Research Progress in the Application of Advanced Oxidation Pretreatment for Biomass Energy Production

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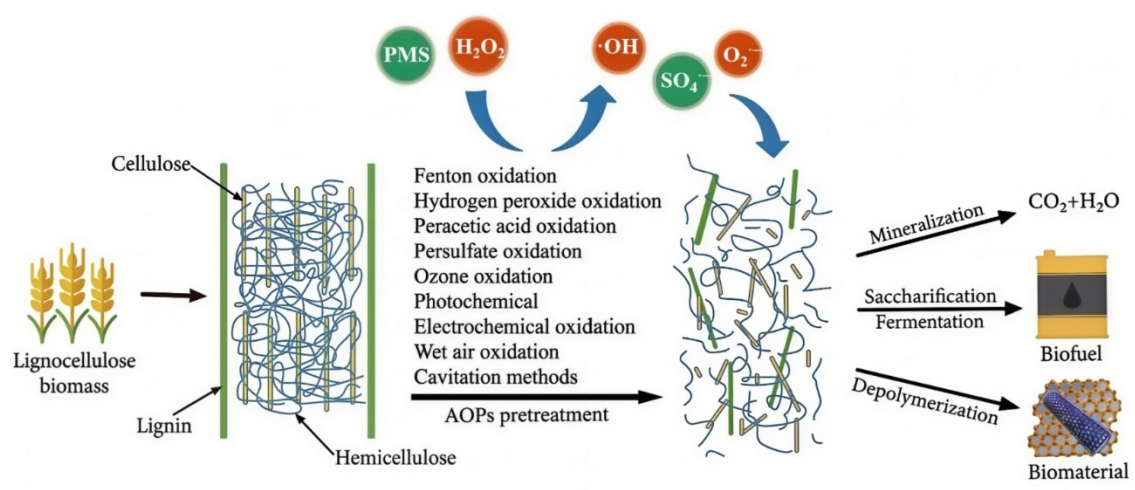
DOI: 10.15376/biores.21.3.Zhang2

GRAPHICAL ABSTRACT



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Lignocellulosic biomass is an abundant renewable energy resource whose total energy content far exceeds current global demand. Its polysaccharides, cellulose, and hemicelluloses are primary targets for valorization; however, the recalcitrant structure of plant cell walls necessitates effective pretreatment. Among physical, physicochemical, chemical, and biological methods, advanced oxidation processes (AOPs) have emerged as an efficient and environmentally compatible chemical strategy. This review systematically evaluates nine major AOPs for lignocellulosic biomass pretreatment: Fenton and Fenton-like processes, alkaline H_2O_2, peracetic acid, persulfate, ozone, photocatalysis, electrochemical oxidation, wet air oxidation, and cavitation-assisted methods. For each, reaction mechanisms, advantages and limitations, recent advances, economic considerations, and scale-up challenges are discussed. Persulfate-based systems, wet air oxidation, and hybrid strategies (*e.g.*, photo-Fenton, alkaline H_2O_2–cavitation, electrochemical–ozone–H_2O_2) are identified as particularly promising. Future research should prioritize novel catalyst development, reactor optimization, multi-mechanism integration, and rigorous techno-economic and life-cycle assessments. Coupling AOPs with renewable energy sources will be critical to improving energy efficiency and enabling cost-effective large-scale application.

DOI: 10.15376/biores.21.3.Zhang2

Keywords: Lignocellulose; Biomass energy; Pretreatment; Advanced oxidation processes (AOPs)

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INTRODUCTION

Lignocellulosic biomass, as the most abundant renewable carbon source, is not only vast in reserves but also low in cost, making it an ideal raw material for large-scale biofuel and bio-based chemical production (Ashokkumar *et al.* 2022; IEA 2024). However, the complex structure and cross-linking properties of lignin hinder the efficient degradation of lignocellulosic biomass, representing a significant barrier in the biomass conversion process (Ashokkumar *et al.* 2022). In lignocellulosic bioconversion, pretreatment modifies the structural and chemical features of biomass to facilitate subsequent enzymatic hydrolysis and fermentation. After pretreatment, enzymatic saccharification releases fermentable sugars, which are then converted into biofuels or other production through microbial fermentation and product recovery (Bansod *et al.* 2024). Because the efficiency

of these downstream processes strongly depends on substrate accessibility and structural disruption, pretreatment plays a decisive role in determining overall conversion performance. Effective pretreatment strategies aim to reduce lignin-associated recalcitrance and enhance cellulose accessibility to improve bioconversion efficiency (Alvira *et al.* 2010; Rezania *et al.* 2020). Advanced oxidation processes (AOPs), characterized by the *in situ* generation of highly reactive radical species such as ·OH and $SO_4^{\cdot-}$, have recently emerged as promising alternatives for lignocellulosic pretreatment (Prado *et al.* 2022). Rather than relying solely on bulk chemical dissolution or harsh physical disruption, AOPs introduce controlled oxidative reactions capable of modifying lignin-rich domains and altering interpolymer linkages within the biomass matrix. In this context, it can be hypothesized that an appropriately regulated advanced oxidation process will be able to partially disrupt lignin barriers and weaken structural constraints without extensive degradation of fermentable polysaccharides, thereby rendering biomass more accessible to cellulases, fermentative microorganisms, and other bioconversion factors. Among these, persulfate-based technologies, particularly peroxymonosulfate (PMS), Fenton-like processes, and wet air oxidation are gaining attention because of their superior oxidative performance. Such approaches are being increasingly used to improve the saccharification rate of lignocellulose. Therefore, this article provides a comprehensive review of recent advances in advanced oxidation-based pretreatment strategies for lignocellulosic biomass, with particular emphasis on their mechanisms, performance, and challenges.

LIGNOCELLULOSIC PRETREATMENT

Lignocellulosic biomass primarily consists of three types of biopolymers: cellulose, hemicellulose, and lignin (Jiang *et al.* 2017). Cellulose forms highly ordered crystalline microfibrils stabilized by extensive intra- and intermolecular hydrogen bonding. Hemicellulose acts as a branched, amorphous polysaccharide that associates with cellulose surfaces, while lignin forms a three-dimensional aromatic polymer that embeds the carbohydrate fraction (Ashokkumar *et al.* 2022). The recalcitrance of lignocellulose arises from several structural factors: (i) cellulose crystallinity, which limits enzymatic accessibility; (ii) the physical shielding effect of lignin; (iii) lignin-carbohydrate complexes (LCCs) that chemically link polysaccharides and lignin; and (iv) restricted pore structure and low surface area. Among these, lignin plays a central role by limiting enzyme adsorption and, in some cases, promoting non-productive enzyme binding. Therefore, effective pretreatment strategies often aim to disrupt lignin structure, increase porosity, and enhance enzyme-substrate interactions while preserving fermentable carbohydrates (Hassan *et al.* 2018; Liu *et al.* 2018).

Pretreatment methods are generally classified into physical, biological, chemical, and physicochemical combined treatments (Putro *et al.* 2016; Sun *et al.* 2016). Physical methods enhance enzymatic hydrolysis by disrupting the cell wall structure and increasing the surface area. While simple to operate, they have high energy consumption and are noted for limited lignin removal, so that they are typically used as supplementary methods. Biological methods, which use enzymes secreted by microorganisms to degrade lignin, are energy-efficient and operate under mild conditions, but they require long processing times and high microbial activity (Rouches *et al.* 2016). Chemical methods break the chemical bonds in lignocellulose using acids, bases, or solvents, lowering crystallinity and

accelerating degradation. These methods offer good degradation efficiency, but they often require strong acids or bases, leading to high equipment requirements and costs (Taherzadeh and Karimi 2008). Additionally, chemical pretreatment includes the use of ionic liquids, low eutectic solvents, and, more recently, advanced oxidation methods (Behera *et al.* 2014). Advanced oxidation processes (AOPs), by generating highly oxidative free radicals, significantly improve the degradability of lignocellulose and offer advantages such as high efficiency, environmental friendliness, and mild operating conditions (Joshi and Manjare 2024).

AOP PRETREATMENT OF BIOMASS AND ITS APPLICATIONS

The Principle of AOP Pretreatment of Lignocellulose

Advanced oxidation processes (AOPs) are chemical pretreatment methods that generate strong oxidizing free radicals, such as ·OH, $SO_4^{\cdot-}$, $O_2^{\cdot-}$. These can effectively break down the lignocellulosic structure. The mechanism of AOP pretreatment of lignocellulosic substrates for energy production is illustrated in Fig. 1. The AOPs have been widely applied in wastewater treatment and soil remediation (Zhou *et al.* 2019; Liu *et al.* 2021a). Recent studies highlight their potential in enhancing cellulose accessibility and promoting the production of biogas, bioethanol, biodiesel, and biohydrogen. The primary mechanisms involve the disruption of lignin barriers, improving cellulose accessibility, and enhancing hydrolysis, and fermentation performance (Dong *et al.* 2018; Zhang *et al.* 2019a; Göncü *et al.* 2021).

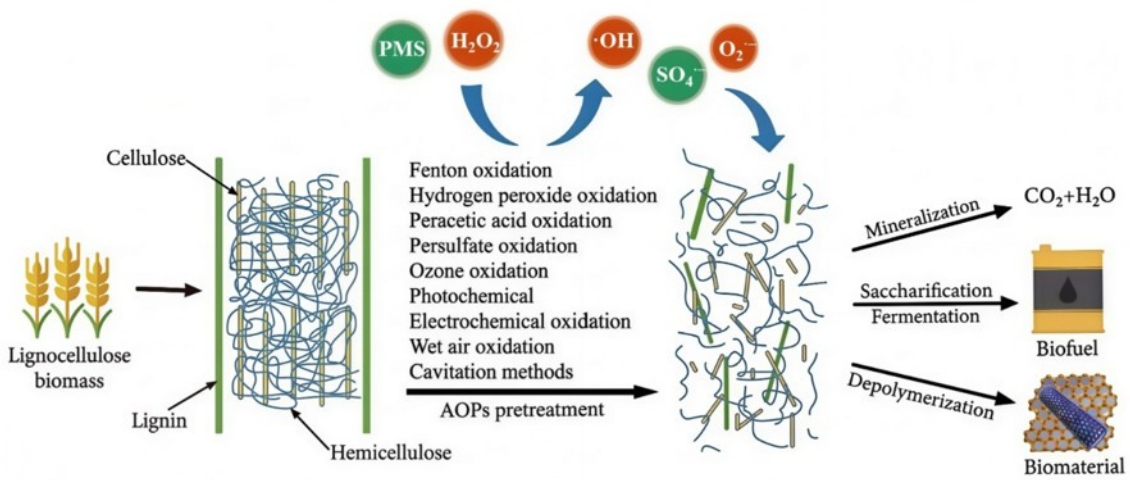

Fig. 1. Schematics of AOPs pretreatment for lignocellulosic biomass

Various AOP Pretreatment Methods

Fenton and Fenton-like pretreatment

Fenton technology is a commonly used method within AOPs. It is widely applied and studied due to its high efficiency and simplicity in operation, particularly in reducing the chemical oxygen demand (COD) content of domestic sewage (Liu *et al.* 2021b). In recent years, Fenton technology has also been used for the pretreatment of lignocellulosic biomass (Zhang and Zhu 2016; Wu *et al.* 2018). The essence of the Fenton technique is the reaction of H_2O_2 with Fe^{2+} under acidic conditions to generate the ·OH radical (Wu *et al.* 2020). The ·OH radical can abstract protons from the substrate, forming organic free

radicals, which can then react with other substrates. The reaction mechanism is shown in Eqs. 1-3 (Wang and Tang 2021):

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^- + \cdot OH \quad (1)$$

$$RH + \cdot OH \rightarrow R\cdot + H_2O \quad (2)$$

$$R\cdot + Fe^{3+} \rightarrow Fe^{2+} + \text{Product} \quad (3)$$

Studies have shown that Fenton pretreatment effectively disrupts the lignin structure and increases cellulose accessibility, thereby enhancing enzymatic hydrolysis and fermentation efficiency. Research has demonstrated that Fenton pretreatment is effective for various biomass substrates such as crop straw, sugarcane bagasse, and corn stover, with sugar yields increasing by 1.5 to 3 times, and improved conversion efficiencies for ethanol and biogas (Zhang and Zhu 2016; Koo *et al.* 2017; Wu *et al.* 2018; Huang *et al.* 2019). However, this method has some limitations. Traditional Fenton reactions generally require acidic conditions (pH ≈ 3) (Clarizia *et al.* 2017), which leads to high consumption of acid during pretreatment, and often requires the pH to be adjusted to neutral after pretreatment, resulting in higher reaction costs. Additionally, large amounts of iron-containing wastewater are generated, causing secondary pollution or additional cost enhancement for treatment (Fernandez *et al.* 1999; Andreozzi *et al.* 2002).

To address the limitations of traditional Fenton treatments, research has gradually shifted toward Fenton-like systems. The core mechanism of Fenton-like processes lies in the metal redox cycling on the catalyst surface (*e.g.*, Fe^{3+}/Fe^{2+} or other transition metal pairs), which can continuously activate oxidants to generate reactive species such as ·OH radicals without relying on excess soluble Fe^{2+}. This alleviates the problems of iron ion leaching and iron sludge accumulation. This mechanism allows the reaction to be no longer limited to the instantaneous release of free radicals. As a consequence, it usually exhibits more sustained oxidative activity and broadening the applicable pH range, while improving the catalyst's stability and recyclability (Lin and Gurol 1998). Catalysts such as $BiFeO_3$, $FeVO_4$, carbon-based composite catalysts, iron oxides, and Fe-Mn composite oxides have been shown to be effective for lignocellulosic biomass pretreatment (Koo *et al.* 2017; Zhang *et al.* 2019b; Liang *et al.* 2024). These Fenton-like catalysts generally exhibit better lignin removal and saccharification efficiency than traditional Fenton in lignocellulosic biomass pretreatment. For example, Fe_3O_4 has bridged the gap from homogeneous to heterogeneous catalysis (Fernandez *et al.* 1999; Koo *et al.* 2017); $BiFeO_3$ generates higher concentrations and longer-lived ·OH in rice straw and sugarcane bagasse treatments, improving saccharification rates more than twice compared to traditional Fenton (Zhang *et al.* 2019b). The $FeVO_4$ catalyst, with Fe^{3+} and V^{5+} metal ions both catalyzing H_2O_2, exhibited superior sugar conversion capability compared to α-Fe_2O_3, Fe_3O_4, α-FeOOH, and FeS_2 (Liang *et al.* 2024).

Furthermore, the combination of Fenton technology with other pretreatment methods has become a recent research focus, such as the synergy of Fenton with photochemical processes (Mahy *et al.* 2023), cavitation (Lee and Han 2021; Li *et al.* 2024; Askarniya *et al.* 2025), and deep eutectic solvents (Gong *et al.* 2024). Photo-Fenton accelerates the generation of free radicals under weak acidic conditions by promoting the photoreduction of Fe^{3+} and the photodegradation of H_2O_2 (Mahy *et al.* 2023); sonochemical Fenton utilizes the high temperatures, pressures, and intense shear forces generated by ultrasonic cavitation to promote the cleavage of H_2O_2 and water molecules, producing ·OH

and enhancing the structural disruption of lignocellulose (Li *et al.* 2024; Askarniya *et al.* 2025). These studies show that the coupling of Fenton with other pretreatment methods offers synergistic benefits, significantly outperforming traditional Fenton in terms of lignin removal and saccharification efficiency.

In summary, Fenton and Fenton-like technologies have broad application prospects in lignocellulose pretreatment, especially Fenton-like methods, which overcome the reliance on acidic conditions and iron sludge issues in traditional Fenton processes, thereby enabling catalyst recyclability. However, some Fenton-like catalysts still face issues such as complex synthesis processes, high preparation costs, and low recycling efficiency. Additionally, combinations of Fenton with light, deep eutectic solvents, and cavitation methods show synergistic advantages in efficiency, economics, and environmental friendliness, providing new insights for enhancing pretreatment efficiency.

Alkaline hydrogen peroxide pretreatment

Hydrogen peroxide (H_2O_2) is an environmentally friendly chemical oxidant with strong oxidative capabilities. Alkaline hydrogen peroxide (AHP) technology is a selective oxidation method for lignin removal, typically performed under mild conditions (at ambient pressure and temperatures ranging from 25 to 100 °C), offering low energy consumption and good environmental adaptability (Mun and Mun 2024). The AHP can effectively generate ·OH and $O_2^{\cdot-}$ under alkaline conditions (at a pH of approximately 11.5) (Gould 1984), with ·OH capable of cleaving ether bonds (such as β-O-4) and side chains in lignin. This initiates a series of reactions, such as ring opening of aromatic structures and the introduction of carboxyl groups, thus facilitating lignin removal and cellulose exposure (Li *et al.* 2012; Maziero *et al.* 2012). Concurrently, the alkaline conditions promote the saponification of ester bonds between lignin and hemicellulose, partially dissolving the hemicellulose and thereby enhancing cellulose accessibility for subsequent enzymatic hydrolysis (Whistler 1993). After AHP treatment, the biomass structure typically exhibits fiber disintegration, increased porosity, expanded specific surface area, and improved crystallinity, significantly enhancing enzymatic hydrolysis efficiency (Cui and Bai 2025). For instance, a critical saturation point (CSP) for lignin removal in bamboo was identified, with a significant increase in removal as H_2O_2 concentration rose from 0% to 2%, but no further improvement beyond 2% (Huang *et al.* 2022). Furthermore, the AHP can be coupled with other methods for enhanced results (Devadasu *et al.* 2020; Askarniya *et al.* 2025; Li *et al.* 2025).

Despite its excellent performance at the experimental level, the industrial application of AHP faces several critical challenges regarding safety and economics. First, the safety issues associated with AHP mixtures must be strictly addressed. High concentrations of hydrogen peroxide in strongly alkaline solutions can undergo rapid, exothermic decomposition, leading to rapid gas evolution and potential explosion hazards (Hart and Rudie 2007). Second, practical applications face economic limitations, such as the high cost of H_2O_2, significant alkaline solution consumption, and longer reaction times. Therefore, rigorous control of H_2O_2 concentrations, precise temperature regulation, and appropriate pressure-relief reactor designs are critical prerequisites for safe operations. Process optimization is urgently needed to reduce the H_2O_2 dosage or combine AHP with other physical/chemical methods to improve pretreatment efficiency, ensure safety, and control costs, thereby facilitating its industrial scale-up in lignocellulosic biorefining.

Peracetic acid pretreatment

Peracetic acid (PAA) is an organic peracid with strong oxidizing properties, bactericidal activity, and the ability to selectively remove lignin. Its chemical structure consists of a peroxide group (-OOH) bonded to an acetyl group (CH_3CO-), with a high oxidation potential of 1.748 V (Shi *et al.* 2022). The molecular structure of PAA contains an active peroxide bond (-O-O-), which has a lower bond energy compared to hydrogen peroxide, allowing for efficient lignin degradation under mild conditions. The PAA is typically synthesized by the reaction of hydrogen peroxide and acetic acid under sulfuric acid catalysis (Zhao *et al.* 2007; Kundu *et al.* 2021). Alternatively, it can be generated *in situ* by perhydrolase enzymes, reducing costs and transportation risks (Hu *et al.* 2022). Its action mechanism involves electrophilic attack on electron-rich sites in the lignin molecule by hydrogen ion (H^+) generated under acidic conditions, initiating a series of oxidative degradation reactions. This can oxidize the hydroxyl groups in lignin side chains to carbonyl groups, cleave the β-O-4 bond in lignin, reduce the molecular weight, and introduce hydrophilic groups (Yin *et al.* 2011; Shi *et al.* 2022). As a result, the depolymerized lignin fragments dissolve in water, effectively removing lignin from lignocellulosic biomass (Teixeira *et al.* 2000).

PAA pretreatment is typically performed under mild conditions below 80 °C to avoid excessive cellulose degradation and PAA decomposition (Yin *et al.* 2011; Hu *et al.* 2022). Numerous studies have demonstrated that PAA pretreatment exhibits good lignin removal efficiency for a variety of biomass types (Darus *et al.* 2022). PAA can also be coupled with other methods to optimize pretreatment effectiveness (Meng *et al.* 2022). Compared to Fenton pretreatment, the PAA pretreatment has been shown to be effective for plant biomass with higher lignin content and crystallinity index (CrI) (Xiao *et al.* 2017).

However, this method has some limitations. On the one hand, the selective lignin removal capability of PAA is lost under more severe conditions (high PAA concentration, high temperature, and metal ions involved), leading to degradation of cellulose and hemicellulose. On the other hand, the chemical synthesis of PAA presents high costs and explosion risks, limiting its large-scale application (Hu *et al.* 2022). Additionally, cost issues continue to hinder the industrialization of this technology. Therefore, current research focuses on improving the efficiency of PAA pretreatment and exploring its coupling with other processes such as alkali treatment, acid treatment, or steam explosion to enhance pretreatment effectiveness and reduce costs.

Persulfate pretreatment

Advanced oxidation technologies can be divided into sulfate radical-based advanced oxidation processes (SR-AOPs) and hydroxyl radical-based advanced oxidation processes (HR-AOPs), based on the type of free radical generated. Studies have shown that both ·OH and $SO_4^{\cdot-}$ possess strong oxidizing abilities (Qiu *et al.* 2020). Compared to ·OH, $SO_4^{\cdot-}$ have a longer half-life (30-40 μs), broader pH applicability, and higher stability (Wang *et al.* 2020; Liu *et al.* 2023), which has led to increasing research interest in recent years.

SR-AOP involves the activation of peroxymonosulfate (PMS) and peroxydisulfate (PDS), breaking the O-O bond to generate strong oxidizing free radicals, such as ·OH, $SO_4^{\cdot-}$, and $O_2^{\cdot-}$. For example, in the case of PDS, the activation mechanism involves two typical reactions: one is the O-O bond cleavage of PDS under high temperature, light, or ultrasound, producing two $SO_4^{\cdot-}$, as shown in Eq. 4; the other is the electron transfer from transition metals, carbon-based materials, or other electron donors to generate a single

$SO_4^{\cdot-}$, as shown in Eq. 5. The $SO_4^{\cdot-}$ can further react to produce ·OH (Eq. 6):

$$S_2O_8^{2-} + energy \rightarrow 2SO_4^{-\cdot} \quad (4)$$

$$S_2O_8^{2-} + M^{n+} \rightarrow SO_4^{-\cdot} + SO_4^{2-} + M^{(n+1)+} \quad (5)$$

$$SO_4^{-\cdot} + OH^- \rightarrow SO_4^{2-} + OH\cdot \quad (6)$$

The reactive ·OH and $SO_4^{\cdot-}$ radicals can attack lignin, breaking it down, as well as targeting internal bonds in hemicellulose and glycosidic bonds in cellulose and hemicellulose, thereby improving the pretreatment efficiency of lignocellulose and yielding other derivative products. In recent years, numerous studies have validated the feasibility of PMS/PDS pretreatment (Ahmed *et al.* 2016; Zhang *et al.* 2024c). In particular, the potassium peroxymonosulfate (PPMS) system has shown excellent performance in pretreating sugarcane bagasse, achieving a lignin removal of 87.5% and a sugar conversion efficiency of 90.3%, while maximizing cellulose retention. This system also generates more free radicals (·OH and $SO_4^{\cdot-}$) than the PDS system, exhibiting higher selectivity for lignin degradation (Li *et al.* 2023).

Persulfate (PS) can be activated *via* several methods, including energy activation (*e.g.*, UV, ultrasound, heat), catalyst activation (*e.g.*, transition metals, non-metals such as carbon-based materials), alkaline activation (*e.g.*, NaOH), and electrochemical activation (*e.g.*, using boron-doped diamond electrodes) (Peng *et al.* 2021). Each activation method has certain drawbacks. Thermal activation is effective but requires high energy consumption and harsh reaction conditions; ultrasonic activation requires complex instrumentation and has low efficiency when used alone, thus often needing to be coupled with other methods (Fagan *et al.* 2020; Moradnia *et al.* 2022). While homogeneous transition metal ions exhibit high initial reactivity for PS activation, they are prone to rapid oxidation into higher valence states during the process (*e.g.*, the rapid conversion of Fe^{2+} to Fe^{3+}). This restricts the continuous activation of PS and typically results in the generation of metal sludge (Wang and Wang 2018). Alkaline activation of PS is effective for lignocellulosic biomass treatment, but it requires a strong alkaline environment, which may lead to environmental pollution (Song *et al.* 2023); UV activation is renewable, eco-friendly, and considered a promising activation method, though solar activation of PS is less efficient, making the development of effective photocatalysts a key issue (Yang *et al.* 2021; Li *et al.* 2022); heterogeneous metal activation and carbon-based carriers must consider economic and recyclability issues (Mei *et al.* 2021). Carbon-based catalysts such as carbon nanotubes, graphene, and biochar have been used to activate PS, but their catalytic processes involve non-radical activation pathways, and the activation mechanism requires further study (Wu *et al.* 2022). Despite these challenges, recent studies have reported the development of a range of advanced PS activation strategies that exhibit strong activation performance in lignocellulose conversion. For example, a composite material of nano zero-valent iron and biochar was developed to activate persulfate, demonstrating excellent activation efficiency (Qu *et al.* 2024; Guo *et al.* 2025).

It is important to note that during the reaction process, persulfate oxidation systems generate sulfate ion as a by-product. If not handled properly, this may lead to the accumulation of salts in wastewater, thereby increasing the burden of subsequent treatment and posing potential environmental risks (Liu *et al.* 2023). Therefore, related research needs to focus on improving pretreatment efficiency while managing by-product control and waste liquid management. In terms of economics, HR-AOPs have lower unit costs compared to SR-AOPs (Crincoli and Huling 2020), but PS has a lower O-O bond

dissociation energy (140 kJ/mol *vs*. 213 kJ/mol for H_2O_2). A smaller oxidant dosage is typically required to achieve effective activation, partially offsetting the higher unit price. The SR-AOPs have a broader pH adaptability, and under certain activation methods such as transition metal activation, the cost of SR-AOP pretreatment can be lower than that of HR-AOP. For example, PDS pretreatment required only 0.2 g/g of oxidant to achieve the same enzymatic hydrolysis efficiency as the Fenton process (0.85 g/g), which resulted in approximately six times lower costs when reagent unit prices were considered (Ahmed *et al.* 2016).

In summary, SR-AOP can activate persulfate through various methods to generate $SO_4^{\cdot-}$, exhibiting excellent lignin removal and saccharification capabilities enhancement in lignocellulosic pretreatment. However, challenges such as energy consumption, by-product control, and catalyst costs remain key obstacles. Future research needs to focus on improving free radical generation efficiency while ensuring environmental and economic feasibility to promote the practical application of SR-AOP in the clean conversion of lignocellulose.

Ozone pretreatment

Ozone is a highly reactive gas with strong oxidizing properties, possessing a high redox potential (+2.07 V). It can react with C-C, C-N, and their double or triple bonds, and has been widely used for environmental applications such as dye degradation and water purification (M'Arimi *et al.* 2020). Similar to most dye molecules, lignocellulose is rich in aromatic structures and unsaturated bonds, which makes ozone not only an ideal oxidant for water treatment but also a unique agent for the selective degradation of lignin.

Ozone degradation of lignin can proceed through two pathways: direct oxidation and indirect oxidation (Hoigné and Bader 1983). Direct oxidation involves ozone molecules acting as electrophilic reagents, directly attacking electron-rich groups in lignin. This process occurs under mild conditions and can be performed at ambient temperature and pressure. It exhibits high selectivity and preferentially degrades lignin (which contains many unsaturated bonds) while retaining over 90% of the cellulose and hemicellulose in lignocellulose pretreatment by ozone (Travaini *et al.* 2013). However, the reaction rate is relatively slow and limited by the diffusion efficiency of ozone molecules, with the lignin degradation being only about one-third that of indirect oxidation. Indirect oxidation is dominant under alkaline conditions, where ozone decomposes to generate ·OH. The oxidation potential of ·OH radical (2.80 V) is higher than that of O_3, making it more efficient for lignin degradation, with a higher radical transfer rate and better penetration into biomass pores. However, indirect oxidation has lower selectivity and requires precise control of pH and reaction time, making industrial control more challenging. Ozone can also cooperate with H_2O_2 to generate more ·OH radical, enhancing the overall oxidation capacity (Staehelin and Hoigne 1982; Glaze and Kang 1989; Adams *et al.* 1994). Relevant reaction processes include reactions in Eqs. 7- 9:

$$H_2O_2 + 2O_3 \rightarrow 2OH\cdot + 3O_2 \quad (7)$$

$$H_2O_2 \rightleftharpoons HO_2^- + H^+ \quad (8)$$

$$HO_2^- + O_3 \rightleftharpoons OH\cdot + O_2^{\cdot-} + O_2 \quad (9)$$

The effectiveness of ozone pretreatment is significantly affected by reaction time, ozone concentration, and moisture content. Moderate moisture can facilitate ozone diffusion, but excessive water films reduce gas-solid mass transfer efficiency, and

prolonged reaction times can lead to cellulose degradation (Zhang *et al.* 2024a). Studies have shown that ozone not only effectively removes lignin but also disrupts the crystalline structure of cellulose, increasing its accessibility for enzymatic hydrolysis (Zhang *et al.* 2024a). Ozone has been shown to have good pretreatment effects on various lignocellulosic biomasses (Travaini *et al.* 2013; Andersen *et al.* 2019; Shamjuddin *et al.* 2021). In addition to its standalone effect, ozone can also be combined with other methods to further improve sugar yield (Perrone *et al.* 2016; Orduña Ortega *et al.* 2020; Li *et al.* 2021). For example, research has used an ozone-alkali-ultrasound combined system to treat straw, achieving a sugar conversion rate of up to 85% (Perrone *et al.* 2016).

Although ozone pretreatment has significant advantages in selectivity and environmental friendliness, the generation of ozone requires a substantial amount of energy (36 MJ kg^{-1} of ozone), and the required dosage in pretreatment is also high, leading to relatively high operational costs (Osuna-Laveaga *et al.* 2020). Therefore, reducing ozone energy consumption and improving ozone utilization efficiency are key to achieving industrial-scale application. Future research directions include the development of low-energy ozone generation devices (Shamjuddin *et al.* 2021), optimization of reactors (Orduña Ortega *et al.* 2020), use of catalyst-assisted systems, and synergies with other methods, along with economic and life cycle analysis to promote large-scale application.

Photocatalytic pretreatment

Photocatalysis refers to the use of ultraviolet (UV) or visible light as energy to degrade lignin. However, ultraviolet irradiation alone requires prolonged exposure, which limits the overall photocatalytic efficiency. To overcome this limitation, photocatalysts are commonly introduced to enhance lignocellulose degradation. These photocatalysts are generally classified as homogeneous or heterogeneous systems. Homogeneous photocatalysis typically relies on ·OH generated from traditional Fenton reactions to drive oxidative degradation. Under light irradiation, the traditional Fenton system can synergistically promote the generation of ·OH with Fe^{2+} (Eq. 10), and studies have shown that light can reduce the cost of the traditional Fenton system and improve lignin removal (Yang *et al.* 2018). However, this system has limitations such as iron ion loss, the need for acidic conditions, and potential secondary pollution, which restrict its application.

$$Fe^{2+} + H_2O_2 \xrightarrow{h\nu} Fe^{3+} + \cdot OH + OH^- \quad (10)$$

The heterogeneous photocatalytic process mainly utilizes semiconductor photocatalytic materials (such as TiO_2, Bi_2O_3, Fe_2O_3, *etc*.) for photocatalysis. Taking TiO_2, a widely used photocatalyst, as an example, when the energy irradiated on the photocatalyst exceeds its bandgap, electron-hole pairs ($TiO_2(h^+)$) and free electrons (e^-) are generated within the semiconductor (Lee *et al.* 2016; Yang and Wang 2018), as shown in reaction (11):

$$TiO_2 \xrightarrow{h\nu} TiO_2(h^+) + e^- \quad (11)$$

The electron-hole pairs ($TiO_2(h^+)$) react with H_2O and OH^- on the surface of the material, generating ·OH (Eqs. 12 and 13) (Yang and Wang 2018). The ($TiO_2(h^+)$) can also directly react with the substrate (*RH*), as shown in reaction (14).

$$TiO_2(h^+) + H_2O \rightarrow TiO_2 + \cdot OH + H^+ \quad (12)$$

$$TiO_2(h^+) + OH^- \rightarrow TiO_2 + \cdot OH \quad (13)$$

$$TiO_2(h^+) + RH \rightarrow TiO_2 + R\cdot + H^+ \quad (14)$$

The e^- reacts with O_2 on the surface of the material, generating $O_2^{\cdot-}$ and the e^- further oxidizes $O_2^{\cdot-}$ to generate H_2O_2, as shown in reactions (15) and (16). The interactions among e^-, $O_2^{\cdot-}$, and H_2O_2 further generate ·OH (reactions (17) and (18)) (Lee *et al.* 2016). The ·OH produced in reactions (12), (13), (17), and (18) react with the substrate, thus degrading lignin and hemicellulose.

$$O_2 + e^- \rightarrow O_2^{\cdot-} \quad (15)$$

$$O_2^{\cdot-} + e^- + 2H^+ \rightarrow H_2O_2 \quad (16)$$

$$O_2^{\cdot-} + H_2O_2 \rightarrow \cdot OH + HO^- + O_2 \quad (17)$$

$$e^- + H_2O_2 \rightarrow \cdot OH + HO^- \quad (18)$$

Among many semiconductor-based photocatalytic materials, TiO_2 is widely studied due to its non-toxicity, stability, corrosion resistance, and absence of secondary pollution. It has been shown to effectively degrade lignin and improve enzymatic sugar yields (Alvarado-Morales *et al.* 2017; Lee *et al.* 2019; Sabeeh *et al.* 2020). However, its application still faces challenges, such as activation only under UV light, easy recombination of photogenerated charge carriers, and difficulty in recovering powdered catalysts. To address these shortcomings, researchers have proposed modification strategies such as ion doping, noble metal deposition, semiconductor composites, and carbon-based material loading to expand the visible light response range and improve the electron-hole separation efficiency (Park *et al.* 2013; Elleuch *et al.* 2020; Zhang *et al.* 2021; Xu *et al.* 2023). In addition to TiO_2, photocatalyst materials such as Bi_2O_3, Fe_2O_3, ZnO, and $ZnIn_2S_4$ have also shown various advantages in terms of visible light response, stability, and selectivity (Zewde *et al.* 2019; Awais *et al.* 2020; Kumar *et al.* 2021; Cao *et al.* 2024).

In recent years, photocatalytic pretreatment has also gradually been combined with other processes to overcome the limitations of single methods (Yang *et al.* 2018; Jia *et al.* 2022). For example, the TiO_2/UV/H_2O_2 synergetic system has significantly removed lignin and improved enzymatic sugar yield during the treatment of straw and sisal waste (Yang *et al.* 2018). Overall, photocatalytic pretreatment offers significant advantages such as mild conditions, green processes, and controllability. However, its industrial application is still limited by insufficient photon utilization, difficulties in reactor scaling, and high energy consumption costs. Future research should focus on designing highly efficient visible-light-responsive catalysts, optimizing light fields and reactor structures, and coupling photocatalysis with other pretreatment methods to achieve efficient hierarchical conversion of lignocellulose and promote the greening of biorefining processes.

Electrochemical pretreatment

Electrochemical pretreatment technology is a synergistic method that combines electrolysis with chemical reactions. It offers advantages such as being environmentally friendly, producing minimal by-products, strong controllability, and requiring no external chemical oxidants (Stiefel *et al.* 2016; Garedew *et al.* 2021). In recent years, it has gradually been applied to the pretreatment of lignocellulosic biomass (Du *et al.* 2020). This method applies a direct current electric field to generate highly active free radicals (such as ·OH) in the reaction system, which initiate the depolymerization of lignin (Wang *et al.*

2015; Panigrahi and Dubey 2019). It also utilizes effects such as electrophoresis, electroosmosis, and ohmic heating to promote structural disruption and component dissolution (Shao *et al.* 2014; Panigrahi and Dubey 2019). Common electrode materials include PbO_2, IrO_2, Ti/Sb, Ti/PbO_2, Ni, and graphite (Di Marino *et al.* 2016; Cai *et al.* 2018; Di Fidio *et al.* 2021). For instance, the use of Pb/PbO_2 anodes and Cu/Ni-Mo-Co cathodes in alkaline solutions was shown to effectively treat corn stover, achieving lignin degradation with the simultaneous production of hydrogen. After 6 cycles of electro-catalysis, the lignin product conversion efficiency reached 96.8% (Cai *et al.* 2018). Furthermore, saponification reactions can occur under high pH conditions, further altering the lignin structure and enhancing biomass digestibility (Di Marino *et al.* 2016).

Although electrochemical methods show unique advantages in improving lignocellulose degradability and achieving high-value utilization of lignin (Wijaya *et al.* 2020), their practical application is still limited by issues such as electrode passivation and corrosion, increased energy consumption at high current densities, and the generation of inhibitory by-products (Shao *et al.* 2014). Current research mostly remains at the laboratory stage, with a lack of overall system integration and energy efficiency assessments for continuous and large-scale operations. To address these bottlenecks, research is gradually shifting towards material and process optimization, such as developing low-cost, highly active, and corrosion-resistant novel electrode materials, or designing compact and efficient reactor structures (Liu *et al.* 2012; Bawareth *et al.* 2019; Zhu *et al.* 2020). Additionally, synergistic strategies are receiving increasing attention, such as the electro-Fenton system (Liu *et al.* 2012), the combination of electrochemical and photocatalysis (Fernandes *et al.* 2021), the combination of electrochemistry and peracetic acid (Yuan *et al.* 2021), or integration with deep eutectic solvents or ionic liquids (Fernandes *et al.* 2021). These studies indicate that synergistic applications provide possibilities for overcoming the current limitations of electrochemical methods and enhancing their role in the efficient conversion of lignocellulose.

Wet air oxidation pretreatment

Wet air oxidation (WAO) is one of the most commonly used lignocellulosic pretreatment methods, typically carried out at 125 to 250 °C and 3 bar to 35 bar pressure conditions to dissolve lignin and hemicellulose (McGinnis *et al.* 1983). The WAO process generates free radicals (such as ·OH, ·HO_2, ROO·) through the interaction of oxygen with the C-H bonds in organic compounds, initiating chain reactions that disrupt the lignocellulosic structure and produce low-molecular-weight products such as organic acids (Tembhekar *et al.* 2015; Demesa *et al.* 2020). The reaction mechanism is as follows:

Oxygen oxidizes the C-H bonds in organic compounds under high temperature and high pressure, generating organic free radicals (R·) and HO_2· (reaction (19)). These R· react with molecular oxygen to form organic peroxyl radicals (ROO·) (reaction (20)).

$$RH + O_2 \rightarrow R\cdot + HO_2^{\cdot} \quad (19)$$

$$R\cdot + O_2 \rightarrow ROO\cdot \quad (20)$$

The rapid decomposition of hydrogen peroxide generates ·OH (reaction 21).

$$H_2O_2 + O_2 \rightarrow HO_2^{\cdot} + \cdot OH \quad (21)$$

$HO_2^{\cdot}$ and ·OH oxidize organic compounds, generating more R· (reactions 22 and 23).

$$RH + HO_2^{\cdot} \rightarrow R\cdot + H_2O_2 \quad (22)$$

$$RH + \cdot OH \rightarrow R\cdot + H_2O \quad (23)$$

ROO· can extract a hydrogen atom from other organic compounds (reaction (24)), leading to the formation of organic peroxides, or ROO· can react with each other (reaction (25)) to generate stable products.

$$ROO\cdot + RH \rightarrow ROOH + R\cdot \quad (24)$$

$$2ROO\cdot \rightarrow ROOR + O_2 \quad (25)$$

The WAO process is an exothermic reaction, so once the reaction is initiated, some of the energy can be self-supplied, reducing the need for external energy input (Gout *et al.* 2022). Additionally, this method does not require large amounts of expensive chemical reagents, and the by-products are mainly organic acids and soluble sugars, making the overall process relatively low in operating costs (Szijártó *et al.* 2009; Poveda-Giraldo and Cardona Alzate 2025). In recent years, the introduction of transition metal oxides (Cu, Co, Mn, Fe, Zn) and noble metals (Ru, Rh, Pt, Ir, Pd) as catalysts has further reduced the dependence on high temperature and high pressure (Tekin *et al.* 2022). Furthermore, alkaline WAO, alkaline peroxidized WAO, and catalytic WAO technologies have been developed. These methods exhibit higher cellulose degradation rates and lignin removal efficiencies compared to uncatalyzed WAO (Castro and Agblevor 2022). For example, wheat bran was pretreated by WAO at 170 °C and 12 bar, followed by forward osmosis (FO) membrane concentration, which yielded a xylose concentration of 13.75 g/L (Bhavana *et al.* 2023). Efficient depolymerization of alkaline lignin was achieved under WAO pretreatment at 150 °C and 1000 psi, leading to the generation of vanillic acid (Irmak *et al.* 2020). Similarly, the use of $CuSO_4/Fe_2O_3$ catalysts during WAO treatment of bamboo at 195 °C and 6 bar yielded 14.9 wt% bio-oil, with vanillin identified as the primary product (McCallum *et al.* 2021).

The main factors affecting WAO efficiency include the nature of the treated material, reaction temperature, reaction time, reaction pressure, solution pH, and the addition of catalysts. Longer residence times favor lignin removal and enhance cellulose recovery. At temperatures exceeding 100 °C, the rising temperature increases dissolved oxygen concentration due to elevated saturated vapor pressure, thereby promoting lignin oxidation, although the solubility of hemicellulose decreases. Moderate pressurization can further facilitate lignin degradation by increasing dissolved oxygen levels. However, the effect of pressure on cellulose recovery is relatively limited, and excessively high pressures significantly increase equipment costs; therefore, practical applications should balance energy consumption and treatment efficiency to determine an appropriate operating pressure (Morone *et al.* 2018b). Overall, WAO demonstrates good pretreatment effects and has high application potential, but issues related to equipment corrosion and energy consumption have yet to be adequately addressed. Future developments should focus on coupling with other pretreatment methods, optimizing catalysts and reactors, and achieving efficient component separation and high-value utilization while reducing energy consumption and operational costs.

Cavitation-based advanced oxidation processes

Cavitation-based advanced oxidation processes (Cavitation-AOPs) have recently been considered one of the most promising technologies for lignocellulose pretreatment. The core mechanism involves the formation, growth, and violent collapse of bubbles in the

liquid (Madison *et al.* 2017). During bubble collapse, extreme conditions are created locally, with instantaneous temperatures reaching 10,000 K and pressures exceeding 500 atm, accompanied by intense shear forces, turbulence, and microjets, which generate strong oxidative species such as ·OH, ·H and $O_2^{\cdot-}$ (Badve *et al.* 2014; Madison *et al.* 2017; Devadasu *et al.* 2020). These physical-chemical effects can effectively break down the dense structure of lignocellulose, promote lignin dissolution, and enhance enzymatic hydrolysis accessibility. Based on the energy input method, cavitation can be divided into two types: ultrasonic cavitation and hydrodynamic cavitation.

Ultrasonic cavitation (UC) refers to the generation, and collapse of bubbles induced by alternating compression and rarefaction in the liquid phase within the sonic frequency range of 20 kHz to 2 MHz. This process simultaneously generates free radicals and strong physical disruption (Passos *et al.* 2014; Costa *et al.* 2020). In lignocellulosic pretreatment, ultrasound can break the lignin-carbohydrate bonds, disrupt the crystalline structure of cellulose, and enhance solvent permeability, showing high lignin removal efficiency (Singh *et al.* 2014). Studies have shown that ultrasonic-assisted hydrogen peroxide pretreatment of sawdust increased lignin removal by 2 to 3 times (Devadasu *et al.* 2020). In the treatment of sisal waste, ultrasound combined with a $UV/TiO_2/H_2O_2$ system achieved over 70% lignin removal and significantly improved enzymatic sugar yields (Yang *et al.* 2018). After pretreating rice straw for fermentation to produce lactic acid, combinations of ultrasound with acid, alkali, or H_2O_2 increased lactic acid yields by 100%, 104%, and 109%, respectively (Askarniya *et al.* 2025). Ultrasonic cavitation has significant advantages in lignin removal and structural disruption, and it can synergize with various chemical methods; however, its application is limited by high energy consumption, high equipment costs, and difficulty in large-scale promotion.

Hydrodynamic cavitation (HC) involves the generation of local pressure drops in the liquid, induced by devices such as venturi tubes, orifices, or valves, which reduce fluid pressure below the saturated vapor pressure, leading to bubble formation and collapse. Its mechanism is similar to that of UC, generating high local temperatures and pressures, as well as free radicals, which disrupt the biomass structure through high-speed microjets and shock waves (Gogate and Pandit 2005; Madison *et al.* 2017). Alkali-assisted HC has shown excellent lignin removal effects in the pretreatment of various lignocellulosic materials such as sugarcane bagasse, reed, and miscanthus (Kim *et al.* 2015; Terán Hilares *et al.* 2017; Bimestre *et al.* 2020; Lee and Han 2021; Tsalagkas *et al.* 2021). Compared to ultrasound, the advantages of HC lie in its simple equipment structure, requiring only pumps and pipes, low energy consumption, and ease of scaling up to industrial levels. However, its cavitation intensity and free radical production rates are typically lower than those of ultrasound, which may limit its effectiveness in standalone applications.

Overall, cavitation-based AOPs provide an important green pretreatment pathway for lignocellulose. Future research should focus on improving reactor design to enhance energy conversion and cavitation uniformity; combining cavitation with other oxidants (such as H_2O_2, ozone, persulfates) to enhance treatment effectiveness; and conducting techno-economic and life cycle assessments to verify its feasibility in the industrialization of biorefining.

Potential Impacts of Residual Metals and Oxidation By-products on Downstream Bioconversion

Advanced oxidation pretreatment frequently involves transition metals (*e.g.*, Fe, Co, Mn) and strong oxidants, raising concerns about the potential influence of residual

metal species and oxidation by-products on subsequent enzymatic hydrolysis and fermentation. In iron-based systems such as Fenton and Fenton-like processes, soluble Fe^{2+}/Fe^{3+} species may remain associated with the solid or liquid fractions after pretreatment. Although iron is an essential micronutrient for many microorganisms, excessive metal concentrations can interfere with enzyme activity through nonspecific binding, alteration of redox conditions, or promotion of secondary oxidative reactions. It is important, however, to distinguish between transient radical species and stable residues (Di Fraia *et al.* 2024). Reactive radicals such as ·OH and $SO_4^{\cdot-}$ exhibit extremely short lifetimes (typically in the nanosecond to microsecond range) and therefore they do not persist into downstream hydrolysis or fermentation stages (Zhang *et al.* 2024b). Observed inhibitory effects are more likely related to excessive metal accumulation or to soluble lignin-derived oxidation products generated under severe conditions. Partial oxidation of lignin can produce low-molecular-weight phenolics, quinone-type intermediates, organic acids, or aldehydes, some of which are known to affect cellulase activity or microbial metabolism when present at high concentrations (Liu *et al.* 2021b) Nevertheless, under controlled oxidant dosage and optimized reaction conditions, many studies report enhanced enzymatic digestibility without significant inhibition, indicating that selective modification of lignin-rich domains can improve accessibility while maintaining downstream compatibility (Toghiani *et al.* 2024; Zhang *et al.* 2024c). In practice, washing, neutralization, solid-liquid separation, or catalyst recovery steps are typically employed to minimize residual metal and soluble inhibitor levels prior to enzymatic processing (Zhang *et al.* 2024c). Despite these mitigation strategies, systematic evaluation of residual metal thresholds and inhibitor tolerance in integrated bioconversion systems remains limited, highlighting the need for standardized reporting and coupled enzymatic performance assessments in future studies.

Comparison of the Advantages, Disadvantages, and Application Effects of Different AOP Pretreatments

The various AOP pretreatment methods of lignocellulosic biomass are compared in Table 1, which outlines their advantages and disadvantages and analyzes their application status. Overall, the Fenton system has been widely studied and applied due to its ability to efficiently degrade lignin and hemicellulose by rapidly generating ·OH under mild conditions. However, its dependence on acidic conditions and the issue of iron salt residue limit its industrial potential. The Fenton-like methods, by introducing heterogeneous catalysts, have expanded the pH range, and improved catalyst recyclability, though there is still room for improvement in stability and cost reduction.

Alkaline hydrogen peroxide (AHP) offers the advantages of being environmentally friendly and operating under mild conditions, effectively breaking down lignin structures and promoting cellulose accessibility. However, it requires a large amount of oxidants and has long reaction times and is still primarily at the laboratory and pilot study stages. Peracetic acid (PAA) has gained attention due to its higher oxidation potential and selective lignin removal, working efficiently at lower temperatures. Its lack of stability and high synthesis cost limit its widespread application. Persulfates (PDS/PMS) generate $SO_4^{\cdot-}$, which have a longer lifetime and higher stability than ·OH, showing excellent lignin removal ability and a broad pH applicability range. However, activation methods and system economics still need optimization. Ozone is highly selective for the degradation of aromatic structures and can remove lignin while retaining carbohydrates, but its high energy consumption and complex equipment requirements limit its feasibility.

Photocatalysis and electrochemical methods are notable for their environmental friendliness and controllability. The former relies on light to generate ·OH, while the latter uses electrode reactions for oxidative decomposition. Both avoid the introduction of large amounts of external chemicals, but photon utilization, electrode material stability, and energy efficiency issues remain challenges for large-scale applications. Wet air oxidation (WAO) can efficiently degrade lignin structures in a short time and offers potential for high-value utilization of by-products under certain conditions, though it requires harsh operating conditions and high equipment requirements. Cavitation oxidation (including UC and HC) generates high temperatures, pressures, and free radicals through bubble collapse, significantly improving substrate structure, and can synergize with acids, alkalis, or peroxides. However, its high energy consumption and limited cavitation intensity in hydrodynamic cavitation still require further verification for industrial applications.

In general, different AOP methods have distinct characteristics in terms of efficiency, selectivity, and application potential. Fenton, AHP, and PAA can achieve efficient lignin removal under mild conditions; ozone offers high selectivity for aromatic structure degradation and can remove lignin while retaining carbohydrates; WAO demonstrates rapid lignin degradation and potential for by-product valorization under high temperature and high pressure. Photocatalysis, electrochemical methods, and cavitation processes have been widely studied for their green, sustainable, and controllable properties. Persulfate oxidation, which produces $SO_4^{\cdot -}$ with long half-lives, high stability, and strong oxidative capacity, has seen rapid development in recent years, particularly in photocatalytic activation, transition metal activation, and carbon-based catalyst activation. It may become one of the most promising AOPs in the future.

Table 2 presents typical case studies of AOP pretreatment of lignocellulosic biomass and their application effects. From Table 2, it is evident that different AOP methods exhibit varying pretreatment effects under different raw materials and operating conditions.

Fenton, Fenton-like, alkaline hydrogen peroxide, peracetic acid, and persulfate have particularly outstanding pretreatment effects. Photocatalysis and electrochemical methods show milder effects but still effectively improve enzymatic hydrolysis rates. In addition, ozone, wet air oxidation, and cavitation methods also show good potential in lignin removal and enhancing enzymatic hydrolysis efficiency. The differences in application effects reflect the potential influence of multiple factors such as raw materials, pretreatment conditions, and catalysts, with further optimization of operating conditions enhancing the overall pretreatment effect.

Table 1. Comparison of the Advantages, Disadvantages, and Application Status of Different AOP Pretreatments

Method	Advantages	Disadvantages	Application Status
Fenton Pretreatment	High reaction efficiency, rapid generation of ·OH, effective degradation of lignin and hemicellulose; mature process, simple operation	Requires acidic conditions (pH≈3), large amounts of acid and base for adjustment, iron salt residue leading to wastewater treatment challenges, potential formation of inhibitory compounds	Widely used in wastewater treatment, primarily experimental in lignocellulose pretreatment; new processes like electro-Fenton and photo-Fenton are under development
Fenton-like Pretreatment	Mild operating conditions; broad pH range; catalysts are easily separated and recyclable	Catalyst stability and economic feasibility need optimization; some new catalysts are still in experimental stages	Overcomes some issues of conventional Fenton methods, but continuous optimization and maturation are required
Alkaline H_2O_2 Pretreatment	Environmentally friendly, with products of H_2O and O_2, no toxic by-products generated	High concentrations of H_2O_2 are costly and hazardous; strict control of reaction conditions is required to avoid by-product formation	More research at the experimental and pilot scales are required; shows good results when combined with other advanced oxidation techniques
Peracetic Acid Pretreatment	High oxidation potential, strong selectivity for lignin removal; effective at low temperatures; can be combined with other methods to enhance effect	High chemical synthesis cost, safety risks	Suitable for biomass with high lignin content, mainly at the laboratory research stage, with biological catalysis for PAA synthesis as a research focus
Persulfate Pretreatment	Generates $SO_4^{\cdot-}$ with long half-lives, high stability, broad pH range, and strong oxidation capacity, resulting in good pretreatment effects	Multiple activation methods but face energy consumption or economic feasibility issues; some catalysts have limited stability and recyclability	Research has rapidly increased in recent years; has shown high efficiency in pretreating materials such as rice straw and bamboo, currently at the experimental stage
Ozone Pretreatment	High oxidation potential, selective for lignin degradation, retains cellulose and hemicellulose; operates under ambient temperature and pressure	High energy consumption for ozone generation, expensive equipment	Mature in water treatment, good results in lignocellulose pretreatment, primarily used in combination with other methods, showing excellent performance
Photocatalytic Pretreatment	Environmentally friendly, strong controllability, uses light energy to generate ·OH for lignin degradation; numerous new catalysts emerging	Low photon utilization, catalysts easily deactivate or hard to recover; limited scalability	Active experimental research shows good performance when combined with Fenton or H_2O_2 synergies

Electrochemical Pretreatment	No need for external chemical oxidants, process is green and controllable; can simultaneously achieve lignin breakdown and by-product utilization	Electrode corrosion and passivation; high energy consumption at high current densities	Primarily at the experimental stage; developing corrosion-resistant electrodes and new reactor designs is a key development direction
Wet Air Oxidation Pretreatment	Efficiently degrades lignin structure; by-products can be valorized; catalytic WAO reduces energy consumption requirements	Equipment corrosion and energy consumption issues remain unresolved	Mature process, widely used in wastewater and sludge treatment; lignocellulose pretreatment is still at the experimental stage
Cavitation-based Pretreatment	Bubble collapse generates extreme conditions, synergizes physical disruption and chemical oxidation; can be used in combination with other pretreatment methods	Ultrasonic cavitation has high energy consumption and scalability issues; hydrodynamic cavitation has limited cavitation intensity, and independent application effectiveness still needs verification	Active experimental research shows good potential in synergistic pretreatment

Table 2. Application Cases of Various AOP Pretreatment Methods

Pretreatment Method	Material	Operating Conditions	Pretreatment Effect	References
Fenton	Poplar	0.01 mol/L Fe^{2+}, 1 mol/L H_2O_2; 0.025 °C; 150 rpm; 48 h	Maximum enzymatic hydrolysis yield of 406 mg/g	(Kato *et al.* 2014)
Fenton-like	Corn stover	Thioglycolic acid; 25 °C; pH 7.0; 16 h	Glucose yield of 86%	(Wang *et al.* 2022)
Alkaline H_2O_2	Bamboo	H_2O_2 (2%); pH 11.5; 90 °C; solid-liquid ratio 1:10; 1 h	Lignin removal rate 70%, glucose yield 80.15%	(Huang *et al.* 2022)
Peracetic Acid	Corn stover	Peracetic acid-maleic acid; 130 °C; 30 min	Glucose yield of 89.65%	(Lyu *et al.* 2021)
Persulfate	Rice straw	75 mmol/L potassium persulfate; 120 °C; 2 h	Enzyme digestion rate of 91%	(Ahmed *et al.* 2016)
Ozone	Wheat straw	Ozone concentration of 2.7% (w/w); air/ozone flow rate 60 (L/ha); 2.5 h	Enzymatic hydrolysis rate increased to 53% to 88.6%	(García-Cubero *et al.* 2009)
Photocatalysis	Rice straw	1.5% NaOH; 2 g/L TiO_2 nano catalyst; photocatalysis for 1 h; 40 °C	Enzymatic hydrolysis rate of 73.96%	(Niu *et al.* 2009)

Pretreatment Method	Material	Operating Conditions	Pretreatment Effect	References
Electrochemical	Wheat straw	Stainless steel-supported molybdenum carbide electrode (SS-Mo_2C); 50 mL pure water and 0.2 mol/L NaOH; 1.5V; 6 h	Yield of reducing sugars of 3.98 mg/mL	(Fang *et al.* 2022)
Wet Air Oxidation	Rice straw	Wet air oxidation reactor; 6.5 g/L Na_2CO_3; 220 rpm; 169 °C; 4 bar; 18 min	Enzyme digestion rate of 42.85%	(Morone *et al.* 2018a)
Cavitation Oxidation	Sawdust	Longitudinal horn-type ultrasonic reactor; 36 kHz; 1M H_2O_2; solid-liquid ratio 1:10; 343K; 70 min	Lignin removal of 87.4%	(Devadasu *et al.* 2020)

PROSPECTS FOR AOP PRETREATMENT

Advanced oxidation processes (AOPs) can generate highly oxidative free radicals such as ·OH, $SO_4^{\cdot-}$, and $O_2^{\cdot-}$, offering high efficiency, environmental friendliness, and non-toxicity in lignocellulose pretreatment. Compared to traditional acid-base and physical methods, AOPs have certain advantages in terms of environmental impact and energy consumption. Currently, research on AOP pretreatment of lignocellulose is mainly at the laboratory or pilot stage, with no large-scale industrial application, primarily due to economic feasibility issues. On the one hand, most AOP systems rely on high-cost oxidants, catalysts, or reaction equipment, leading to high operating costs. On the other hand, there is a lack of comprehensive techno-economic analysis (TEA) of AOPs, making it difficult to evaluate the investment cost versus the economic or environmental benefits generated. To promote the industrialization of AOPs in the future, several approaches can be considered: (1) Conducting techno-economic analysis (TEA) and life cycle assessments (LCA) to determine whether a process has investment benefits or needs further optimization for large-scale industrial application; (2) Focusing on high-value utilization of pretreatment products to directly increase the process's profitability; (3) Further understanding the radical reaction mechanisms and by-product generation pathways to achieve more precise process control and optimize process parameters (such as temperature, pressure, and the ratio of raw materials to oxidants); (4) Developing new low-cost, high-efficiency, recyclable catalysts, optimizing reactor design, or developing dual/multi-mechanism synergistic technologies to improve reaction efficiency; (5) Promoting the coupling of AOPs with green energy sources (such as solar energy, bioelectricity) and downstream product upgrading systems to improve overall energy efficiency.

Overall, although AOP pretreatment currently faces economic and technical barriers for large-scale application, it offers significant advantages in selective oxidation, reaction activity, environmental sustainability, and the diversity of system activation methods and synergistic effects. The potential for process optimization is vast, and AOPs are considered to have a strategic position in future biorefining systems. With continued technological innovation and system integration, advanced oxidation technologies are expected to become key pillar technologies in the production of second- and third-generation green biofuels.

CONCLUDING STATEMENTS

This review has provided a comprehensive analysis of nine major advanced oxidation processes (AOPs) for lignocellulosic biomass pretreatment, including Fenton and Fenton-like, alkaline hydrogen peroxide, peracetic acid, persulfate, ozonolysis, photocatalytic, electrochemical, wet air oxidation, and cavitation. These methods show significant potential for enhancing lignin removal and enzymatic efficiency, particularly under mild conditions. Among these, persulfate, wet air oxidation, and combinations of multiple pretreatment technologies (such as light-Fenton, alkaline H_2O_2-cavitation, and electrochemical-ozone-H_2O_2) offer the most promising prospects for future applications. To achieve large-scale industrial implementation, future research should focus on developing novel catalysts, optimizing reactor design, and integrating multiple

pretreatment techniques to reduce costs and improve efficiency, ultimately advancing the sustainable utilization of lignocellulosic biomass.

ACKNOWLEDGEMENTS

This work was supported by Open project of Xinjiang Biomass Solid Waste Resources Technology and Engineering Center (KSUGCZX202408); Fundamental Research Funds for the Central Universities (No. 2662023DKPY003), China.

Author Contribution

Zhang Xueshuai: Writing – original draft, writing – review and editing; Huang Mengtian: Writing – original draft, writing – review and editing, Methodology; Long Yao: Writing – original draft, visualization; Li Qihang: Methodology; Cao Yao: Methodology; Zhou Wenbing: Conceptualization, Writing – review and editing, Methodology and Supervision; Xiao Naidong: Methodology; Cai Jianbo: Methodology.

Funding

This work was financially supported by Xinjiang Biomass Solid Waste Resources Technology and Engineering Center (KSUGCZX202408) and Fundamental Research Funds for the Central Universities (No. 2662023DKPY003), China.

Declarations

The authors declare no competing interests.

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Article submitted: January 31, 2026; Peer review completed: February 21, 2026; Revised version received and accepted: May 26, 2026; Published: June 11, 2026.
DOI: 10.15376/biores.21.3.Zhang2