaldehyde resins reduces the impregnation time of kraft paper and improves the surface quality and elasticity of the impregnated paper, with the use of such paper making it possible to decrease the water absorption of laminated particle board materials and the emission of formaldehyde.

**Keywords:** cardanol, resols, phenolic films, lamination, wood composites

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Laminated wood composites, due to a complex of improved performance characteristics, are used in various industries. Laminated plywood is widely used in the manufacture of surfaces resistant to external influences: reusable panel timbering for concrete works; floors and inner walls of vehicles, freight wagons, and containers; work sites and levels in construction; materials for the automotive industry; and athletic fields and playgrounds.

Lamination improves water and chemical resistance, abrasion resistance, and mold and mildew resistance; reduces formaldehyde emissions; improves the appearance; and extends the life of wood composites.

Wood composites are laminated using phenolic film, a special kraft paper with a density of 40–200 g/m<sup>2</sup> impregnated with phenol–formaldehyde resole resin. The density of the phenolic film is 120–300 g/m<sup>2</sup>.

Resins used to impregnate kraft papers are water-soluble phenol–formaldehyde resols modified with alcohols (methanol, ethanol) to improve impregnation properties. Solutions for the impregnation of kraft papers, in addition to phenol–formaldehyde resins, may contain anti-adhesives, pigments, or dyes.

In the finished phenolic films, the resin is in the form of resitol (insoluble but still melting state); under further heating during the lamination of wood composites, resitol cures completely and becomes resite [1].

The main disadvantage of phenolic–formaldehyde resins is that they contain residual monomers (phenol and formaldehyde), which can migrate into the environment and the air of the working area during industrial treatment of such resins.

To obtain resole-type adhesives based on renewable plant products, it is of interest to consider cardanol, which is isolated from cashew nut shell liquid [2]. The world reserves of cashew nut shell liquid in 2013 amounted to 1 mln t [3], which corresponds to 670000 t in terms of cardanol.

Cardanol contains C<sub>15</sub> *meta*-alkylphenols: 3-pentadecylphenol, 3-(8(*Z*)-pentadecenyl)phenol, 3-(8(*Z*),11(*Z*)-pentadecadienyl)phenol, and 3-(8(*Z*),11(*Z*),14-pentadecatrienyl)phenol [2].

Resins containing cardanol can be used as adhesives for a wide range of environmentally friendly wood composites with high physical and mechanical characteristics (chipboard and fiberboard, wood laminate, oriented strand boards (OSBs)) [4–8].

According to the literature data, cardanol-containing resole resins are used for the manufacture of composite materials based on plant fibers: sisal (*Agave sisalana*) [9], white jute (*Corchorus capsularis*) [10], bamboo (*Bambusa vulgaris*) [11], and sugar cane (*Saccharum officinarum* L.) [12]. The use of cardanol-containing resole resins as a binder and materials of plant origin as a filler makes it possible to obtain composites containing more than 70% of renewable raw materials.

In [13–15], the authors describe synthesis of cardanol-containing resole resins and study the mechanism of the reaction of polycondensation of cardanol with formaldehyde in the presence of an alkaline catalyst when the cardanol : formaldehyde molar ratio was varied from 1 : 0.85 to 1 : 1.75. The authors of [16] studied the kinetics of condensation between cardanol and formaldehyde in the presence of a catalyst—sodium hydroxide—at temperatures of 28, 50, and 70°C. It was noted that the bulky hydrocarbon substituent located in the *meta*-position of the phenol ring, due to the influence of the steric factor, prevents the formation of methylol groups in position 2 at 28°C. This position becomes active when the temperature rises to 50–70°C.

Work [17] describes features of synthesis and properties of phenol–formaldehyde resins in which up to 40% phenol was replaced by cardanol. With a higher content of cardanol, the resin is characterized by high viscosity and low solubility.

Cardanol, having high reactivity in electrophilic substitution reactions in the *ortho* and *para* positions, is potentially suitable for producing water-resistant composites.

The aim of this work was to obtain impregnating phenol–cardanol–formaldehyde resole resins and phenolic films based on them and to investigate the curing kinetics of resins and the characteristics of plywood and chipboards laminated with cardanol-containing phenolic films.

## MATERIALS AND METHODS

The study was carried out using cardanol produced by Southern Agro Phenols Limited (SAPL, India); phenol, methanol, and sodium hydroxide from Aldrich; formalin 37% produced by PAO Uralkhimplast; 40 g/m<sup>2</sup> kraft paper from Gebr. Hoffsümmmer Spezialpapier GmbH & Co. KG (Germany); polished birch plywood 8 mm thick from Sveza Co. (Verkhnyaya Sinyachikha settlement); and polished particle boards 16 mm thick from OOO Lestekh (Alapavsk).

The structure of impregnating phenol–cardanol–formaldehyde resins (IPCFRs) was analyzed using a Shimadzu IR Prestige-21 FTIR spectrophotometer with KRS-5 glass.

NMR spectra were analyzed using a Bruker AVANCE II spectrometer.

Heat fluxes during the curing of phenol–cardanol–formaldehyde resins were measured using a DSC 823e/700 differential scanning calorimeter manufactured by Mettler Toledo. The kinetic parameters of the polycondensation reaction were calculated by the method of model-free kinetics [18, 19] using the STAReSW 9.01 software. The measurements were carried out at heating rates of 5, 10, and 20°C/min in the temperature range from 80 to 150°C.

In the manufacture of laboratory samples of IPCFRs, up to 30 wt % formulation phenol was replaced by cardanol while maintaining the molar ratio of the mixture of phenols (phenol + cardanol) to formaldehyde at 1 : 2.3 at the same percentage content of water and sodium hydroxide.

In a glass three-necked reactor equipped with a stirrer, thermometer, and reflux condenser, the calculated amount of cardanol, phenol, and sodium hydroxide was loaded.

Formalin was added in portions to the resulting mixture of phenolates for 2 h at 60–70°C, after which a formulation amount of water was loaded into the reactor. The resins were condensed at 50–70°C until the viscosity of the reaction mixture reached 16–17 s according to the VZ-246 viscometer (nozzle 4 mm), after which methanol was introduced into the resin.

The samples of kraft paper were impregnated in a laboratory bath for 150 s at 22–24°C. The impregnated paper was initially dried at room temperature for 30 min and then in a drying chamber without convection at 135°C for 2.5 min.

To obtain laboratory samples of laminated boards, polished birch FSF plywood with a thickness of 8 mm and a polished three-layer chipboard with a density of 750 kg/m<sup>3</sup> and thickness of 16 mm manufactured using KFMT-15 carbamide–formaldehyde resin were used as a base. Bilateral lamination of the boards was carried out at hot pressing temperature of 135°C and pressure of 1.6 MPa for 7 min. The resulting slabs, after conditioning for at least 1 day, were tested in accordance with the current regulatory documents.

Water absorption of phenolic films upon unilateral wetting (Cobb's method) was determined according to ISO 535–91 on samples of laminated plywood and chipboards 120 × 120 mm in size at 20 ± 5°C and an air humidity of 65 ± 5% for 24 h.

The abrasion resistance of the coating was determined according to ISO 7784 using a Taber 5135 tester. For testing, we used samples of laminated wood composites 100 × 100 mm in size with a hole in the center of 7 mm in diameter; 33 grit sanding paper was used as an abrasive material. Abrasion resistance of the coating after 300 revolutions was determined by the change in the weight of the sample.

**Table 1.** Properties of impregnating phenol cardanol formaldehyde resins

| Indicator   | IPFR    | IPCFR-4 | IPCFR-10 | IPCFR-15 | IPCFR-26 | IPCFR-30 |
|---|---------|---------|----------|----------|----------|----------|
| Viscosity according to VZ-246 (nozzle 4 mm), s            | 16.1    | 16.4    | 16.0     | 16.6     | 16.2     | 16.2     |
| Mass fraction of nonvolatile substances (2 h at 105°C), % | 50.3    | 47.5    | 46.5     | 50.4     | 48.1     | 48.5     |
| Mass fraction of alkali, %                                | 1.91    | 1.91    | 1.91     | 1.89     | 1.91     | 1.89     |
| Mass fraction of free phenol, %                           | 0.30    | 0.30    | 0.51     | 0.46     | 1.73     | 2.65     |
| Mass fraction of free formaldehyde, %                     | 1.12    | 1.10    | 0.72     | 0.87     | 1.65     | 2.04     |
| Miscibility of resin with water by volume                 | >1 : 25 | >1 : 25 | >1 : 25  | 1 : 11.7 | 1 : 0.7  | 1 : 0.4  |
| Penetration at 20°C, s                                    | 11.0    | 14.0    | 8.2      | 9.7      | 3.8      | 3.3      |

The numbers in the resin type indicate the proportion of phenol replacement for cardanol in wt %.

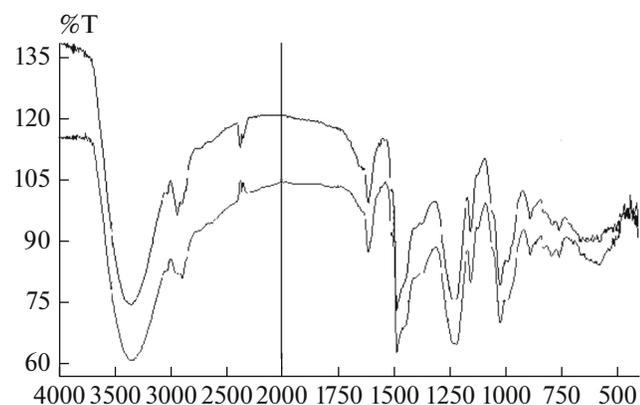
## RESULTS AND DISCUSSION

The properties of IPCFRs synthesized with the replacement of up to 30 wt % phenol by cardanol are presented in Table 1.

An increase in the proportion of cardanol in the composition of phenol cardanol formaldehyde resins shortens the polycondensation process. The condensation time of the resin made with the replacement of 26 and 30 wt % phenol by cardanol is 2.6 and 4 times, respectively, less than the condensation time of the resin that does not contain cardanol.

An increase in the proportion of cardanol in the resin is accompanied by an increase in the content of free phenol and formaldehyde in the finished product, which is possibly associated with a lower degree of condensation of resins, since, at similar temperature and time regimes of manufacturing, the viscosity of the resin with a higher content of cardanol will be significantly higher.

It should be noted that, according to gas chromatography data, all the synthesized resin samples contained no unreacted cardanol.



**Fig. 1.** IR spectra of IPCFR-10 (top) and IPFR (bottom) impregnating resole resins in the range of 4000–400  $\text{cm}^{-1}$ .

When a larger amount of cardanol is added to the resin, the resin penetration time is reduced. Presumably, this is due to an increase in the proportion of low-molecular-weight substances in the finished resin, which reduces the time of kraft paper impregnation. Also, a decrease in the penetration time may be associated with an increased wetting ability of cardanol, which acts as a surfactant under alkaline conditions.

The resins become significantly less miscible with water when more than 15 wt % cardanol is introduced into the resin and almost immiscible when the proportion of cardanol in the resin is 26 wt % and higher, which is due to the effect of the  $\text{C}_{15}$  hydrophobic alkyl substituent.

During storage, the resin with 15 wt % cardanol became slightly turbid after two weeks, while the resins with 26 and 30 wt % cardanol became opaque after 2 and 1 days, respectively. This is probably due to further condensation of the resins during storage, as a result of which the resulting polymer containing hydrophobic  $\text{C}_{15}$  substituents in its composition stopped mixing with water. The highest increase in viscosity during storage of the resins was observed for samples containing 26 and 30 wt % cardanol. Within 2 months, the viscosity of the above resins increased by from three to four times, which is associated with high content of free phenol and formaldehyde in the resins at the time of manufacture. The viscosity of the remaining resin samples at the end of the storage period remained almost at the initial level.

The structure of the obtained IPCFRs was studied using Fourier transform infrared spectroscopy (Fig. 1). The IR spectra of the resole impregnating resins IPFR (without cardanol) and IPCFR-10 (the extent of substitution of phenol for cardanol 10 wt %) are almost identical, except for the absorption band in the region of  $2924 \text{ cm}^{-1}$  and lower intensity of the absorption band at  $825 \text{ cm}^{-1}$ , which is associated with the presence of the  $\text{C}_{15}$  hydrocarbon substituent in the oligomer structure.

The broad absorption band centered at  $3350 \text{ cm}^{-1}$  confirms the presence of stretching vibrations of the

phenolic hydroxyl group. The peak at  $3014\text{ cm}^{-1}$  corresponds to stretching vibrations of aromatic CH groups. The peak at  $2924\text{ cm}^{-1}$ , which is characteristic only for IPCFR-10, corresponds to the presence of asymmetric stretching vibrations of the  $\text{CH}_2$  groups of the  $\text{C}_{15}$  linear alkyl substituent. The peak at  $1610\text{ cm}^{-1}$  corresponds to the stretching vibrations of the  $\text{C}=\text{C}$  bonds of the aromatic ring, and the peak at  $1454\text{ cm}^{-1}$  corresponds to bending vibrations of the  $\text{C}=\text{C}$  bonds of the aromatic ring bound to  $\text{CH}_2$  groups. The absorption bands at  $1226\text{ cm}^{-1}$  confirm that there are stretching vibrations of the phenolic hydroxyl  $\text{C}-\text{OH}$ , bands at  $1153\text{ cm}^{-1}$  indicate asymmetric stretching vibrations of the  $\text{C}-\text{O}-\text{C}$  groups, and bands at  $1022\text{ cm}^{-1}$  indicate stretching vibrations of the methylol groups  $\text{CH}_2-\text{OH}$ . The peak at  $825\text{ cm}^{-1}$  can be attributed to out-of-plane bending vibrations of H atoms in the aromatic ring, which are characteristic of *para* substitution (adjacent 2H atoms), indicating an increase in the proportion of *para* substitutions as compared to IPFR, which is associated with the steric factor of the bulky  $\text{C}_{15}$  substituent. It should be noted that the reaction of electrophilic substitution  $\text{S}_{\text{E}2}$  between phenols (a mixture of phenol and cardanol) and formaldehyde in the presence of a catalyst (sodium hydroxide) proceeds nonselectively both in the *ortho*- and *para*-position of the phenol ring; however, due to the influence of the steric factor of the  $\text{C}_{15}$  substituent in the case of cardanol units, substitution at position 4 (*para*) is superior than at position 2 (*ortho*).

The other absorption bands of the IR spectra of phenol–formaldehyde (IPFR) and phenol–cardanol–formaldehyde (IPCFR-10) resins are almost identical, which suggests a similar structure of resins with the structure shown in Fig. 2.

To confirm the composition and structure of IPCFR-10 and IPFR,  $^1\text{H}$  NMR spectroscopy was used. When comparing the  $^1\text{H}$  NMR spectra of the two samples (Fig. 3), the only difference is found in the presence of proton signals in a strong field at 2.78, 2.56, 2.02, 1.28, and 0.89 ppm. The signals are due to the resonance of protons at saturated ( $\text{sp}^3$  hybridized) carbon atoms. A signal is also observed at 5.35 ppm characteristic of protons at the  $\text{C}=\text{C}$  double bond ( $\text{sp}^2$  hybridized carbon atoms). All these signals refer to the signals of the aliphatic substituent of cardanol and are due to its high content in the sample. The structure of the spectrum in the region of the weak field (aromatic ring) is identical for both resins, which indicates that cardanol has no effect on the selectivity of substitution in the *ortho*- and *para*-positions of the phenol ring.

For selection of the most optimal conditions for lamination of wood-based boards, it is important to investigate the kinetic parameters of curing of binders depending on temperature.

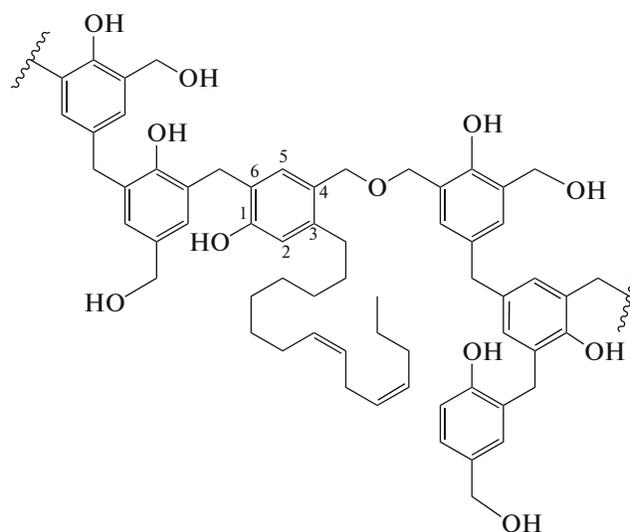


Fig. 2. Hypothetical structure of IPCFR impregnating resole resins.

Temperature and heat fluxes are measured using differential scanning calorimetry (DSC), which makes it possible to characterize the curing process of thermosetting systems.

The kinetic parameters of curing were calculated at  $80\text{--}150^\circ\text{C}$ . To interpret the DSC results, the model-free kinetics (MFK) method was used [18, 19]. Kinetic calculations of the following parameters were carried out: the time to reach 95% conversion ( $\alpha = 95\%$ ) and effective activation energy of the curing reactions  $E_a$ .

The effect of cardanol in the structure of phenol–cardanol–formaldehyde resole on the characteristics of thermal curing in comparison with phenol–formaldehyde resole is shown in Figs. 4 and 5. It can be noted that the IPCFR-10 cardanol-containing resole is more reactive than IPFR, especially in the low-temperature region. The time for the polycondensation reaction to reach  $\alpha = 95\%$  for the IPCFR-10 and IPFR impregnating resole resins is, respectively, 22.5 and 38.1 min at  $110^\circ\text{C}$ , 10.6 and 13.4 min at  $130^\circ\text{C}$ , and 7.5 and 8.3 min at  $140^\circ\text{C}$  (Fig. 6). This is, presumably, associated with the positive inductive effect (+I eff.) of the  $\text{C}_{15}$  alkyl substituent on the reactivity of cardanol-containing oligomers in  $\text{S}_{\text{E}2}$  reactions.

Activation energy  $E_a$  of polycondensation of the IPCFR-10 resin is lower than that of IPFR in the entire range of  $\alpha$  (Fig. 7).

The high rate of curing of the IPCFR-10 resin creates the following favorable prerequisites for the technology of laminating wood composite materials:

- shortening the duration of hot pressing;
- lowering the temperature of hot pressing; and
- improving the performance of laminated boards (higher water resistance, lower formaldehyde emissions) due to the greater extent of resin curing.

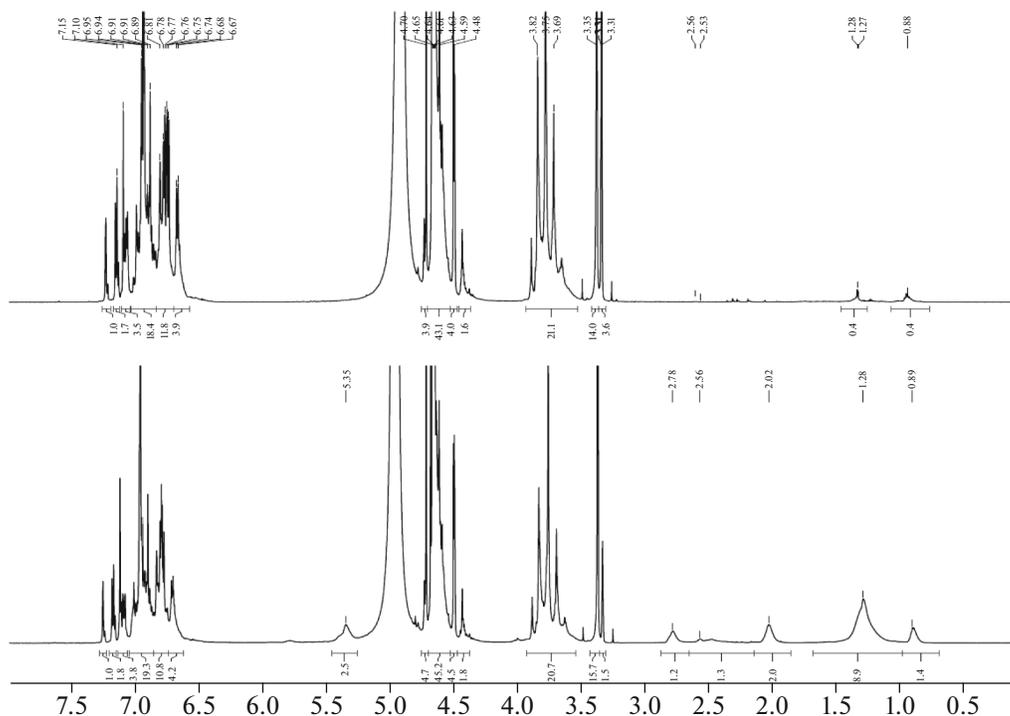


Fig. 3. Comparison of  $^1\text{H}$  NMR spectra of IPCFR-10 (bottom) and IPFR (top).

Resole phenol–cardanol–formaldehyde resins intended for the production of impregnated paper can be of considerable interest for industrial production. The higher degree of substitution of phenol for cardanol decreases the time of paper penetration, improves the spreadability of the resin on the surface of the paper, and increases the elasticity of the impregnated paper. A decrease in the paper penetration time may be due to the increased wetting ability of cardanol-containing oligomers, which exhibit surfactant properties in alkaline media. Due to the plasticizing effect

of the  $\text{C}_{15}$  hydrocarbon radical, the higher proportion of cardanol in the composition of the impregnating resin improves the elasticity of the impregnated paper.

Cardanol in the binder structure imparts barrier properties to laminated materials with respect to the action of water due to the presence of the hydrophobic  $\text{C}_{15}$  substituent in the binder structure. The Cobb value of water absorption of laminated particle boards and plywood significantly decreases when the content of cardanol increases from 0 to 30 wt % phenol (Fig. 8).

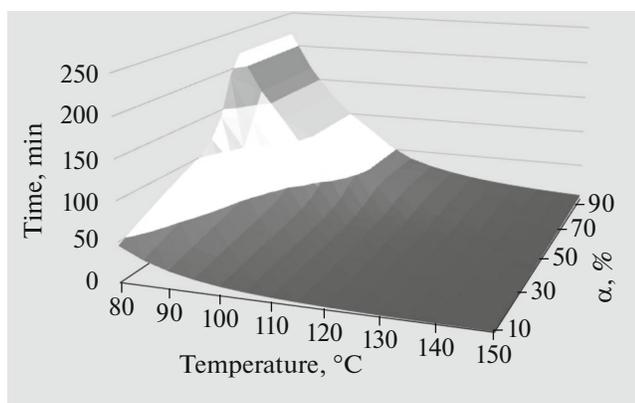


Fig. 4. Surface of the dependence of the IPFR polycondensation time on temperature and degree of conversion  $\alpha$ .

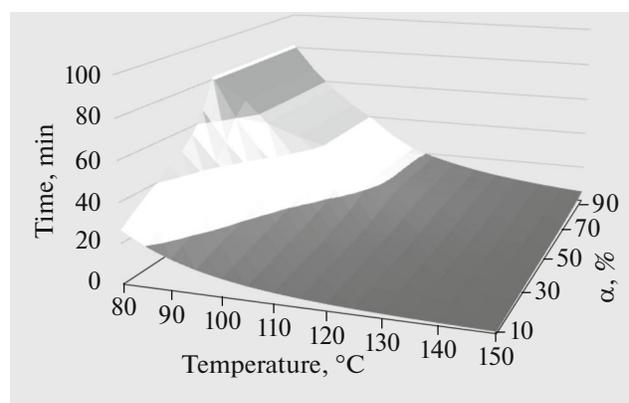
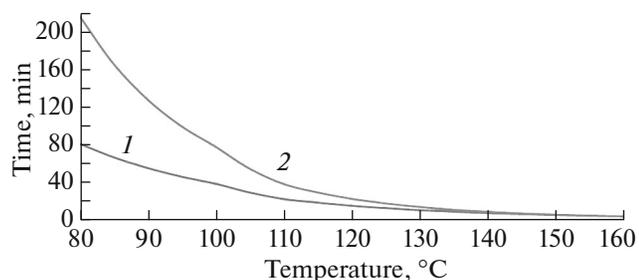
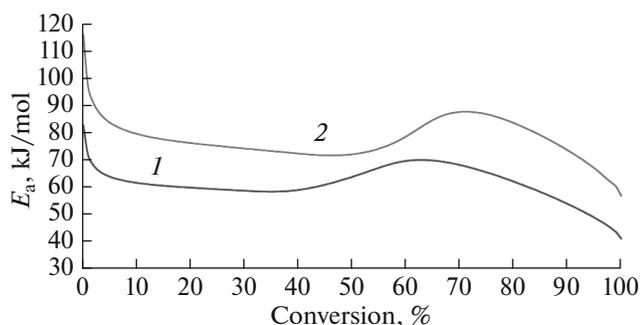


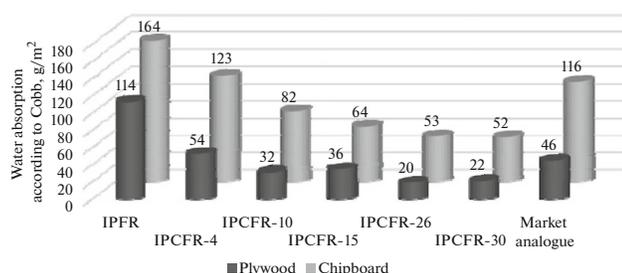
Fig. 5. Surface of the dependence of the IPCFR-10 polycondensation time on temperature and degree of conversion  $\alpha$ .



**Fig. 6.** Dependence of the polycondensation reaction time on temperature until conversion degree  $\alpha = 95\%$  is reached for (1) IPCFR-10 and (2) IPFR resins.



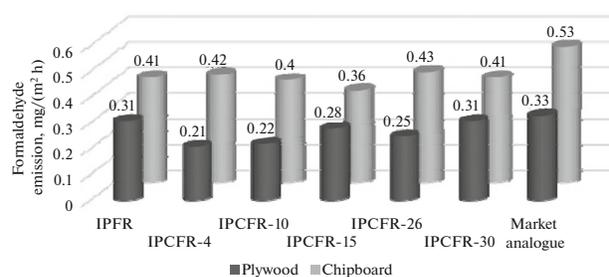
**Fig. 7.** Dependence of the change in activation energy  $E_a$  of polycondensation of (1) IPCFR-10 and (2) IPFR resins on the degree of conversion.



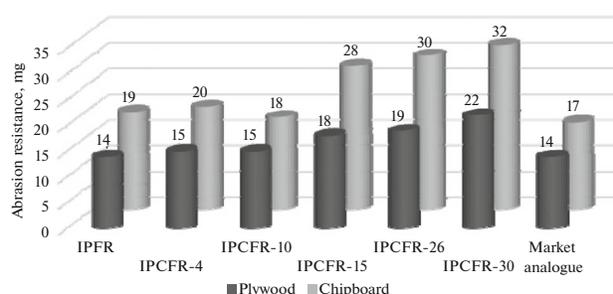
**Fig. 8.** Water absorption according to the Cobb method for samples of plywood and chipboards laminated with phenolic film with different contents of cardanol.

The use of paper impregnated with IPCFR-10 made it possible to halve the water absorption in plywood by 3.5 times and in chipboards. It should be noted that, to improve the performance of paper, the manufacture of commercially available analogues of impregnated paper involves the addition of hydrophobizers and other components to impregnating compositions, while no additional substances were introduced when using phenol–cardanol–formaldehyde resins.

The release of formaldehyde from laminated materials depends not only on the base (wood–polymer



**Fig. 9.** Emission of formaldehyde from samples of plywood and chipboards laminated with phenolic film with different contents of cardanol.



**Fig. 10.** Abrasion resistance of plywood and chipboard samples laminated with phenolic film with different contents of cardanol.

composite), but also on the laminate. Lamination of plywood with phenolic film made using cardanol-containing oligomers results in lower formaldehyde emissions compared to samples made without cardanol and using commercially available phenolic film. The best results for formaldehyde emission, determined by the gas analysis method (ISO 12460-3) for laminated plywood, 0.21 and 0.22 mg/(m<sup>2</sup> h) (29–32% reduction), are similar to the use of phenolic film made with IPCFRs containing 4 and 10 wt % cardanol (Fig. 9). In the case of laminated particle boards, a decrease in formaldehyde emission by 12 wt % (0.36 mg/(m<sup>2</sup> h)) is observed when using a phenolic film made from IPCFR containing 15 wt % cardanol.

Cardanol in the structure of the cured polymer, due to the plasticizing effect of the C<sub>15</sub> alkyl linear substituent, reduces the coating hardness and worsens the coating's resistance to abrasion, starting with samples of wood composites obtained using IPCFRs with a cardanol content of more than 15 wt % (Fig. 10). The abrasion properties of coatings of laminated plywood and particle boards made using IPCFR-10 are similar to those of composites made using IPFR and the market analogue.

## CONCLUSIONS

The effect of cardanol introduced into the structure of resole oligomers on the properties of phenolic

films and on the characteristics of laminated particle boards and plywood was studied. The introduction of cardanol into the structure of resols increases the reactivity of binders, especially in the low-temperature region, which makes it possible to optimize the technological process of laminating wood composite materials.

It was found that lamination of plywood and particle boards with cardanol-containing phenolic film significantly decreases the water absorption determined according to Cobb. The new impregnating resol resins comply with the principles of “green chemistry” and are promising for the production of laminated wood composites with improved insulating properties.

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