

Effect of acid leaching on the ash fusion behavior and slagging/fouling indices of bagasse: focus on potassium and chlorine removal

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Managing ash-related issues, such as slagging, fouling, and corrosion, is critical for the energy utilization of agricultural residues. This study proposes a sustainable pretreatment using mild organic acids to enhance the combustion characteristics of bagasse, specifically focusing on the selective removal of potassium (K) and chlorine (Cl). Under optimal conditions (80 °C for 30 min), the process achieved maximum removal efficiencies of 96.8% for K (with citric acid) and 93.4% for Cl (with oxalic acid), while maintaining high solid recovery. X-ray diffraction analysis revealed that while the mineral framework remained stable, the treatment effectively targeted problematic low-melting-point species. This improvement significantly enhanced the ash fusion behavior, with the initial deformation temperature (IDT) increasing from 1130 °C to beyond the instrument's maximum measurable temperature of 1500 °C. Furthermore, comprehensive evaluations using predictive indices (base-to-acid ratio, bed agglomeration index, fouling index, and slag viscosity index) indicated a consistent transition from high-risk to low-risk regimes. Citric acid was identified as the most effective solvent due to its superior selectivity and energy preservation. These results demonstrate that organic acid leaching is a practical strategy for upgrading low-quality biomass into high-grade biofuels, ensuring the operational reliability of biomass-fired power systems.

Keywords: Sugarcane bagasse, Organic acid leaching, Ash fusion behavior, Slagging and fouling indices, Potassium and chlorine removal

Introduction

With growing awareness of fossil fuel depletion, climate change, and environmental issues, global interest in utilizing renewable fuels, such as waste-derived fuels and biomass, has been steadily increasing [1-4]. Among the various energy conversion pathways, the direct combustion of biomass remains the most widely adopted method for energy production [5-7]. Biomass offers several advantages as a fuel, including high volatile matter content and relatively low ash, nitrogen, and sulfur contents [8, 9]. However, as conventional woody pellets are increasingly replaced by agricultural byproduct-based biofuels, several operational issues have emerged during combustion, with slagging and fouling being the most critical.

Potassium, which is prevalent in agricultural waste, leads to the formation of low-melting-point compounds. These compounds easily adhere to particles and heating surfaces during combustion, resulting in severe slagging and fouling [10-12]. In particular, alkali metals such as potassium and sodium accumulate on the superheaters and reheaters of boilers, playing a key role in these phenomena [13-15]. Furthermore, slagging and fouling

are closely associated with boiler corrosion. During combustion, potassium and sodium react with chlorine to form potassium chloride (KCl) and sodium chloride (NaCl), respectively; these are released with the flue gas as molten salts at temperatures exceeding 900 °C. During the subsequent cooling process, the chlorides deposited on the boiler surfaces react with the oxide film, accelerating steel oxidation and ultimately leading to severe corrosion.

Various strategies for corrosion mitigation have been reported, including the use of corrosion-resistant materials, optimized boiler design, and modifications to fuel characteristics [16-20]. For instance, thermochemical conversion of biomass, such as pyrolysis and gasification, has been proposed as effective approaches to remove corrosive components from fuel [21]. While these methods can significantly reduce corrosion, they must be integrated early in the design process and inevitably increase initial investment costs due to the requirement for expensive materials. Alternatively, studies have investigated blending fuel additives with biomass to alter ash composition and reduce the concentration of problematic components [22]. While this approach offers advantages in transportation and storage without requiring additional pretreatment, it may lead to excessive ash formation, which reduces the calorific value. Furthermore, it is difficult to apply universally

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to all biomass types, and the long-term effects of ash accumulation require further verification. Therefore, a fundamental solution lies in the development of technologies that can simultaneously eliminate the root causes of boiler corrosion while maintaining the fuel's heating value. A promising approach involves using acid agents to selectively remove alkali metals, such as potassium (K) and sodium (Na), which are primarily responsible for slagging and fouling. The combustion of biomass treated with acid is considered a simpler and more practical alternative for existing boiler systems, as it effectively reduces the propensity for ash-related issues by leaching out these alkali species. Consequently, this approach is expected to enhance combustion stability and improve overall operational reliability. However, conventional inorganic acids, such as hydrochloric acid (HCl) and sulfuric acid (H₂SO₄), contain chlorine (Cl) and sulfur (S) components, respectively, that are detrimental to solid fuel quality and can lead to secondary corrosion. To address these limitations, the use of organic acids—environmentally friendly alternatives that do not introduce harmful elements—has been proposed as a sustainable strategy.

Accordingly, this study conducted acid treatment using organic acids to mitigate ash-related issues arising from the combustion of bagasse, a byproduct of sugarcane processing predominantly produced in Southeast Asian countries such as Vietnam and Indonesia [23]. In addition to mitigating the risks of secondary corrosion and environmental impact associated with inorganic acids, organic acids offer improved process safety and operability. In this work, the effects of acid treatment on ash characteristics, including chemical composition, mineral phases, and fusion behavior, were systematically investigated. These changes were further utilized to predict slagging and fouling tendencies during bagasse combustion. Ultimately, this study evaluates the potential of organic acid treatment as an effective and sustainable strategy for alleviating operational problems in boilers and combustion systems.

Experiment

Materials

Bagasse was sourced from the Institute for Advanced Engineering (IAE, Yongjin, Republic of Korea). The IAE imports sugarcane from Vietnam and produces bagasse via juice extraction. The bagasse was cut using a mixer and used directly without any further pretreatment. The reagents used in the experiments included acetic acid (99.5%, CAS No. 64-19-7, DAEJUNG, Korea), citric acid (99.5%, CAS No. 5949-29-1, DAEJUNG, Korea), oxalic acid (99.5%, CAS No. 6153-56-6, DAEJUNG, Korea), distilled water and ethanol. Acetic acid, citric acid, and oxalic acid were diluted with distilled water.

Experimental method

Bagasse was immersed in the acid solution to leach alkali components. Based on preliminary screening experiments, the acid concentration was fixed at 0.75 M, and the solid-to-liquid ratio was maintained at 20 g/L to ensure sufficient interaction between the biomass and the leaching solution while minimizing excessive solvent consumption. The leaching process was conducted under static soaking conditions without external stirring. This condition was intentionally selected to evaluate the feasibility of a simple and energy-efficient pretreatment process applicable to practical large-scale biomass treatment systems. The reaction temperatures and residence times were systematically varied to determine the optimal leaching conditions for potassium and chlorine removal and solid recovery.

After the reaction, the solid and liquid phases were separated by vacuum filtration using a Whatman filter No. 41 (Cativa, Marlborough, USA) and an aspirator (WJ-15, Taewon Shibata Co., Ltd., Japan). The solid product was thoroughly washed with distilled water to remove residual impurities and then dried in an oven.

Analytical method

The physicochemical properties of raw and acid treated bagasse were characterized using various analytical techniques. Inductively coupled plasma optical emission spectrometer (ICP-OES; iCAP 7600 ICP-OES Duo, Thermo fisher scientific, USA) and X-ray fluorescence (XRF; NEX-CG, Rigaku Co., Japan) were employed to determine the chemical composition. X-ray diffraction (XRD; MiniFlex, Rigaku Co., Japan) was used to investigate the crystalline structure.

To predict the potential for fouling and slagging during combustion, several slagging and fouling indices were calculated based on the chemical composition of the generated ash. The calculation formula for the base-to-acid ratio (B/A), bed agglomeration index (BAI), fouling index (FI), and slag viscosity index (SVI) are provided in Table 1.

Among the ash melting characteristics, the initial deformation temperature (IDT), softening temperature (ST), hemispherical temperature (HT), and flow temperatures (FT) were measured according to ISO 21404-2020 (ISO 2020). To analyze the ash melting behaviors, bagasse samples were compression molded into pellets with a height and diameter of 0.5 cm using a biomass ash mold (Chansha Kaiyuan Instruments, Changsha, China) to ensure uniformity. The manufactured pellets were loaded into an ash melting point measuring device (5E-AF4115, Changsha Kaiyuan Instruments) and heated in an oxidizing atmosphere at a constant heating rate of 5 °C min⁻¹.

All experiments were conducted in triplicate to ensure reproducibility and statistical reliability. The reported values represent the average of three independent experiments.

Table 1. Summary of predictive indices for ash-related operational risks, including their respective equations and threshold criteria

Index	Equation	Criteria	Ref.
Base-to-acid ratio (B/A)	$B/A = \frac{Fe_2O_3 + CaO + MgO + K_2O + Na_2O}{SiO_2 + Al_2O_3 + TiO_2} \quad (1)$	< 0.5 : Low 0.5 – 1 : Medium 1 – 1.75 : High > 1.75 : Severe	[24]
Bed agglomeration index (BAI)	$BAI = \frac{Fe_2O_3}{Na_2O + K_2O} \quad (2)$	< 0.15 : High	[25, 26]
Fouling Index (FI)	$FI = \left(\frac{B}{A}\right)(Na_2O + K_2O) \quad (3)$	< 0.6 : Low 0.6 – 40 : Medium > 40 : High	[27]
Slagging viscosity Index (SVI)	$SVI = \frac{SiO_2}{SiO_2 + CaO + MgO + Fe_2O_3} \times 100 \quad (4)$	> 72 : Low 63 – 72 : Medium < 63 : High	[25]

Note: References for each index and its criteria are provided in the rightmost column

Results and Discussion

Raw bagasse contains various inorganic constituents, as detailed in Table S1. In particular, it contains a substantial amount of potassium (32.82 mg kg⁻¹), which must be removed to prevent boiler corrosion. Sodium, another major element contributing to boiler corrosion, was present in negligible amounts in the samples used in this study. Chloride is present in a low concentration of 0.19 mg kg⁻¹. Despite this low content, chlorine poses a substantial risk of high-temperature corrosion, which underscores the importance of its significant reduction during the pretreatment process. After acid treatments, the potassium and chlorine contents decreased substantially, regardless of the acid type (Table S1-S3). The removal of these elements tended to increase with higher reaction temperatures and longer treatment times. Under all experimental conditions, the potassium removal efficiency exceeded 82%, while the chlorine removal efficiency was over 79%. The highest leaching performance was observed at 80 °C for 30 min, where citric acid achieved a 96.8% removal of potassium and oxalic acid reached a 93.4% removal of chlorine. Increasing the leaching temperature to 100 °C did not yield a significant improvement in efficiency, suggesting that 80 °C is sufficient to provide the optimal conditions for effective leaching. The remaining potassium and chlorine may be strongly embedded within the organic matrix, posing physical and chemical limitations for further extraction.

The solid recovery efficiencies after acid treatment are shown in Table S4. Across all experimental conditions, citric acid exhibited the highest solid recovery efficiency, followed by oxalic acid, while acetic acid treatment resulted in the lowest efficiency. Interestingly, while both citric and oxalic acids showed similar high efficiencies in removing potassium and chlorine—outperforming acetic acid—the solid recovery for acetic acid was

notably lower than that of the other two solvents. This suggests that acetic acid promotes non-selective removal by leaching out a broader range of components beyond potassium and chlorine. In contrast, citric acid treatment demonstrates the highest selectivity among the three organic acids investigated.

Based on the data provided in Table S1-S3, the slagging and fouling indices were calculated using the equations defined in Table 1 (Fig. 1). For raw bagasse, all predictive indices were notably high, indicating a severe propensity for slagging and fouling. The slagging tendency of ash typically correlates with the base-to-acid ratio (B/A); a B/A value below 0.5 indicates a low slagging tendency. Raw bagasse exhibited a B/A value of 2.19, categorized as a very high slagging tendency (Fig. 1a). However, after acid treatment, the B/A ratio generally decreased to below 1.0, confirming that the treatment effectively mitigated the potential for slagging. The bed agglomeration index (BAI) is used to assess operational issues associated with low ash fusion temperatures; previous studies have indicated that bed agglomeration occurs when the BAI is below 0.15 [24]. The BAI of raw bagasse ash was 0.06, which increased to 0.22-0.99 following acid treatment (Fig. 1b). These BAI values suggest that acid treatment of biomass will significantly reduce bed agglomeration during combustion. Similarly, the fouling index (FI), which indicates the propensity for ash deposition, was remarkably high at 71.93 for the raw material. Upon acid treatment, the FI significantly decreased to below 5 (Fig. 1c). Based on these results, it can be inferred that acid treatment effectively reduces the fouling tendency of bagasse. While the base-to-acid (B/A) ratio, bed agglomeration index (BAI), and fouling index (FI) exhibited marked improvements following acid treatment, the slagging viscosity index (SVI) did not show a similarly significant enhancement. Substantial improvements in the SVI were only observed under a limited number of solvent types and reaction conditions

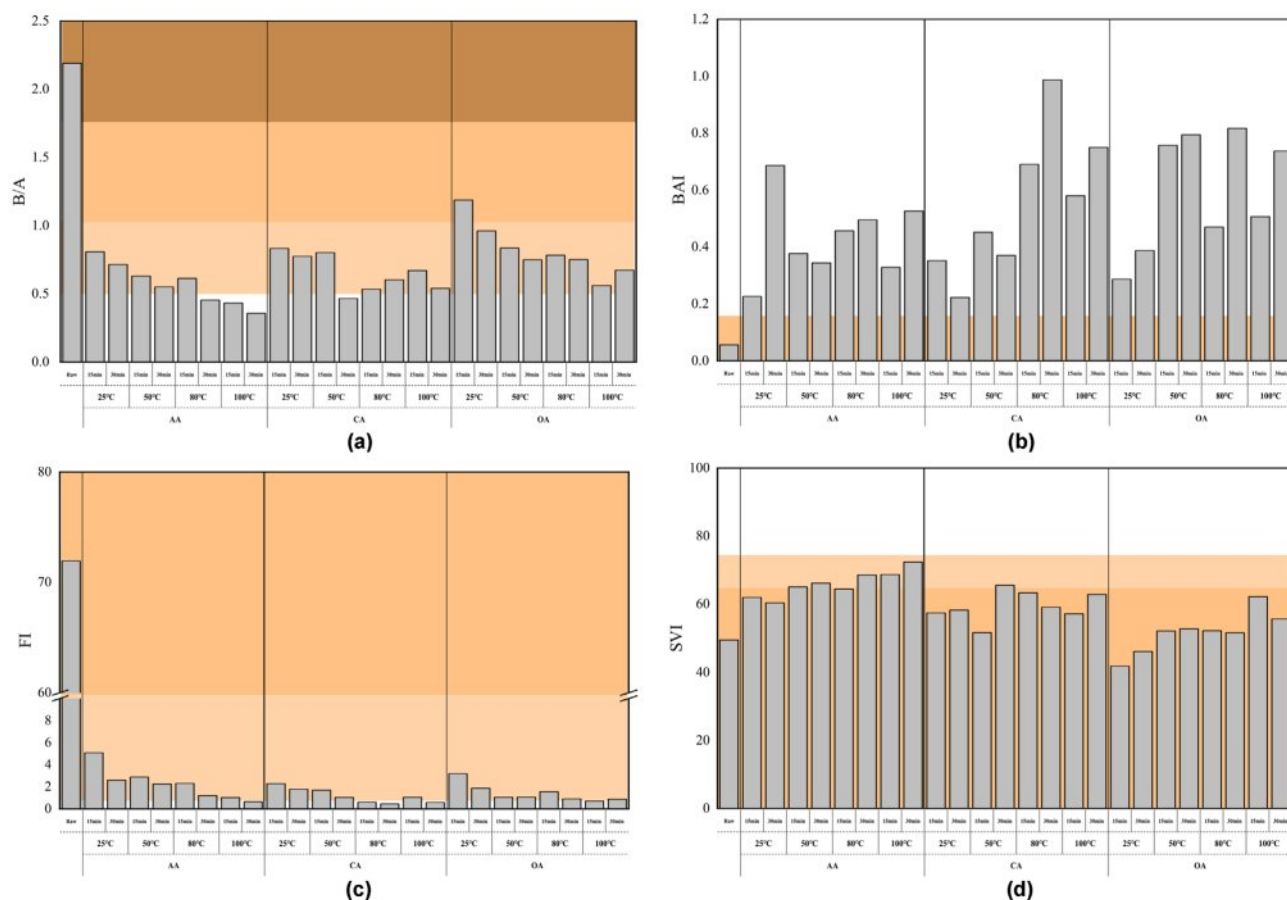


Fig. 1. Predictive indices for slagging and fouling of bagasse samples: (a) base-to-acid ratio (B/A), (b) bed agglomeration index (BAI), (c) fouling index (FI), and (d) slagging viscosity index (SVI). The x-axis denotes the acid leaching conditions (reaction time and temperature) for each organic acid (AA: acetic acid; CA: citric acid; OA: oxalic acid). The shaded backgrounds indicate the risk levels defined in Table 1, where increasing color intensity corresponds to a higher risk—ranging from "low" (white) to "severe" (most intense shade).

(Fig. 1d). Based on these SVI values, although the propensity for slagging was slightly mitigated compared to the raw bagasse, the acid-treated samples still maintained a high potential for slagging. This suggests that while acid treatment is highly effective in reducing potassium and chlorine contents and improving most predictive indices, its impact on the specific viscous behavior of the ash remains constrained, necessitating careful consideration of operating temperatures in actual boiler systems. This discrepancy arises because the SVI primarily reflects the viscosity behavior of ash under highly molten conditions, whereas the B/A ratio, BAI, and FI are more closely associated with the formation and deposition tendency of low-melting-point alkali species during the initial stages of ash melting. Therefore, although the SVI values still indicate a relatively high slagging tendency under fully molten conditions, the substantial increase in ash fusion temperatures and the suppression of low-melting-point phases suggest that the practical onset of slagging and fouling was significantly delayed after acid treatment. Despite this general trend, the treatment with citric acid at 80 °C for 30 minutes was identified as the most

favorable condition, as it provided the most balanced improvement across all evaluated indices, including a relatively higher SVI compared to other treatments.

A previous study has shown that potassium sulfate and potassium chloride are the main causes of slagging, fouling, and high-temperature corrosion by forming highly adhesive deposits during combustion [28]. Ideally, the problematic compounds within the ash would either be entirely eliminated or transformed into more stable potassium phases when the acid-treated bagasse is combusted. In our previous study, an aluminosilicate-based fuel additive was utilized to induce a phase transition of potassium-based ash compounds from low-melting-point phases to those with higher melting points [22]. In the present study, X-ray diffraction (XRD) was employed to investigate the mineralogical transformations in the ash derived from solid fuels where potassium and chlorine components were removed through acid treatment, rather than through chemical additives. The mineral phases of the raw bagasse primarily consisted of dipotassium tetrathiosulfate and dipotassium thiosulfate, which are characterized by a low melting point of 680 °C

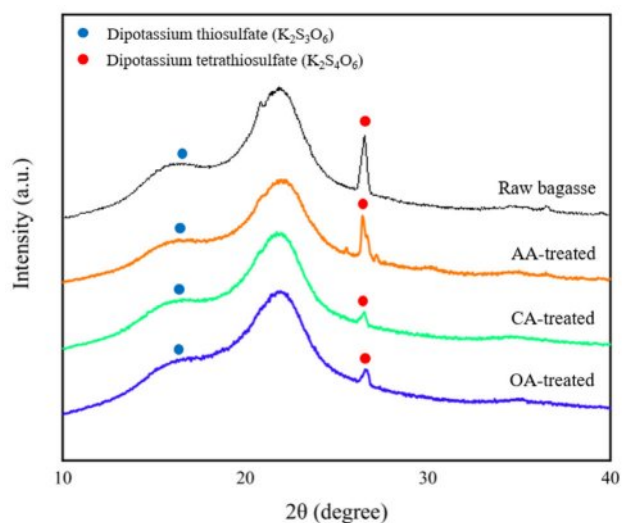


Fig. 2. XRD patterns of raw and acid-treated bagasse ash showing the reduction of potassium-based mineral phases. AA-treated: acetic acid-treated; CA-treated: citric acid-treated; OA-treated: oxalic acid-treated.

(Fig. 2). These low-melting-point potassium compounds have a strong propensity to form molten, tenacious ash particles that facilitate agglomeration and condensation on heat transfer surfaces, ultimately compromising boiler efficiency. It has been reported that high-temperature corrosion is most severe within the temperature range of 868–978 K (approximately 595–705 °C), where alkali sulfates exist in a liquid state. This underlines that the melting behavior of ash is a critical factor governing corrosion kinetics [29]. In this study, as potassium and chlorine were removed solely through acid treatment without the external addition of inorganic additives, no significant shifts in the XRD spectra or major phase transitions were observed (Fig. 2). However, in the samples treated with citric and oxalic acids, a substantial reduction in the dipotassium tetrathiosulfate peaks was identified, alongside a decrease in the intensity of dipotassium thiosulfate peaks. This confirms that the treatment effectively suppresses the formation of low-melting-point compounds, thereby reducing the potential for ash-induced slagging and fouling. This indicates that while the overall mineral framework remains relatively stable, the selective leaching of potassium and chlorine by organic acids specifically targets the most problematic, low-melting-point species. To verify whether these mineralogical transitions—specifically the reduction of low-melting-point phases—correlate with the actual thermal behavior of the ash, the four characteristic ash melting temperatures (SST, IDT, HT, and FT) were measured (Table 2). As anticipated from the XRD analysis, the raw bagasse without acid treatment exhibited IDT, HT, and FT at relatively low temperatures, thereby increasing the risk of slagging and bed agglomeration. Specifically, the raw bagasse

Table 2. Ash fusion temperatures of raw and organic acid-treated bagasse samples.

	Raw bagasse	AA-Treated	CA-Treated	OA-Treated
SST (°C)	1040	1410	1310	1350
IDT (°C)	1130	> 1500 [†]	> 1500	> 1500
HT (°C)	1290	> 1500	> 1500	> 1500
FT (°C)	1340	> 1500	> 1500	> 1500

Note: AA-Treated: acetic acid-treated; CA-Treated: citric acid-treated; OA-Treated: oxalic acid-treated; SST: Shrinkage temperature; IDT: Initial deformation temperature; HT: Hemisphere temperature; FT: Flow temperature.

[†]Temperatures recorded as 1500 °C reached the maximum limit of the measurement apparatus.

began to deform at 1130 °C and reached a full flow state at 1340 °C, conditions highly conducive to ash liquefaction during combustion. In contrast, following the acid treatment, the IDT of the samples significantly increased to over 1500 °C, representing a substantial improvement of approximately 32.7% compared to the raw bagasse. Furthermore, the acid treatment effectively shifted the FT to above 1500 °C. These findings are highly consistent with the observed decrease in low-melting-point potassium-based compounds in the XRD analysis, confirming that acid treatment substantially improves ash fusion characteristics and significantly reduces the propensity for slagging and agglomeration.

The acid treatment strategy developed in this work achieved impressive removal efficiencies of over 82% for potassium and 79% for chlorine, presenting a straightforward and potentially cost-effective approach to addressing the fundamental issue of boiler corrosion. By suppressing the formation of low-melting-point potassium species, the treatment successfully elevated the IDT, HT, and FT, thereby substantially mitigating the risks of slagging and bed agglomeration. However, despite these promising experimental results, several practical aspects warrant further consideration. A primary concern is the significant volume of wastewater generated during the acid leaching process, which poses a potential environmental burden. To ensure the viability of this process, integrated strategies for wastewater treatment and solvent recycling must be established. Furthermore, while the current treatment demonstrated high reproducibility and effectiveness at the laboratory scale, its scalability to continuous, large-scale industrial production remains to be validated. Consequently, subsequent efforts should focus on optimizing the process for mass production, verifying its long-term functional performance in practical boiler environments, and conducting a rigorous economic feasibility analysis to support its transition from the laboratory to industrial application.

Conclusion

This study evaluated mild organic acid leaching as a sustainable pretreatment to improve the combustion stability of sugarcane bagasse. The key findings are as follows:

High Removal Efficiency and Selectivity: Under optimal conditions (80 °C, 30 min), the process achieved maximum removal efficiencies of 96.8% for K (citric acid) and 93.4% for Cl (oxalic acid). The treatment demonstrated high selectivity, effectively removing inorganic precursors while maintaining excellent solid recovery.

Mineralogical Stability: XRD analysis confirmed that the treatment successfully suppressed low-melting-point compounds, such as dipotassium tetrathiosulfate. Unlike additive-based methods, this approach focuses on the fundamental elimination of problematic species without altering the primary mineral framework.

Enhanced Thermal Performance: The removal of K and Cl led to a dramatic increase in ash fusion temperatures, with both the IDT and FT exceeding the instrument's maximum measurable temperature of 1500 °C.

Improved Operational Reliability: Predictive indices (B/A, BAI, and FI) consistently shifted from "high-risk" to "low-risk" regimes. Citric acid was identified as the most effective solvent for maximizing ash stability and energy preservation.

In conclusion, organic acid leaching is a practical and robust strategy for upgrading low-quality biomass into high-grade biofuels. By addressing the major contributors to slagging and fouling, this method can significantly improve the operational reliability of biomass-fired power systems and supports the transition toward carbon-neutral energy.

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