

## INFLUENCE OF DIFFERENT ORGANIC ACIDS ON CHARACTERISTICS OF LIGNIN-BASED ADHESIVES FROM BREADFRUIT SKIN BIOREFINERY

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**Abstract.** This study aims to analyze and synthesize research findings related to the effect of various types of organic acids on the characteristics of breadfruit (*Artocarpus altilis*) peel lignin as a raw material for bio-based adhesives and to identify its potential use as an alternative environmentally friendly adhesive. The study uses a literature study method sourced from national and international scientific articles obtained through the Scopus, ScienceDirect, Google Scholar, and SpringerLink databases with a publication range of 2014–2024. The analysis was conducted on studies discussing lignin extraction, precipitation processes using organic acids, lignin characterization, and its application in lignin-based adhesive formulations. In addition, a bibliometric analysis using VOSviewer on 114 publications was conducted to map research developments, keyword trends, and the relationship between dominant research themes. The results of the bibliometric analysis indicate that the topics of lignin, bio-based adhesives, biomass valorization, and organic acid treatment have become the main focus of research in the last decade. The results of literature synthesis indicate that the use of organic acids, such as acetic acid, maleic acid, and citric acid, produces different lignin characteristics, especially in chemical structure, phenolic group content, thermal stability, and adhesive reactivity. Characterization using FTIR, TGA, NMR, and SEM indicates that breadfruit peel lignin has high potential as a raw material for bio-based adhesives for more sustainable wood and plywood applications.

**Keywords:** Organic Acids, Lignin, Breadfruit Skin, Bio-Based Adhesives, Biorefinery

**Abstrak.** Penelitian ini bertujuan untuk menganalisis dan mensintesis temuan penelitian terkait pengaruh berbagai jenis asam organik terhadap karakteristik lignin kulit sukun (*Artocarpus altilis*) sebagai bahan baku perekat bio-based serta mengidentifikasi potensi pemanfaatannya sebagai alternatif perekat ramah lingkungan. Penelitian menggunakan metode studi literatur yang bersumber dari artikel ilmiah nasional dan internasional yang diperoleh melalui database Scopus, ScienceDirect, Google Scholar, dan SpringerLink dengan rentang publikasi tahun 2014–2024. Analisis dilakukan terhadap penelitian yang membahas ekstraksi lignin, proses presipitasi menggunakan asam organik, karakterisasi lignin, serta aplikasinya pada formulasi perekat berbasis lignin. Selain itu, analisis bibliometrik menggunakan VOSviewer terhadap 114 publikasi dilakukan untuk memetakan perkembangan penelitian, tren kata kunci, dan keterkaitan tema riset yang dominan. Hasil analisis bibliometrik menunjukkan bahwa topik lignin, bio-based adhesive, biomass valorization, dan organic acid treatment menjadi fokus utama penelitian dalam satu dekade terakhir. Hasil sintesis literatur menunjukkan bahwa penggunaan asam organik, seperti asam asetat, asam maleat, dan asam sitrat, menghasilkan karakteristik lignin yang berbeda, terutama pada struktur kimia, kandungan gugus fenolik, stabilitas termal, dan reaktivitas perekat. Karakterisasi menggunakan FTIR, TGA, NMR, dan SEM mengindikasikan bahwa lignin kulit sukun memiliki potensi tinggi sebagai bahan baku perekat bio-based untuk aplikasi kayu dan plywood yang lebih berkelanjutan.

**Kata Kunci:** Asam Organik, Lignin, Kulit Sukun, Perekat Bio-Based, Biorefinery

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## INTRODUCTION

Lignin, a complex aromatic polymer abundantly found in plant cell walls, plays a crucial role in providing mechanical strength and resistance to biological degradation in plants. The utilization of lignin as a renewable resource has gained significant attention, particularly in the development of sustainable materials such as bio-based adhesives. Breadfruit skin represents a promising lignocellulosic biomass source because it contains considerable amounts of lignin, cellulose, and hemicellulose that can be valorized into high-value products (Rizal et al., 2024). Breadfruit peel waste also contains high starch content and has potential as a renewable biomass material (Adhitama et al., 2023; Tira et al., 2023). The large volume of delignified breadfruit skin generated by the breadfruit industry is often underutilized, thus isolating lignin from this waste not only adds value but also aligns with the principles of the circular economy (Sangjan & Widjaja, 2022).

The isolation of lignin from delignified breadfruit skin is commonly performed through precipitation methods typically involves a precipitation method, wherein lignin is separated from the cooking liquor (black liquor) by reducing the pH using acids (Zakaria et al., 2022). This method takes advantage of lignin's solubility changes with pH, enabling it to precipitate as a solid and be separated from other components in the solution. Acid treatment is commonly applied in biomass hydrolysis and conversion processes due to its effectiveness in breaking down lignocellulosic structures (Tira et al., 2023). The use of organic acids, particularly carboxylic acids such as acetic acid, maleic acid, and citric acid, has gained popularity due to their more environmentally friendly properties compared to inorganic acids (Sangjan & Widjaja, 2022; Zakaria et al., 2022). Moreover, the type of acid used can influence the characteristics of the resulting lignin, offering opportunities to tailor its properties for specific applications (Rizal et al., 2024).

Several studies have demonstrated that the type of acid used during lignin precipitation significantly influences the chemical and physical characteristics of isolated lignin, including purity, molecular weight, functional group distribution, and thermal stability (del Río et al., 2013; Zakaria et al., 2022). Citric acid, which contains three carboxyl groups, can form stronger hydrogen-bonding interactions with lignin, potentially improving lignin yield and stability (Sangjan & Widjaja, 2022). Meanwhile, maleic acid possesses a double-bond structure that may participate in lignin modification reactions during the isolation process, thereby enhancing lignin reactivity and adhesive performance (Zakaria et al., 2022). Although previous studies have reported the effects of various organic acids on lignin recovery and modification, most investigations have focused on conventional lignocellulosic feedstocks such as wood residues,

sugarcane bagasse, corn stover, and oil palm biomass. Furthermore, these studies generally evaluated lignin extraction efficiency or chemical characterization separately, with limited attention to how different organic acids influence the suitability of lignin for bio-based adhesive applications. Research on breadfruit skin biomass remains particularly scarce, despite its high lignin content and abundance as an underutilized agro-industrial waste. To date, no comprehensive synthesis has specifically compared the effects of acetic acid, maleic acid, and citric acid precipitation on the physicochemical characteristics of lignin derived from breadfruit skin biorefinery and their implications for adhesive performance. Therefore, this study addresses this gap by systematically reviewing and synthesizing existing evidence regarding the role of different organic acids in modifying breadfruit-skin-derived lignin and evaluating its potential as a sustainable bio-based adhesive material.

Lignin isolated from breadfruit skin possesses a distinctive structure predominantly composed of guaiacyl (G) units, accompanied by smaller proportions of p-hydroxyphenyl (H) and syringyl (S) units, as well as p-hydroxybenzoate and acetate substituents attached to the lignin side chains (del Río et al., 2013). These structural characteristics contribute to favorable mechanical and chemical properties for the development of durable bio-based adhesives. In addition, breadfruit skin lignin has the potential to be processed into nanolignin, which may enhance adhesive interactions, dispersion, and mechanical strength within adhesive matrices (Oliveira et al., 2025; Rizal et al., 2024). Therefore, This study aims to review, synthesize, and compare findings regarding the effect of different organic acids on the isolation and characteristics of lignin derived from breadfruit skin biomass and to evaluate its potential as a renewable raw material for sustainable bio-based adhesives. Through this approach, the study is expected to provide scientific insights into environmentally friendly lignin isolation strategies while supporting the utilization of agro-industrial waste into value-added green materials for adhesive and plywood applications.

## **METHOD**

The literature review in this study was conducted systematically through several stages, including identification, selection, evaluation, and synthesis of relevant scientific literature related to lignin extraction, organic acid precipitation, and lignin-based adhesive applications derived from breadfruit skin biomass. Scientific publications were collected from reputable databases such as Google Scholar, ScienceDirect, SpringerLink, and Scopus using keywords including “breadfruit skin lignin,” “organic acid precipitation,” “bio-based adhesive,” “lignin characterization,” and “biorefinery biomass.” The inclusion criteria consisted of peer-reviewed

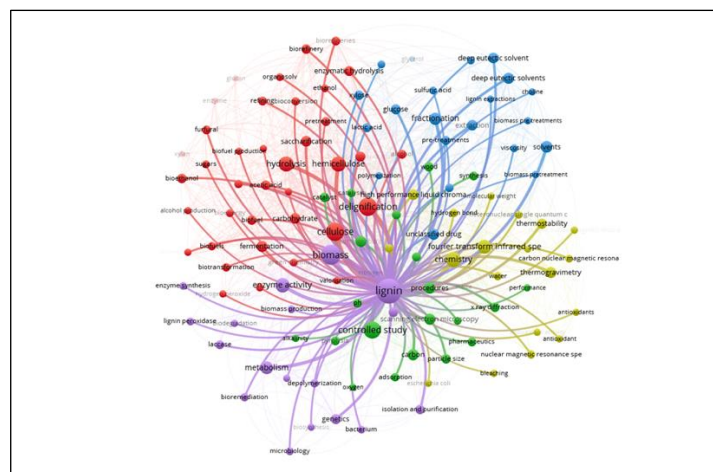
journal articles, conference papers, and scientific reports published within the last ten years that discussed lignin isolation methods, lignin characterization, and adhesive applications. Articles that were not directly related to lignin precipitation or adhesive development were excluded from the analysis.

The selected literature was then analyzed descriptively and comparatively to identify research trends, differences in precipitation methods, the influence of various organic acids on lignin properties, and the potential application of lignin as a sustainable adhesive material. In addition, bibliometric analysis using VOSviewer was conducted to map publication trends, keyword co-occurrence, and dominant research themes in lignin-based adhesive studies. The collected data were further categorized based on lignin source, precipitation agent, characterization techniques, and adhesive performance to provide a comprehensive understanding of current developments and research gaps in sustainable lignin-based adhesive technology.

## RESULTS

### Bibliometric Analysis

Bibliometric analysis was conducted to explore dominant research themes and conceptual structures related to the development of lignin-based adhesives derived from breadfruit skin biorefinery processes. The analysis was performed using VOSviewer version 1.6.20, based on a keyword co-occurrence network generated from 114 selected scientific publications. The visualization was developed using bibliographic data, with keywords as the unit of analysis. A minimum threshold of 5 keyword occurrences was applied, and all items meeting this criterion were included in the mapping. This analysis helps to identify major clusters, frequently used terms, and interconnections within the research landscape.



**Figure 1.** A network visual map

Figure 1 illustrates a network visual map generated by keyword co-occurrence analysis of the selected articles on lignin-based adhesives from breadfruit skin biorefinery. The visualization presents five overarching thematic clusters, each represented by a different color. The red cluster contains keywords such as “biorefinery,” “ethanol,” “fermentation,” and “enzymatic hydrolysis,” reflecting a strong focus on bioconversion processes and biofuel production from lignocellulosic biomass. The green cluster is characterized by terms like “delignification,” “controlled study,” “adsorption,” and “cellulose,” suggesting research interest in chemical characterization and process optimization of biomass and lignin-derived materials.

Meanwhile, the blue cluster highlights keywords such as “deep eutectic solvents,” “extraction,” and “fractionation,” indicating attention to pretreatment technologies and environmentally friendly solvent systems for lignin isolation. The purple cluster includes terms such as “enzyme activity,” “laccase,” “biodegradation,” and “microbiology,” which are associated with enzymatic and microbial pathways in lignin degradation. Lastly, the yellow cluster involves keywords like “thermogravimetry,” “Fourier transform infrared spectroscopy,” and “nuclear magnetic resonance,” pointing to thermal and structural analyses of lignin-based materials. The overall network organization reveals interconnectedness among biomass transformation, pretreatment methods, enzymatic activity, and advanced material characterization techniques.

**Table 1.** Summary of five major clusters were Identified

Cluster	Primary Focus	Key Keywords
Cluster 1 (Red)	Bioconversion and biofuel production	biorefinery, ethanol, fermentation, enzymatic hydrolysis, saccharification
Cluster 2 (Green)	Chemical characterization and optimization	delignification, controlled study, adsorption, cellulose, biomass
Cluster 3 (Blue)	Pretreatment and solvent systems	deep eutectic solvents, extraction, fractionation, solvents, biomass pretreatment
Cluster 4 (Purple)	Enzymatic/microbial degradation	enzyme activity, laccase, biodegradation, lignin peroxidase, microbiology
Cluster 5 (Yellow)	Structural and thermal analysis	thermogravimetry, FTIR, nuclear magnetic resonance, thermal stability, x-ray diffraction

Table 1 presents the results of the bibliometric analysis, which identified five major research clusters related to lignin extraction, biomass processing, and lignin-based adhesive development. Cluster 1 (red) mainly focuses on bioconversion and biofuel production, emphasizing keywords such as biorefinery, ethanol, fermentation, enzymatic hydrolysis, and saccharification, indicating strong attention toward biomass utilization for renewable energy. Cluster 2 (green) highlights studies on chemical characterization and optimization, including

delignification, adsorption, cellulose, and biomass processing. Cluster 3 (blue) is associated with pretreatment and solvent systems, particularly the application of deep eutectic solvents, extraction, and biomass fractionation techniques. Cluster 4 (purple) emphasizes enzymatic and microbial degradation processes involving laccase, lignin peroxidase, and biodegradation mechanisms. Meanwhile, Cluster 5 (yellow) focuses on structural and thermal analysis of lignin using characterization techniques such as FTIR, thermogravimetry, nuclear magnetic resonance (NMR), and X-ray diffraction. Overall, these clusters demonstrate that current research trends are dominated by biomass valorization, lignin characterization, green extraction technologies, and the development of sustainable lignin-based materials.

## DISCUSSION

This section synthesizes the findings from both the systematic literature review and the bibliometric analysis to provide a comprehensive overview of the current state of research on the development and application of lignin-based adhesives derived from breadfruit skin biorefinery processes. By combining qualitative insights and quantitative mapping, this discussion aims to capture the depth and scope of existing knowledge in the field. It highlights emerging research trends, uncovers gaps that remain underexplored, and identifies areas with significant potential for further investigation. Additionally, the integration of both methods allows for a more robust interpretation of how the research landscape has evolved and what directions future studies might pursue to advance sustainable and bio-based adhesive technologies.

### Lignin

The bibliometric analysis identified lignin as the most frequently occurring keyword and a central research node, indicating its strategic role in biomass valorization and sustainable material development. The keyword network further showed strong associations between lignin, biomass, bioadhesives, and renewable resources, reflecting the growing scientific interest in replacing petroleum-derived adhesives with lignin-based alternatives. However, only a small proportion of publications specifically addressed lignin derived from breadfruit biomass, suggesting that this feedstock remains underexplored despite its considerable potential.

Lignin is a highly cross-linked polyphenolic polymer naturally present in lignocellulosic biomass such as agricultural residues, fruit peels, and agro-industrial waste. Breadfruit peel biomass has been widely explored as a renewable raw material for sustainable material

production (Adhitama et al., 2023). The extraction of lignin is a critical step in biorefinery schemes, as it enables the production of value-added chemicals, bio-based adhesives, and polymeric materials (Rinaldi et al., 2024). Conventional lignin isolation techniques are dominated by alkaline delignification, such as the Kraft and soda processes, where lignocellulose is treated with NaOH or Na<sub>2</sub>S at elevated temperatures to cleave ether and ester bonds between lignin and polysaccharides (Irawan et al., 2018; Deuss et al., 2023).

More environmentally benign methods such as organosolv extraction, ionic liquid, and deep eutectic solvent (DES) systems are increasingly explored due to their ability to preserve lignin's native structure and functionality (Lobato-Rodríguez et al., 2023). This trend is also reflected in the bibliometric results, which show a growing occurrence of keywords related to green extraction technologies and sustainable processing methods during recent years. Such developments indicate a shift in research focus from conventional lignin recovery toward environmentally friendly approaches that improve lignin quality and functionality for advanced applications.

## **Preparation**

### *Preparation of Lignin-Based Adhesives*

The bibliometric analysis identified lignin extraction, biomass valorization, and bio-based adhesives as major research themes, indicating growing interest in sustainable alternatives to petroleum-based adhesives. However, studies focusing specifically on breadfruit skin as a lignin source remain limited. This finding highlights the potential of breadfruit skin biorefinery residues as an underutilized raw material for lignin-based adhesive production. Based on the reviewed literature, the preparation of lignin-based adhesives generally involves alkaline delignification followed by organic acid precipitation. Organic acids such as acetic acid, maleic acid, and citric acid are used to reduce the pH of black liquor, causing dissolved lignin to precipitate. Compared with inorganic acids, these organic acids are considered more environmentally friendly and better able to preserve lignin's reactive functional groups, including phenolic hydroxyl and carboxyl groups, which are important for adhesive performance (Rahmawati et al., 2024; Vazquez Torres et al., 2025).

The synthesis of previous studies indicates that the type of organic acid influences the physicochemical properties of the recovered lignin. Citric acid tends to enhance hydrogen-bonding interactions and lignin stability, while maleic acid may increase lignin reactivity due to its unsaturated structure. Acetic acid, on the other hand, is widely used because of its effectiveness and mild reaction conditions. These differences affect lignin functionality,

crosslinking potential, and ultimately the quality of the resulting bio-based adhesive, demonstrating the importance of selecting appropriate precipitation agents in lignin valorization processes.

### **Characterization**

The bibliometric analysis identified lignin characterization as one of the most frequently studied topics in lignin valorization research, reflecting its importance in determining the suitability of lignin for bio-based adhesive applications. The reviewed studies consistently emphasized the need to evaluate the chemical structure, functional groups, thermal stability, and morphology of lignin to predict its adhesive performance and reactivity. Based on the literature, Fourier-transform infrared spectroscopy (FTIR) and nuclear magnetic resonance (NMR) are the most commonly used techniques to identify lignin functional groups and aromatic structures. These analyses reveal the presence of phenolic hydroxyl, methoxy, and carboxyl groups that play important roles in adhesive bonding and crosslinking reactions (Latif et al., 2023; Hu et al., 2021). In addition, gel permeation chromatography (GPC) is frequently employed to determine molecular weight distribution, which influences lignin processability and adhesive formulation.

Thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and scanning electron microscopy (SEM) are further used to evaluate thermal stability and surface morphology. The reviewed studies indicate that lignin precipitated using organic acids generally retains favorable structural characteristics, including reactive functional groups and adequate thermal stability, making it suitable for bio-based adhesive production. These findings demonstrate that comprehensive characterization is essential for understanding how lignin properties influence the performance and sustainability of lignin-based adhesives.

### **Application**

The reviewed studies indicate that the type of organic acid used during lignin precipitation plays a crucial role in determining the adhesive performance of lignin-based formulations. Differences in acid structure influence lignin purity, functional group preservation, molecular interactions, and thermal stability, which subsequently affect bonding strength and curing behavior. Bibliometric analysis further revealed a growing research focus on the relationship between lignin modification and bio-based adhesive performance, highlighting the importance of optimizing lignin recovery processes for material applications.

Among the organic acids investigated, citric acid generally exhibited advantages in preserving hydroxyl and carboxyl functional groups due to its multifunctional carboxylic structure. These functional groups enhance intermolecular hydrogen bonding and increase the potential for crosslinking within adhesive systems, resulting in improved bonding strength and adhesive stability (Sangjan & Widjaja, 2022). In contrast, maleic acid contains unsaturated double bonds that can promote additional chemical interactions and increase lignin reactivity, making it particularly attractive for adhesive formulations requiring enhanced curing performance. Acetic acid, although structurally simpler, remains widely used because it effectively precipitates lignin while maintaining adequate structural integrity and processing efficiency (Zakaria et al., 2022).

The synthesis of previous studies suggests that lignins recovered using different organic acids produce distinct adhesive characteristics. Lignins with higher retention of phenolic and hydroxyl groups generally demonstrate stronger adhesion, better thermal resistance, and improved compatibility with crosslinking agents such as PVOH, hexamine, and glutaraldehyde. Consequently, plywood bonded with lignin-based adhesives can achieve satisfactory mechanical properties while maintaining low formaldehyde emissions (Kanti et al., 2022; Song et al., 2023). These findings demonstrate that adhesive performance is not determined solely by lignin content, but also by the specific organic acid employed during lignin precipitation, which directly influences the physicochemical properties of the recovered lignin and its suitability for sustainable adhesive applications.

## CONCLUSION

This study demonstrates that lignin derived from breadfruit skin biomass has considerable potential as a sustainable raw material for bio-based adhesive production. The bibliometric analysis of 114 publications revealed a growing research interest in lignin valorization, bio-based adhesives, biomass utilization, and sustainable material development. The keyword mapping further indicated that research has primarily focused on lignin extraction, characterization, and adhesive applications, while studies specifically investigating breadfruit skin lignin and the comparative effects of different organic acids remain relatively limited. These findings highlight important opportunities for future research in this emerging field.

The systematic review showed that the type of organic acid used during lignin precipitation significantly influences the physicochemical characteristics of the recovered lignin. Citric acid, maleic acid, and acetic acid were found to produce lignins with different structural properties, functional group compositions, thermal stability, and reactivity, which subsequently affect

adhesive performance. Characterization techniques such as FTIR, TGA, NMR, and SEM consistently demonstrated that breadfruit-skin-derived lignin contains reactive phenolic and hydroxyl groups that support crosslinking and bonding processes in adhesive formulations.

Overall, the findings suggest that breadfruit skin biomass represents a promising and underutilized renewable resource for environmentally friendly adhesive production. The integration of bibliometric and systematic review approaches provides a comprehensive understanding of current research trends and scientific evidence, confirming the potential of breadfruit skin lignin as a viable alternative to conventional petrochemical adhesives. Further studies are recommended to optimize organic acid selection, improve adhesive performance, and evaluate the scalability of lignin-based adhesive production for industrial applications.

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