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DETERMINATION OF THE OPTIMAL COMPOSITION OF ENVIRONMENTALLY FRIENDLY WOOD GLUE BASED ON OXIDIZED STARCH

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Abstract

In this article, the composition, film-forming properties and structural changes of composite wood glues based on oxidized starch (OS) were studied. The compositions included polyacrylic acid (PAA), polyvinyl alcohol (PVA), citric acid and urea, and the physicomaterial properties of film samples in their different mass ratios were studied. The results of the study confirmed that the composition and ratio of components have a significant effect on the film moisture content, thickness and relative elongation. In particular, the highest elasticity and low moisture content were observed in the OS: PAA: CA system with a ratio of 60:35:5. IR-spectral analysis showed the formation of hydrogen bonds and ether bonds during the modification process. Based on the results, it was found that oxidized starch-based compositions form a dense and stable polymer structure and are a promising material for creating environmentally friendly wood glues.

Keywords: *oxidized starch; composition; glue; film; IR spectrum*

Introduction

When developing a wood glue composition, the main attention is often paid to the adhesion strength, viscosity, setting time and water resistance. However, from the point of view of modern materials science, the film-forming property of the glue is also one of the important indicators. In order for the glue to bond firmly between the wood surface,

the glue must form a uniform, dense, elastic and microcrack-free layer after drying. If the composition does not have good film-forming properties, even if its adhesion strength is satisfactory in laboratory conditions, the layer will crack during the technological process, loosening under the influence of moisture or separation under the influence of mechanical force (Cui Z., Beach E. S., Anastas P. T., 2011).

Also, in scientific articles, indicators such as film thickness, relative elongation, humidity, water vapor permeability and cracking rate have been identified when evaluating the film-forming properties of glues. In this regard, when choosing the composition of oxidized starch (OS)-based composites, film-forming ability is considered an equally important criterion as adhesion strength (Zdanowicz M., Johansson C., 2016).

Therefore, when optimizing the composition of OS-based wood adhesives, special attention should be paid to film-forming properties. Because it is the quality of the resulting composite polymer films that determines the mechanical strength, water resistance, service life and efficiency of industrial application of the adhesive.

Materials and Methods

Oxidized starch, poly(acrylic acid), poly(vinyl alcohol), citric acid ($C_6H_8O_7$), and carbamide ($CO(NH_2)_2$) were obtained from Sigma-Aldrich and used as received.

Composition of oxidized starch-based compositions and obtaining films based on them. The following were used in the development of the composition of the composite adhesive based on OS: polyvinyl alcohol (PVA), polyacrylic acid (PAA), citric acid (CA) and urea (U). To determine the composition of the composite adhesive based

on OS with these selected substances, they were mixed in different mass ratios and film samples were taken from 10% aqueous solutions.

Film moisture content was determined by drying the samples at 105 °C until constant mass was reached, and the mass loss was estimated as a percentage (Sanyang M. L., Sapuan S. M., Jawaid M., 2015).

Film thickness was measured at various points on the film using a digital micrometer and calculated based on the average value (Rhim J. W., Wang L. F., 2013).

Film elongation was determined by stretching the sample to break on a universal testing machine, and the elongation at break was expressed as a percentage (Ma X., Chang P. R., Yu J., 2008).

IR spectroscopy. IR spectra of the samples were recorded on a Perkin Elmer 2000 IR Fourier spectrometer in the frequency range of 400–4000 cm^{-1} in a tablet with KBr (Radjabov O. I., Yariev O. O., Azimova L. B., Filatova A. V., 2025).

Results and Discussion

To determine the optimal composition of the composite adhesive based on OS with the selected substances, the physical and mechanical properties of film samples obtained from their 10% aqueous solutions were studied. The experimental results are presented in Table 1.

Table 1. Effect of composition and mass ratio on the properties of films obtained from OS and its based compositions

No.	Composition			Film properties	
	Compound	Mass ratio	Humidity, %	Thickness, μm	Relative elongation, %
1.	OS	100	14,3±0,3	170±10	14±1
2.	OS: PAA: PVA	80:10:10	11,9±0,3	150±7	19±1
	OS: PAA: PVA	70:15:15	11,4±0,2	150±7	23±1
	OS: PAA: PVA	60:20:20	10,6±0,2	130±5	26±1
	OS: PAA: PVA	50:25:25	9,7±0,2	120±5	28±2
3.	OS: PAA: CA	85:13:2	10,7±0,3	140±7	20±1
	OS: PAA: CA	75:22:3	10,3±0,2	140±7	26±1
	OS: PAA: CA	70:25:5	9,1±0,2	120±5	29±1
	OS: PAA: CA	60:35:5	8,4±0,2	120±5	33±1
4.	OS: PAA: PVA: U	80:8:8:4	11,2±0,3	140±6	20±1
	OS: PAA: PVA: U	70:12:12:6	9,8±0,2	130±6	23±1

No.	Composition		Film properties		
	Compound	Mass ratio	Humidity, %	Thickness, μm	Relative elongation, %
	OS: PAA: PVA: U	60:20:15:5	9,2 \pm 0,2	120 \pm 5	27 \pm 1
	OS: PAA: PVA: U	60:15:15:10	8,6 \pm 0,2	120 \pm 5	22 \pm 1

The experimental results presented in Table 1 confirm that the composition of the components and their mass ratios in the compositions based on OS have a significant effect on the film-forming properties. The OS film obtained as a control showed a moisture content of 14.3 \pm 0.3%, a thickness of 170 \pm 10 μm , and a relative elongation of 14 \pm 1%. This result is explained by the high hydrophilicity of OK and weak interchain hydrogen bonds. In the OK: PAA: PVA compositions, an improvement in the film properties was observed with an increase in the amount of components.

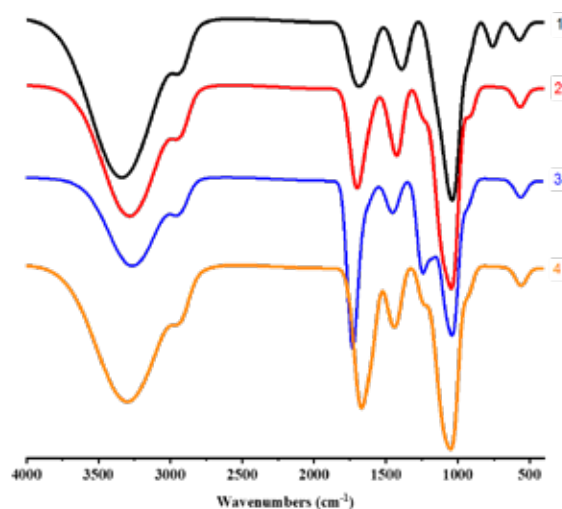
In particular, in the ratio of 80:10:10, the moisture content was 11.9 \pm 0.3%, the elongation was 19 \pm 1%, while in the ratio of 70:15:15, it was 11.4 \pm 0.2% and 23 \pm 1%, respectively. At a ratio of 60:20:20, the moisture content decreased to 10.6 \pm 0.2%, and the film elongation reached 26 \pm 1%. The highest result in samples with this composition was observed at a ratio of 50:25:25, with a moisture content of 9.7 \pm 0.2%, a thickness of 120 \pm 5 μm , and a film elongation of 28 \pm 2%. In the OS: PAA: CA system, more significant changes were observed, with a ratio of 85:13:2, the film elongation reached 20 \pm 1%, while at 75:22:3 it reached 26 \pm 1%, and at 70:25:5 it reached 29 \pm 1%. The most optimal result was determined at a ratio of 60:35:5, with a moisture content of 8.4 \pm 0.2%, and a film thickness of 120 \pm 5 μm . The relative elongation of the film was observed to reach 33 \pm 1%. Thus, it can be concluded that the composition with this composition has a dense and stable structure.

In samples of the composition with the composition OK: PAA: PVA: U, the plasticizing effect of U was observed. In the ratio of 80:8:8:4, the relative elongation of the film was 20 \pm 1%, in the ratio of 70:12:12:6–23 \pm 1%. In the composition with the composition OS: PAA: PVA: U, the optimal result was observed in the ratio 60:20:15:5, the film moisture content was 9.2 \pm 0.2%, the thick-

ness was 120 \pm 5 μm , and the relative elongation was 27 \pm 1%. An adverse effect on the properties of the foam was observed when the amount of U was increased (in the mass ratio of 60:15:15:10).

The functional groups and their interactions in the obtained compositions with the optimal composition were studied using the IR-spectral method. The analysis results made it possible to determine the structural changes that occur during the modification process. The IR spectra presented in Figure-1 below clearly show how the molecular interactions between components change in composite systems based on OS.

Figure 1. IR spectra of the compositions: 1-OS, 2-OS: PAA: PVA, 3-OS: PAA: CA, 4-OS: PAA: PVA: U



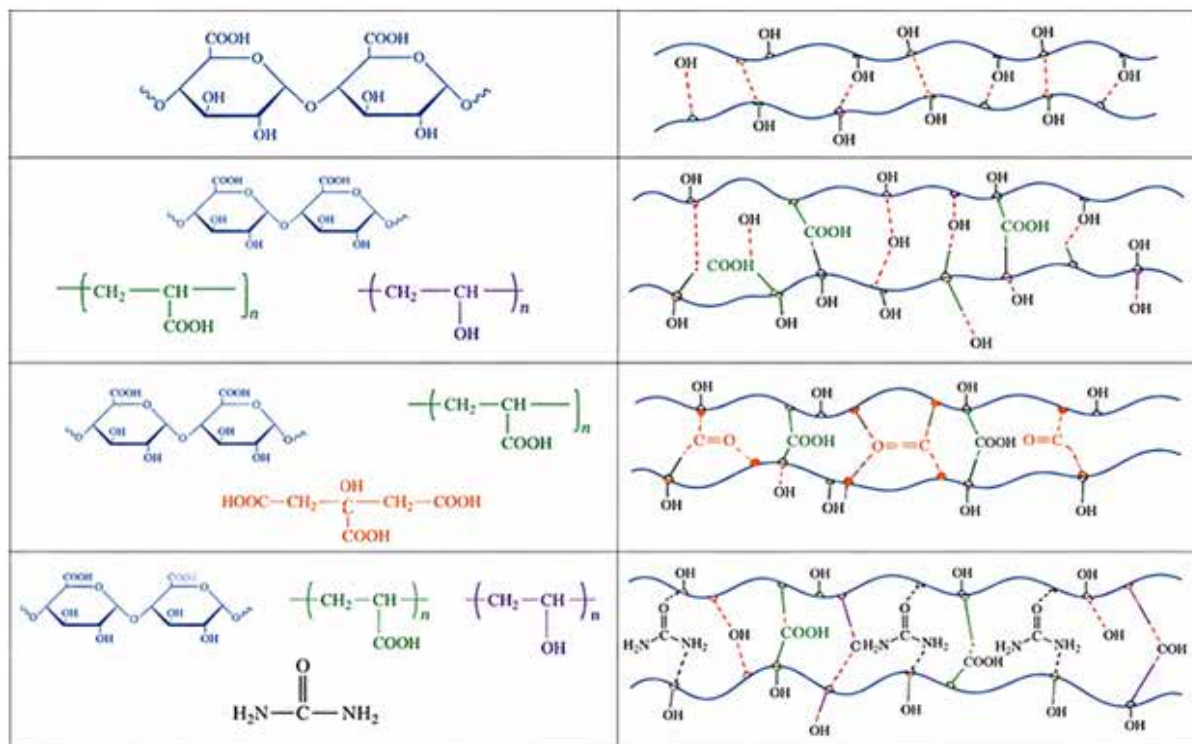
As can be seen from Figure 1, the broad absorption line observed in the 3200–3500 cm^{-1} region for all samples belongs to the –OH groups, indicating the presence of hydrogen bonds in the samples. In the OS sample (spectrum 1 in Figure 1), this absorption is relatively broad and intense, indicating the presence of a large number of free hydroxyl groups in the molecular chains and confirming its high hydrophilicity and relatively loose structure.

In the OK: PAA: PVA sample (spectrum 2) in Figure-1, a slight decrease and a shift to a lower frequency of the absorption in this region were observed. This result indicates the formation of strong hydrogen bonds between the components. The functional groups in the PVA and PAA components interact with OS, reducing the interchain distance and leading to a denser structure. The C–H stretching vibrations in the 2800–3000 cm^{-1} region are almost unchanged in all spectra, indicating that the main hydrocarbon skeleton is preserved. However, significant changes were observed in the 1700–1750 cm^{-1} region. In the OS: PAA: PVA system, there are absorptions in this region related to the carboxyl (C=O) groups of PAA. In the OS: PAA: CA (spectrum 3) composition sample, a clear and strong absorption appeared in the region of 1725–1740 cm^{-1} , which is characteristic of ester groups (formed as a result of etherification), confirming that an etherification reac-

tion occurred in the presence of CA, that is, a chemical crosslinking (covalent bonding).

As a result, this composition formed a more dense structure and improved mechanical properties. Intense absorptions in the regions of 1000–1200 cm^{-1} belong to C–O–C and C–O bonds, and these absorptions were observed in all examined samples. However, unlike the OS sample, a partial increase in the absorption intensity was observed in its modified compositions. This result can be explained by the increased interaction between polymer chains. In the OS: PAA: PVA: U sample in spectrum 4, the absorptions in the 1650–1680 cm^{-1} and 1550 cm^{-1} regions are vibrations characteristic of the carbonyl (C=O) and N–H groups of urea, indicating that U forms hydrogen bonds with other components in all the composition and acts as a plasticizer of the system. The results of the IR spectra analysis confirm that the structural changes in OS-based compositions have formed a stable structure.

Figure 2. Molecular interactions and structural formation mechanisms of OS-based composites



Based on the results of the initial study, an optimal composition of the OS-based composite was selected and structural schemes of molecular interactions between the components were proposed (Figure 2).

Conclusion

The results of the study showed that the physical, mechanical and structural properties of composite adhesives based on oxidized starch depend on the composition and mass ratio of the components. The addition of

PAA, PVA and citric acid increased the elasticity and density of the films and reduced their moisture content. The best result was observed in the ratio of the OS: PAA: CA system 60:35:5. IR-spectral analysis confirmed

the formation of hydrogen and ether bonds. The results obtained showed that these compositions are promising for creating environmentally friendly, biodegradable and highly effective wood adhesives.

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