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STRUCTURE AND PROPERTY RELATIONSHIPS IN OXIDIZED STARCH BASED COMPOSITE WOOD ADHESIVES

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Abstract

Composite wood adhesives based on oxidized starch were prepared using different formulations, including OS: PAA: PVA, OS: PAA: CA, and OS: PAA: PVA: U systems. The relationship between structure and properties was investigated using scanning electron microscopy (SEM) and thermogravimetric analysis (TG). SEM analysis revealed that the OS: PAA: CA system forms a dense, homogeneous, and compact structure, while the OS: PAA: PVA and OS: PAA: PVA: U systems exhibit more heterogeneous and porous morphologies. Thermogravimetric results showed that all samples undergo three main stages of thermal degradation, with the OS: PAA: CA composition demonstrating the highest thermal stability due to the formation of a well-developed three-dimensional network. The results indicate that the performance of oxidized starch-based adhesives strongly depends on the composition and intermolecular interactions between components. Among the studied systems, the OS: PAA: CA composition exhibited the most favorable structural and thermal properties, making it a promising candidate for wood adhesive applications.

Keywords: *oxidized starch; composite adhesives; wood adhesive; SEM analysis; thermal stability; structure–property relationship*

Indroduction

The development of functional materials based on natural polymers has gained significant attention due to environmental concerns and the demand for renewable resources. Natural macromolecules offer versatile chemical modification and tunable physicochemical properties, making them attractive for advanced applications. Starch, one of the most abundant and cost-effective polysaccharides, exhibits biodegradability and film-forming ability. However, its practical use is limited by low mechanical strength, high moisture sensitivity, and poor thermal

and adhesive properties (Liang, J., Li, D., Luo, Z., Yang, Y., Meng, T., Chen, C., Li, H., Zuo, N., Li, Q., Yang, H., & Wu, Z., 2025).

Oxidation is an effective strategy to overcome these limitations, introducing reactive aldehyde and carboxyl groups into the starch structure. This enhances its reactivity and enables stronger intermolecular interactions, allowing its use in composite systems. Incorporation of additional polymers further improves material performance through hydrogen bonding and possible chemical crosslinking, which govern morphology, viscosity, thermal stability, and adhesion. This study aims to synthesize oxidized starch-based composite wood adhesives and to investigate the relationship between their structure and physicochemical properties (Shen, D., Li, X., Liu, Y., Liu, C., Zheng, A., Wang, X., Liu, Y., & Liu, G., 2023).

Materials and Methods

Starch, sodium periodate ($NaIO_4$), 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.

Preparation of oxidized starch. Oxidized starch was prepared using sodium hypochlorite (NaClO). A 5 wt% starch suspension (50 g starch in 450 g water) was ultrasonically treated (20 kHz, 400 W, 25 °C, 10 min), followed by pH adjustment to 9.5 using 1 M NaO H. The reaction was carried out at 35 °C with dropwise addition of 18% NaClO (starch: active chlorine ratio = 100:1––100: 3), while maintaining pH at 9.5 using 1 M H_2SO_4. After 30 min, the product was filtered, washed with distilled water, and dried at 50 °C for 12 h. The carboxyl group content was determined by the calcium acetate method. A 1 g dried sample was treated with 60 mL of 0.25 M $Ca(CH_3COO)_2$ and 100 mL of water and stirred for 6 h. A 25 mL aliquot was titrated with 0.01 M NaOH using bromothymol blue as an indicator. All measurements were performed in triplicate (Palić, N., Gržetić, J., Đolić, M., Kovačević, T., Pecić, Lj., Radovanović, Ž., & Marinković, A., 2020).

Preparation of composite adhesives. Composite adhesives were prepared using OS: PAA: PVA, OS: PAA: CA, and OS: PAA: PVA: U formulations. The components were dissolved or dispersed in distilled water and mixed under continuous stirring until homogeneous systems were obtained. The mixtures were further stirred to ensure uniform distribution and promote intermolecular interactions (Wang, P., He, H., Cai, R., Zhang, X., Chen, J., Tao, G., & Xiang, H., 2019).

Scanning electron microscopy (SEM). The surface morphology of the samples was examined using a scanning electron microscope (EVO MA10). Prior to analysis, the samples were coated with a thin conductive layer (~10 nm) using a Quorum Q150R ES sputter coater. The images were obtained at an accelerating voltage of 15 kV using SmartSEM software.

Thermogravimetric analysis (TG). Thermal stability of the samples was evaluated using thermogravimetric analysis. The measurements were carried out in the temperature range of 30–600 °C at a heating rate of 10 °C/min under an air atmosphere (McHugh, T. H., Avena-Bustillos, R., & Krochta, J. M., 1993).

Results and Discussion

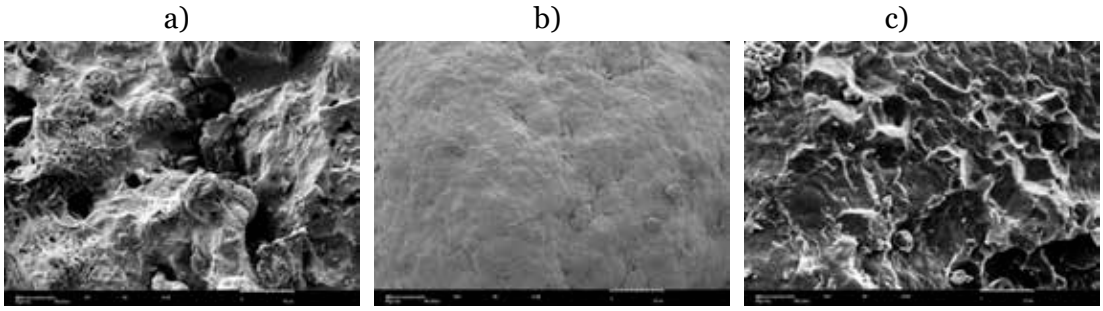
Composite adhesives with different compositions were synthesized based on oxidized starch. During the preparation process, physicochemical interactions occurred between the components, leading to the formation of three-dimensional networks with varying degrees of crosslinking.

In the OS: PAA: PVA system, the network structure was mainly formed through hydrogen bonding between hydroxyl and carboxyl groups, resulting in a physically crosslinked network. Due to the absence of strong chemical crosslinking, the resulting structure was relatively loose and less dense. In the OS: PAA: CA composition, citric acid acted as a crosslinking agent, promoting strong intermolecular interactions between polymer chains. As a result, a dense and stable three-dimensional network structure was formed. In the OS: PAA: PVA: U system, urea acted as a plasticizer, reducing intermolecular interactions and increasing chain mobility. Although this enhanced the flexibility of the system, it led to a decrease in structural compactness.

The structural characteristics of the composite adhesives were investigated by scanning electron microscopy (SEM) in order to

evaluate their surface morphology, phase compatibility, degree of component dispersion, porosity, and the presence of microstructural defects. These parameters are directly related to the practical performance of adhesives, particularly the integrity of the adhesive layer, cohesive strength, and operational stability.

Figure 1. *SEM micrographs of oxidized starch based composite adhesives: (a) OS: PAA: PVA, (b) OS: PAA: CA, and (c) OS: PAA: PVA: U*

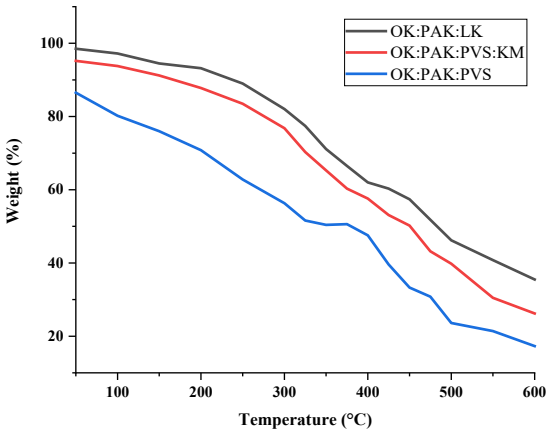


A comparative analysis revealed that the morphology of the oxidized starch-based composites strongly depends on their composition and the nature of intermolecular interactions. The OS: PAA: PVA system exhibited a heterogeneous and irregular surface with partially porous features. The presence of fibrillar elements, microvoids, and non-uniform regions indicates that a fully homogeneous phase was not formed. In this system, hydrogen bonding between hydroxyl groups of PVA and functional groups of oxidized starch and polyacrylic acid leads to the formation of a physically crosslinked network. However, due to the absence of strong chemical crosslinking, the resulting structure is relatively loose and less compact, which may reduce cohesive strength and water resistance.

In contrast, the OS: PAA: CA composition exhibited a smooth, homogeneous, and dense structure. This behavior can be attributed to the role of citric acid as a multifunctional crosslinking agent. Its carboxyl groups interact strongly with the functional groups of oxidized starch and polyacrylic acid, leading to the formation of a compact three-dimensional network. As a result, the structural integrity, mechanical strength, and resistance to environmental factors are significantly improved. The OS: PAA: PVA: U system displayed a rough, granular, and partially porous morphology. The surface contained irregular domains, protrusions, and microvoids, indicating incomplete structural compactness.

The presence of urea acts as a plasticizer, reducing intermolecular interactions and increasing chain mobility. Consequently, the structural density decreases, leading to partial heterogeneity and increased porosity, which may negatively affect the water resistance of the material. Overall, the SEM analysis confirms that the morphological structure of oxidized starch-based composite adhesives is closely related to their composition and the degree of intermolecular interactions.

Figure 2. *Thermogravimetric (TG) curves of oxidized starch based composite adhesives with different compositions (OS: PAA: CA, OS: PAA: PVA: U, and OS: PAA: PVA)*



Systems with a dense and homogeneous structure exhibit improved mechanical and эксплуатацион (operational) properties, with the OS: PAA: CA composition showing the most optimal structural characteristics. In the next stage of this study, the thermal stability of the composite adhesives was evaluated by thermogravimetric analysis (TG).

The results showed that all samples exhibited three main stages of mass loss:

evaporation of moisture and volatile components at 30–150 °C, degradation of the main polymer chains at 180–350 °C, and carbonization processes at 350–600 °C. Significant differences were observed among the systems. The OS: PAA: PVA composition exhibited the lowest thermal stability due to the lack of strong chemical crosslinking. The OS: PAA: PVA: U system showed moderate stability as a result of the plasticizing effect. The highest thermal stability was observed for the OS: PAA: CA composition, which is attributed to the formation of a dense three-dimensional network.

Conclusion

Composite wood adhesives based on oxidized starch with different compositions were synthesized, and their structure–property relationships were investigated. The results showed that composition determines morphological structure and thermal stability, where denser and more homogeneous systems exhibit higher thermal resistance. The OS: PAA: CA system formed a compact structure and showed the highest thermal stability, while OS: PAA: PVA and OS: PAA: PVA: U systems exhibited more porous structures and lower stability. Overall, the performance of the adhesives is governed by intermolecular interactions, with OS: PAA: CA showing the most optimal properties.

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