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REVIEW ARTICLE

Progress in applications of advanced oxidation processes for promotion of biohydrogen production by fermentation processes

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Abstract

Advanced oxidation processes (AOPs) are powerful methods for treating substrates using radicals that are generated in situ. This study reviewed applications of AOPs in enhancement of biohydrogen production. The AOPs are applied in substrate pretreatment because of their ability to break the complex structure of lignocellulosic biomass for ease of subsequent hydrolysis. The mechanism of solubilization of complex organics resulting in increased biodegradability of substrate during pretreatment has been suggested. Documented studies indicate that up to 98% color removal from organic wastewater is possible by the use of AOPs. Furthermore, a combination of AOPs with biological processes can achieve more than 90% COD removal from biohydrogen production effluent. Sonication, microwave-enhanced AOPs, and electrochemical treatment are the most applied AOPs in enrichment of biohydrogen with up to fivefold increase in biohydrogen yield achieved after electrochemical pretreatment. The mechanism of enhancement of hydrogen yield in dark fermentation after pretreatment of the substrate and inoculum with AOPs has been proposed. The excess sludge produced during hydrogen fermentation can be pretreated with ozone and ultrasound before biomethanation process. More studies on co-production of biohydrogen and electricity through electrochemical oxidation in fuel cells are necessary. This study proposes the integration of AOPs with conventional processes in biorefinery production approach with aim of improving biohydrogen yields, co-producing it with other biofuels, and reducing the process costs. Future studies should focus on the scale-up of AOPs for commercial applications. Comparative studies on energy requirements for various AOPs applications are lacking and should be carried out.

Keywords Biohydrogen inoculum · Microbial fuel cell · Biodegradability enhancement · Bioenergy substrate treatment · Anaerobic digestion · Excess sludge treatment

Highlights
1. Mechanism of AOP Pretreatment of biohydrogen substrate given
2. Treatment of bioenergy effluent for color and COD by AOPs reviewed
3. Use of AOPs to co-produce H_2 with different energy forms like MFC shown
4. Biorefinery production concept can be promoted by use of AOPs
5. AOPs can be used to enrich biohydrogen inoculum and treat excess sludge

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1 Introduction

Biohydrogen gas is among the promising biofuels that has generated great interest in recent past as a potential alternative energy source to fossil fuels. It has a high energy density compared to other hydrocarbons and biofuels. The energy yield per mole from hydrogen gas is more than 2.5 times higher than the energy generated from liquid petroleum fuels [1]. Furthermore, its combustion does not result in the release of greenhouse emissions. Hydrogen can be produced biologically though various processes including dark fermentation [2, 3], biophotolysis [4], dark fermentation [5, 6], photofermentation [7], and microbial electrolysis cell [8].

Dark hydrogen fermentation process (DHFP) occurs in the absence of oxygen and does not require light. The process entails the use of specialists' microbes on organic substrates in anaerobic digestion. Furthermore, the DHFP is simple and most of organic materials that can be used as substrate for biohydrogen production are readily available. These include food wastes [9], animal wastes [10], organic wastes effluent [11], solid wastes [12], lignocellulosic biomass [13], activated sludge [14], and agricultural residues [15]. De almeida Silva (2020) reported a possibility of producing hydrogen and volatile acids products using glycerol substrate [16].

In dark fermentation, organic substrates are metabolized by the microbes to produce energy. In DHFP, excess electrons are produced which in the absence of external electron acceptor reduces protons to produce hydrogen. The first step of DHFP when using complex substrates like starch or cellulose substrate is hydrolysis to produce simple sugars which then undergo glycolysis to produce pyruvate. The pyruvate undergoes metabolic reactions in the presence of cofactors and enzymes to produce formate or acetyl coenzyme A. Furthermore, metabolism of the two in anaerobic conditions and catalyzed by right enzymes in the presence of cofactors produces biohydrogen and volatile products. The metabolic pathway of conversion of sugar to biohydrogen through dark fermentation has been elaborated [17]. The two types of hydrogenase enzymes involved in dark hydrogen fermentation are (FeFe) and (NiFe) hydrogenases. The reactions involved in hydrogen production is as shown in Eq. 1;

$$2H^{+} + 2\overline{e} \rightarrow H_2 \tag{1}$$

The other factors involved in dark fermentation include nicotinamide adenine dinucleotide phosphate (NADP), Nicotinamide adenine dinucleotide (NAD), flavin adenine dinucleotide (FAD), and ferredoxin [18]. The products of the DHFP include hydrogen, volatile acids like acetate, propionate, and butyrate [19, 20]. The yield of hydrogen and volatile acids produced is dependent on the process-operating conditions like temperature, pH, retention time, and organic loading. Furthermore, biohydrogen yield is greatly affected by the type of biohydrogen microbes used and the presence of competing microbes [19].

The main limitation of biohydrogen production from organic substrates by fermentation method is low energy yield and productivity. Multi-facet strategies have been tried for improvement of the yield and productivity of biohydrogen production from biomass substrate. These include modifying of the biohydrogen reactor to enhance the retention of biohydrogen substrates and optimizing the bioreactor conditions. Other strategies include enrichment of biohydrogen specialist culture by selectively eliminating the microbial competitors which utilize the substrate to produce other products and by-products at the expense of biohydrogen. The use of advanced oxidation processes (AOPs) to pretreat biohydrogen inoculum and eliminate competing microbes is among the strategies which are generating great interest among researchers and industrial practitioners. Other methods commonly applied to achieve the same include heating and chemical treatment.

Biohydrogen yields can also be enhanced through pretreatment of substrates which can be achieved through various methods including chemical treatment [21], heat treatment [22], and thermo-chemical methods [23]. The traditional method of biomass substrate pretreatment which has wide application in biohydrogen fermentation is alkali-based thermo-chemical treatment [23]. Furthermore, nanoparticles including Ni^{2+} and Fe^{2+} have been reported to increase biohydrogen yield in dark hydrogen fermentation [2, 3]. In recent past, AOPs have found useful applications as alternative pretreatment for breaking the complex lignocellulosic structure of biomass substrates [24]. This helps in exposing hemicelluloses which are then hydrolyzed to fermentable sugars. In other applications, AOPs are used to selectively oxidize the recalcitrant which enhances biodegradability of the substrates and results in higher biohydrogen yields.

Advanced oxidation processes entail the utilization of in situ generated radicals to pretreat the substrate. The radicals are oxidants that are powerful in reacting and mineralizing the recalcitrant in biofuel substrates. The AOPs were traditionally used for the remediation of hazardous materials [25] but they have found other uses including promotion of bioenergy production. Some of these new applications in bioenergy production include pretreating of the substrate and final treatment of the effluent from the bioenergy production processes. One of the main advantages of using AOPs as a treatment method is their ability to increase biodegradability of the substrate.

Moreover, the AOPs are very selective towards unstable bonds. Many recalcitrant, toxicants, and colorants in bioenergy substrate and effluents contain unstable bonds like in phenolic compounds which are selectively mineralized by the AOP processes [26]. Various AOP processes commonly applied in bioenergy treatment include Fenton, ozonation, ultraviolet treatment, ultrasonication, photocatalysis, microwave enhanced AOPs, hydrogen peroxidation, electrochemical oxidation, and wet air oxidation. This paper reviews the application of AOPs in the promotion of biohydrogen production. Moreover, the possibility of using AOPs to promote co-production of biohydrogen with other energy types like bioelectricity and biogas has been discussed.

2 Enhancement of biohydrogen production from fermentation by various methods

The low yields and productivity of biohydrogen fermentation has stirred investigations globally on how to enhance the same [27]. A lot of investigations have been carried out on various aspects of biohydrogen production process including substrates choice, bioreactor technologies and their modifications, microbial species choice and enrichment, metabolic engineering, and process optimization. The use of AOPs has also been incorporated in these investigations. It is expected that in the near future, the combination of results from these studies will enable cost effective production of biohydrogen by fermentation.

2.1 Microbial species choice

Mixed culture which is a common inoculum for anaerobic digestion contains many species of micro-organisms including methane-producing bacteria (methanogens), hydrogen-producing bacteria, and sulfur-reducing bacteria. These microbes compete for the substrates which reduces the yields of biohydrogen. The slow growth of biohydrogen specialists compared to methanogens and other competing microbes in normal culture enables the competitors to outgrow the biohydrogen specialists. As a result, more substrate is converted to methane and other products at the expense of hydrogen. This can be minimized by pretreatment of culture to remove the competitors in a process called culture enrichment which can be affected by either heat [28] or chemical pretreatment [29]. The AOPs can also be used to enrich the culture of biohydrogen fermentation.

2.2 Bioreactor choice

Biomass washout is a big problem in biohydrogen fermentation for continuous processes with liquid or semi-liquid substrates. The type of the reactor used for biohydrogen fermentation should promote retention of microbes thereby reducing biomass washout. The use of a stirred tank reactor is therefore not optimal because of their short solid retention time which results in a biomass washout [30]. Application of reactors like upflow anaerobic sludge blanket (UASB), fluidized bed, or fixed bed reactors is most preferable in this regard. The immobilization of biomass in a fluidized bed can also help in reducing the biomass washout [31].

2.3 Optimization of process parameters

To maximize on biohydrogen recovery, the operation parameters in the bioreactor should be optimized. One of the most crucial parameters for optimization in biohydrogen fermentation is the operating pH. Most biohydrogen specialists have optimal pH values between 5.5 and 6.0 [32]. At higher pH values, the process is dominated by methanogens which work to reduce biohydrogen yields. Other parameters include the temperature [32], organic loading [33], and hydraulic retention time [33]. Operating at high temperature (> 40^{o}C) increases process kinetics and results in an increment of biohydrogen yields. However, the cost of operating at elevated temperatures adds to the process costs.

Operating the process with high organic loads generally increases hydrogen yields over methane [34]. There is however a limitation of low substrate conversion for operating at very high organic loads. The short hydraulic retention time (< 1 day) favors biohydrogen production over biomethanation. However, the use of very short retention time results in a reduction of substrate conversion [35]. This can only make economic sense when biohydrogen production process is followed by a subsequent process of biomethanation to maximize energy production.

2.4 Metabolic engineering

The main limitation of biohydrogen fermentation is low substrate conversion (< 20%) and poor yields. The process is limited in that a normal glucose molecule having twelve hydrogen atoms will have a maximum of only four atoms that can be converted to hydrogen fuel through fermentation [36].

One of the most recent strategies under investigation for improving biohydrogen yield entails biotechnological studies aimed at converting more hydrogen atoms from substrate molecules to hydrogen fuel. This entails genetic engineering where the microbes have their genetic composition or metabolic pathways modified [36]. Some of the enzyme modifications under investigations aimed at improving biohydrogen yields include studies on utilization of different metabolic pathways [37].

2.5 Use of nano-particles

Documented studies indicate that nano-particles do increase the biohydrogen yield of dark fermentation processes [38]. The most investigated nanoparticles in this regard are inorganic particles including Fe, Ni, TiO_2, and FeO [2]. However, there are reports of the use of organic nanoparticles in enhancement of biohydrogen yield [3]. The ability of nanoparticles to increase the electron transfer efficiency at the hydrogenase enzyme function site makes them effective in increasing biohydrogen yield. A study on biohydrogen production from bagasse hydrolysate with nanoparticles added to inoculum observed accumulation of biohydrogen specialists on inoculum immobilized on magnetite and iron nanoparticles which either increased hydrogen yields by more than 60% [39]. The mechanism of how the nanoparticles enhance biohydrogen fermentation has been documented [3].

2.6 Uses of AOPS

Advanced oxidation processes entail in situ generation of radicals which are reacted with the substrates through oxidization. The most common oxidant intermediate species produced in these processes is hydroxyl radical which is the most powerful oxidizing agent. Other notable oxidants include ozone, sulfate ion, manganate ion, hydrogen peroxide, chlorine, and perchlomate ion. Some of the advanced oxidation processes commonly applied in bioenergy substrate treatment include ozonation, Fenton processes, ultraviolet, electrochemical, microwave enhanced AOPs, wet air oxidation, and hydrogen peroxide oxidation [40].

3 Advanced oxidation processes (AOPs)

3.1 Ozonation

Ozone is a state of oxygen where its molecules occur in 3 atoms (O_3) formation. It has second highest oxidation potential (+ 2.07 V) after hydroxyl radical. This makes it possess high reactivity. The gas has high selectivity for unstable bonds like olefins as illustrated in Eq. 2.

$$CH_2 = CH_2 + O_3 \rightarrow H_2CO + H_2COO^* \tag{2}$$

The process mechanism may entail either electrophilic addition which results in the formation of substrate radical or insertion process that results in prolongation of the substrate chain. In the presence of little hydrogen peroxide, the peroxone reaction results in the formation of radicals like hydroxyl radical which has the highest oxidation potential (+ 2.80 V) among all oxidants. Therefore, the ozonation process is enhanced by the addition of a small amount of hydrogen peroxide and water at alkaline conditions.

One of the applications of ozone in the bioenergy sector is in pretreatment of excess sludge and anaerobic sludge for biohydrogen fermentation [41]. The ability of the process to increase the biodegradability of recalcitrant without much elimination of chemical oxygen demand (COD) makes the process appropriate for application as a substrate pretreatment for energy production. The excess sludge from the dark and light fermentation is collected and ozonated before biomethanation for biogas production. The process results in size reduction, increase in biodegradability, and solubilization of particulate organics as demonstrated in Fig. 1. This results in higher substrate conversion, better bioenergy yields, and improved biohydrogen productivity. The little COD loss which occurs due to mineralization has little effects in reducing the energy yields.

Ozone can also be applied in the pretreatment of biohydrogen substrate like palm olive mill wastewater (POMW) to increase their biodegradability [42]. The presence of a high amount of polyphenols in POMW makes it completely non-biodegradable despite its high COD load. Moreover, it can be applied in the pretreatment of leachate from landfills for biodegradability increase [43]. This effluent also contains a high amount of phenolics which are toxic to anaerobic digestion. As a pretreatment, ozone has good potential in the removal of inhibitors to the bio-digestion of toxicants like phenolics [44].

3.2 Ultrasonication or sonolysis method

This is among the cavitation methods where microbubbles/cavities are created and crashed in a very short time. The process results in the release of high energy which is used to create hydroxyl radicals from water as shown in Eq. 3.

$$2H_2O + O_2 \xrightarrow{\text{Ultrasound energy}} 2OH^- + 2OH^* \tag{3}$$

The radicals formed react with the substrate by mineralizing it or creating other substrate radicals as illustrated in Eq. 4.

$$OH^* + RH \rightarrow R^* + H_2O \tag{4}$$

Sonication has one advantage over other methods in that no chemicals are added to the process. Some of the applications of sonication in biohydrogen production include treatment of solid waste [45] and waste sludge [46]. The ability of the process to solubilize organic particles without reducing the organic load makes it good pretreatment for biosolids and solid wastes before anaerobic digestion [47].

3.3 Wet air oxidation

Wet air oxidation (WAO) is used to pre-treat refractory substrates by thermochemical treatment. The process takes place at high oxygen pressures and high temperatures (300 $^\circ$C). The extreme condition enables the solubilization of substrate and enhancement of biodegradability. The presence of molecular oxygen dissolved in the aqueous phase helps in the formation of radicals that react and mineralize the substrate. The formation of substrate radicals by wet air oxidation is illustrated in Eq. 5.

$$RH + O_2 \rightarrow R^* + HO_2{}^* \tag{5}$$

The wet air oxidation pretreatment process results in high content of volatile compounds like acetic acid [48]. This makes the process advantageous especially for hydrogen photo-fermentation where the microbes utilize the volatile compounds.

3.4 Fenton oxidation

One of the methods for the treatment of recalcitrant or toxicants in organic effluent is by Fenton treatment [49]. The process utilizes the hydrogen peroxide and ferrous ions as reactants which generate hydrogen radicals as shown in Eq. 6.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^* \tag{6}$$

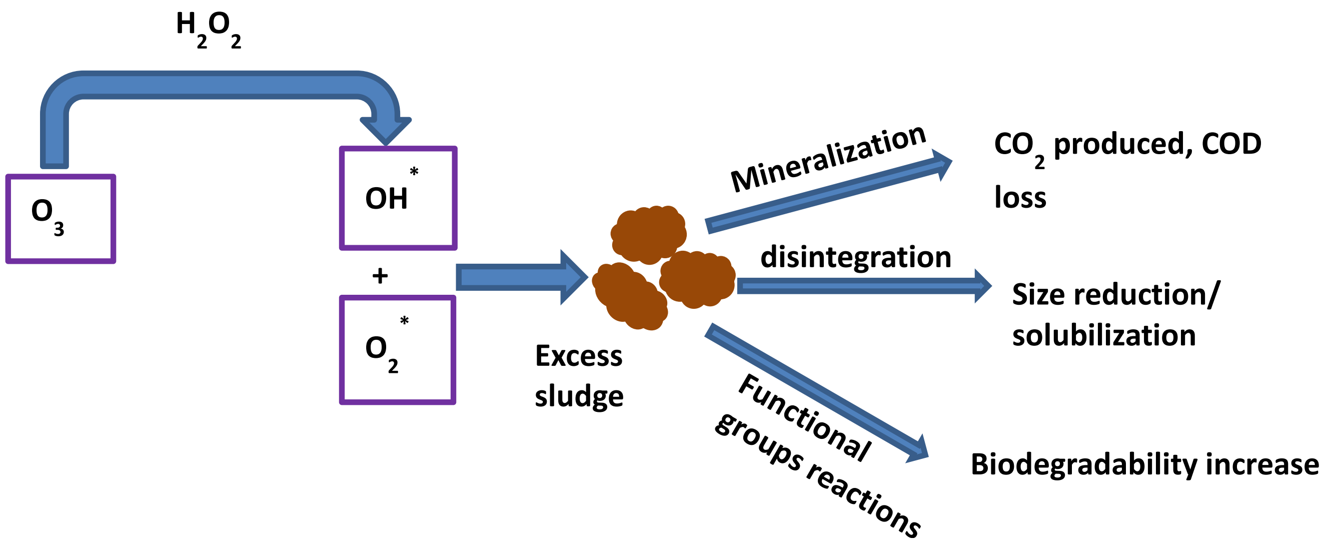

Fig. 1 Schematic diagram of collection and pretreatment of excess sludge by ozonation, 1 (ii), Demonstration of the effects on the excess sludge after ozone pretreatment, 1 (ii)

In classical Fenton, the process takes place at low pH values (pH 3), which makes it expensive for applications where the substrate has low pH sensitivity. However, modifications like the use of photo-Fenton and heterogeneous Fenton processes are aimed at operating at higher pH values. In addition to the final treatment of effluent after bioenergy production for COD and color removal [50], the Fenton process has been reported to improve the biodegradability of leachate substrate for bioenergy production [51]. A combination of Fenton and other processes like biological or physical-chemical processes has been reported to be very effective in the treatment of recalcitrant in complex effluents [52]. The Fenton process like all oxidation processes can mineralize phenolic toxicants into simpler compounds thereby increasing its biodegradability. Figure 2 shows how hydroxyl radicals produced in Fenton oxidation mineralize the phenolic substrates.

3.5 Electrochemical process

The principle of electrode and electrolyte in electrolysis is used to treat the substrate for bioenergy production in the electrochemical oxidation process. The electrode on which oxidation occurs is called the anode. The reduction takes place in the negative electrode, the cathode. Traditional electrochemical oxidation was used in the treatment of recalcitrant effluent [54]. Many effluents from bioenergy production have refractory compounds like melanoidins found in molasses distillery effluents, polyphenols in olive mill wastewater, and winery effluent. In the application of electrochemical oxidation for the treatment of bioenergy effluent, the organic particles are mineralized by alkali bacteria to produce carbon dioxide. Volatile acids are produced as by products which dissociate to release hydrogen ions. The summary of the reactions is shown in Eq. 7. At the cathode, hydrogen gas is liberated by the reduction of hydrogen ions as shown in Eq. 8.

$$R \rightarrow CO_2 + H^+ + \bar{e} \tag{7}$$

$$2H^+ + 2\bar{e} \rightarrow H_2 \tag{8}$$

In addition to the treatment of bioenergy effluent for COD and color, the electrochemical process can be used to produce bioenergy [55]. The process has in recent past attracted great interest because of its potential to produce biohydrogen plus other forms of bioenergy from biomass [56]. The organic matter is mineralized on the anode while the hydrogen ions produced at the cathode as shown in Fig. 3. The proton exchange membrane enables the hydrogen ions produced at the anode to move to the cathode.

3.6 Photocatalysis

The photons on the surface of some semiconductors like titanium dioxide, zinc oxide, and zinc sulfide can be excited by reaction with certain chemicals in solution to produce free radicals. The process is usually induced by irradiation of the surface with an energy source like ultra-violet radiation. The photocatalytic reaction starts with the generation of the electrons and electron holes at the surface of the metal (M) which are then used to produce radicals as shown in Eqs. 9, 10, 11, and 12 [57].

$$M + energy(hv) \rightarrow M(h^+) + \bar{e} \tag{9}$$

$$M(h^+) + H_2O \rightarrow M + OH^* + H^+ \tag{10}$$

$$M(h^+) + OH \rightarrow OH^* \tag{11}$$

$$O_2 + \bar{e} \rightarrow O_2^{*-} \tag{12}$$

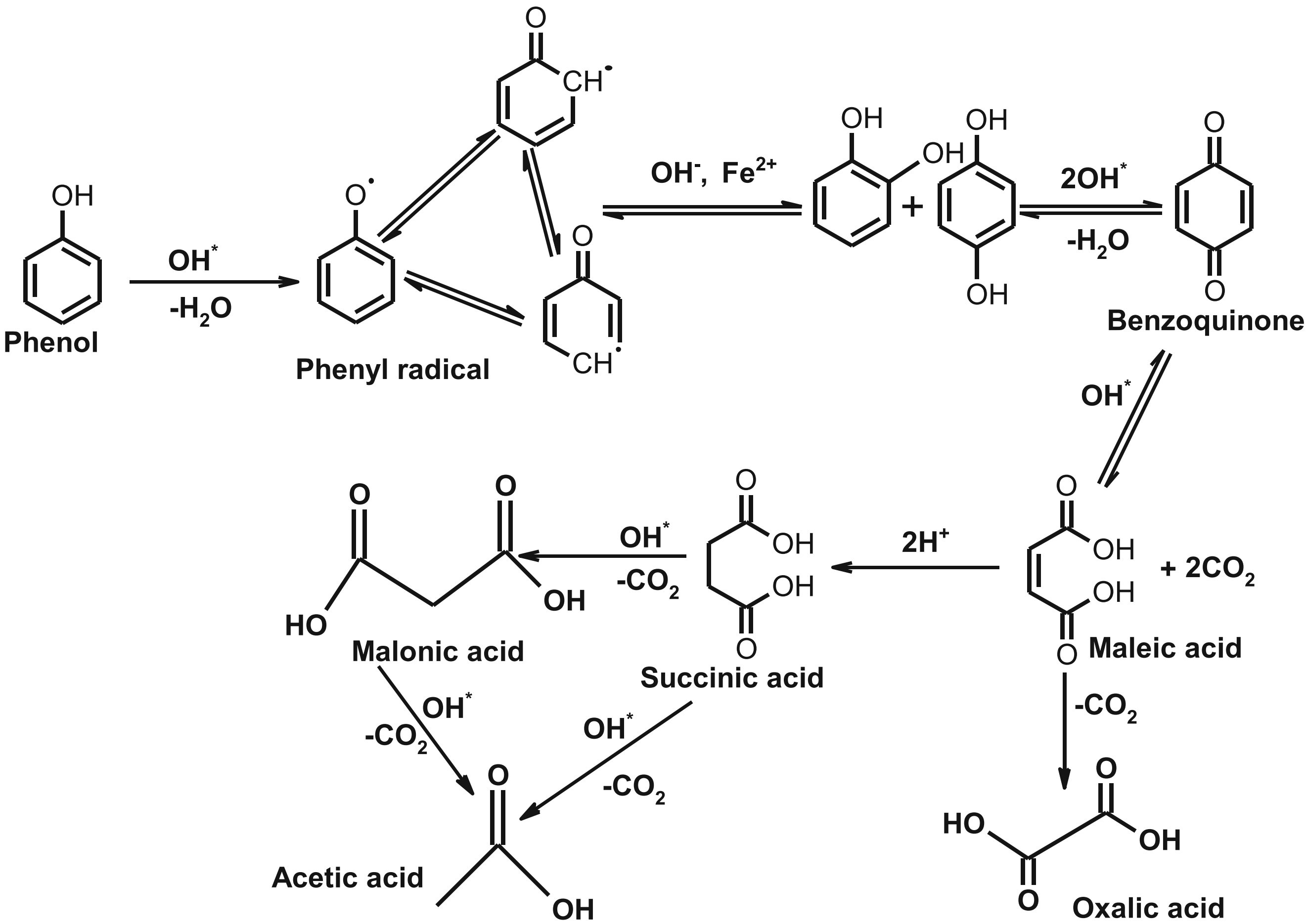

Fig. 2 Pathways for phenol mineralization by Fenton oxidation as modified from Bremer et al 2006 [53]

One of the most promising applications of photocatalysis is the treatment of bioenergy effluent for COD and color removal [58]. However, a more unique application entails upgrading of low-value bio-products to higher value biofuels. Through photocatalytic processes, hydrogen can be produced from acetate which is one of the main byproducts of dark hydrogen fermentation [59]. This implies that coupling of dark hydrogen fermentation to photocatalytic oxidation can increase biohydrogen yields. Moreover, the photocatalytic process can be used to produce other valuable products like ketones and aldehydes from volatile acids [60].

4 Application of AOPs in biohydrogen fermentation

4.1 Use of AOPs in pretreatment of organic substrates for biohydrogen fermentation

The lignocellulosic biomass is the most abundant organic matter on earth surface. This makes it potentially the cheapest substrate for biohydrogen fermentation. However, the low conversion of lignocellulosic substrates and poor productivity remain the main bottleneck to biohydrogen fermentation. Pretreatment of substrate is among the most investigated strategies for improvement of biohydrogen substrate conversion and energy yields for both lignocellulosic biomass [61] and liquid organic substrates [62]. Various substrate pretreatment methods for biohydrogen production have been reviewed where sonication and microwave-enhanced AOPs were found to be among the most applied methods [63]. There are also new methods like solar photocatalysis which have been reported for pretreatment of biohydrogen substrates [64]. The ultrasonic treatment was among the methods that resulted in enhancement of bioethanol and biohydrogen production by hydrolyzing lignin [65].

4.1.1 Mechanisms of AOPs substrate pretreatment

The main mechanism for enhancement of energy yield from organic substrates by treatment with AOPs is through breakdown of complex substrate structure which promotes its hydrolysis to produce fermentable sugars and accumulate volatile acids [66]. In addition to substrate disintegration, the AOPs help in solubilization of the substrates [67]. A study with marine algae biomass observed more than 27% increase

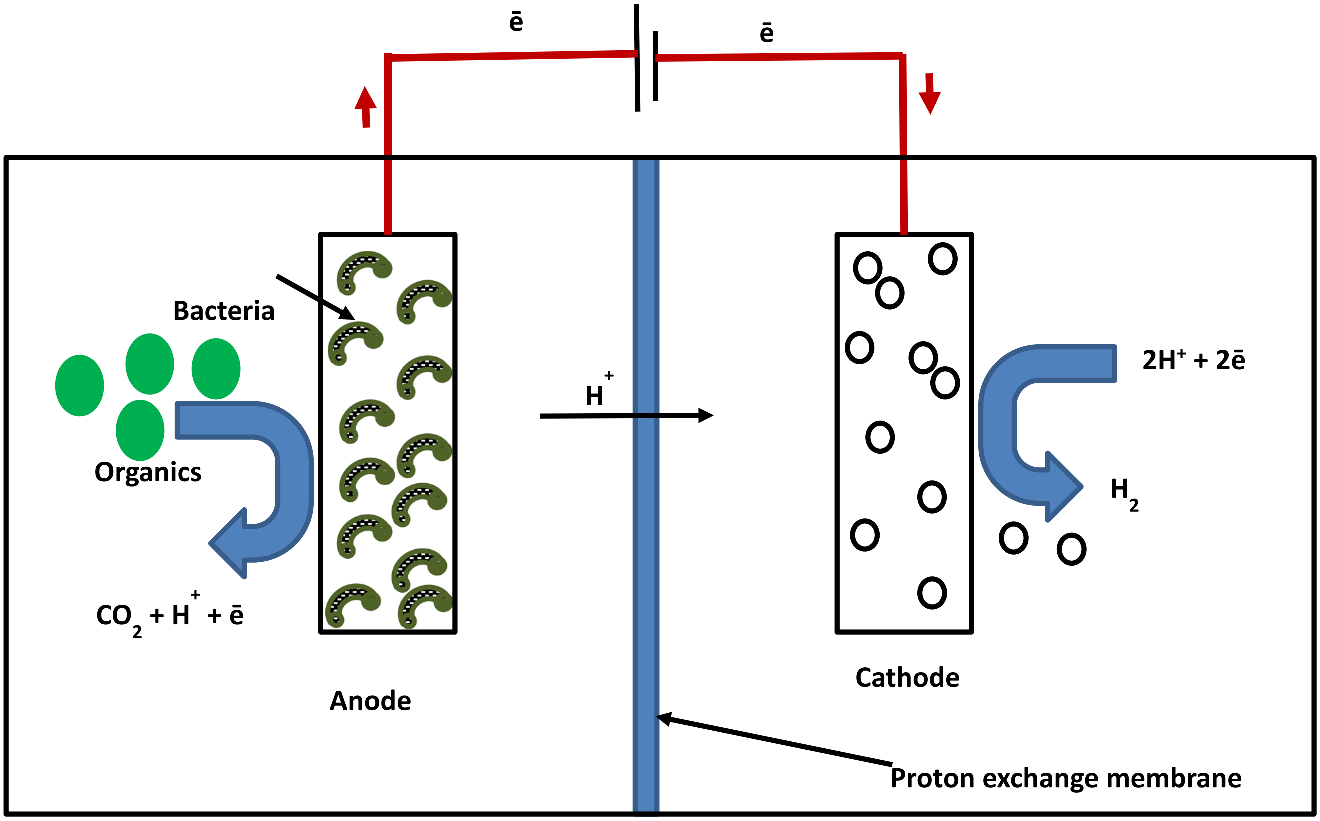

Fig. 3 Electrochemical hydrogen production from organic substrates

in solubilization after treatment with hydrogen peroxide, microwave, and acid [67]. There are also reports of increased biochemical acidogenic potential of substrate after AOP treatment [68]. In treatment of sludge substrates, the disintegration of huge particles to smaller ones is desired to improve the biodegradability of the substrate. The treatment with AOPs can help disintegrate large particles to smaller ones and therefore enhance biodegradability [69]. Moreover, AOPs can be used to selectively eliminate the inhibitors of fermentation process by mineralizing them or reacting with their functional groups. This helps increase substrate biodegradability and biohydrogen yields.

Figure 4(i) demonstrates the application of AOPs pretreatment of lignocellulosic substrates to enhance biohydrogen fermentation compared to the process without pretreatment (Fig. 4(ii)). The high solubilization and saccharification achieved after the pretreatment enable high recovery of biohydrogen gas.

The pretreatment of complex biohydrogen substrates by AOPs entails breakdown of selected bonds to produce simpler compounds that are more biodegradable. In cellulosic biomass, the breakdown or mineralization of the lignin and hemicellulose makes the cellulose substrates available for further reactions. The solubilization of the substrates by AOPs is caused by hydrolysis of the substrates to simpler compounds. Figure 5 below is an illustration of how hydroxyl radicals can hydrolyze cellulose substrate to simple sugars by breaking the β(1-4) glucosidic linkages. Assuming the first radical reacts with the substrates at the position shown in (a), a monosaccharide β-glucose is cut-off from the chain. If this reaction is followed by another attack at (b), a disaccharide sugar, β(1-4) cellubiose is cut off from the main chains. Each of the reactions for every hydroxyl radical attack follows Eq. 13, where a neutral compound and another radical are produced.

$$R_1\text{-}R_2 + {}^*OH \rightarrow R_1OH + R_2^* \tag{13}$$

The chain is propagated according to Eq. 14 to produce different substrates. If the substrate radical reacts with water molecule, R_3 is OH and therefore hydroxyl radical is generated.

$$R_2^* + R_3H \rightarrow R_2H + R_3^* \tag{14}$$

4.1.2 Enhancement of biohydrogen yields after AOPs pretreatment

The low yields of biohydrogen from fermentation of biomass substrate can be improved by pretreatment. There are investigations showing that the pretreatment of substrates by the use of AOPs can improve biohydrogen yields. The removal of

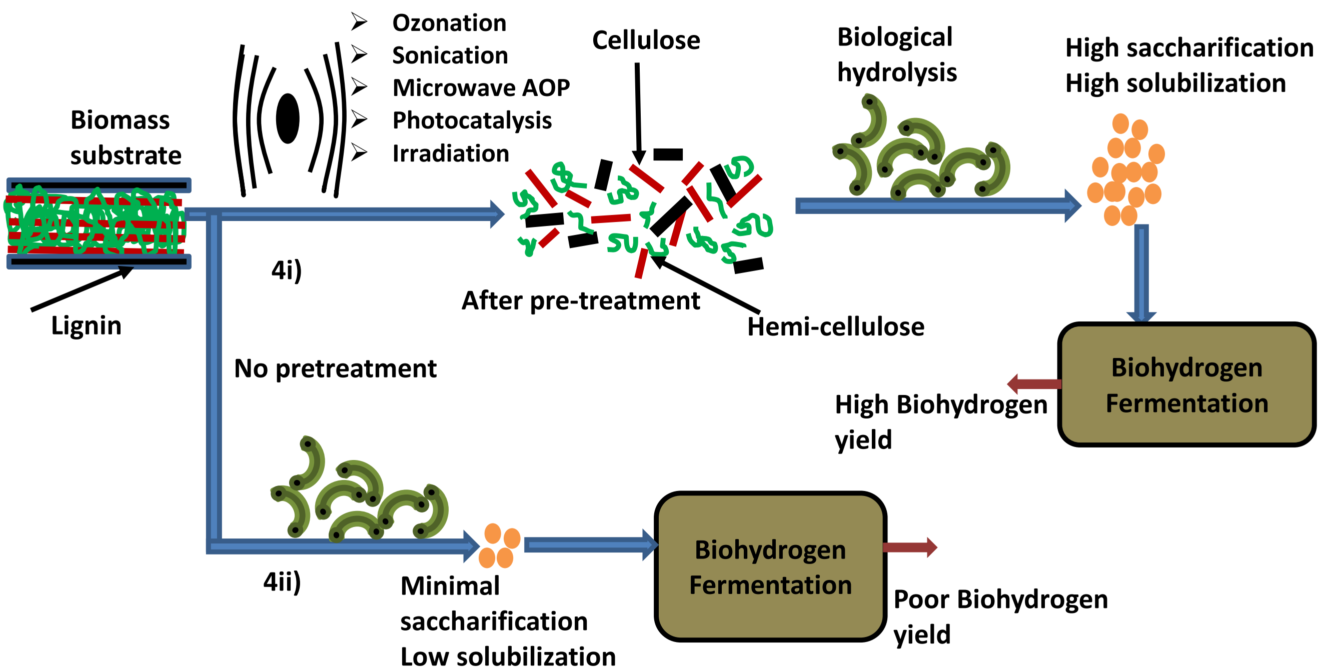

Fig. 4 Enhancement of biohydrogen yield from lignocellulosic biomass through pretreatment with AOPs (4i) and the control without pretreatment (4ii)

ammonia ions from by-products of dark hydrogen fermentation, which are one of the inhibitors of anaerobic digestion process was achieved by pretreatment with nano-TiO_2 [70]. This produced more than 45% increase in hydrogen yield in subsequent photofermentation [70]. The pretreatment of grass with combined ultrasound and acid resulted to more than 100% and 300% increase in solubilization and biohydrogen yields respectively [71]. A summary of documented studies on application of AOPs in pretreatment of biomass substrates is given in Table 1.

The results indicate that AOPs have promising application in enhancement of biohydrogen yields from various substrates. However, the suitability of the applying AOPs in substrate pretreatment is determined by its effectiveness in yields enhancement and associated process costs. There are few documentations detailing the cost-effectiveness of using AOPs pretreatments for biomass substrates. A combination of AOPs and other methods especially heat and chemical treatment can significantly reduce the processes costs. There are reports of high biohydrogen from lignocellulosic biomass after pretreatment by combination of AOPs and acid [71].

4.2 Enrichment of inoculum for biohydrogen fermentation by pretreatment with AOPs

The enhancement of biohydrogen production through treatment with AOPs may entail either elimination of microbial competitors to biohydrogen specialists or production of charged particles which promote the flow of electrons. The two theories are explained below.

(a) Elimination of microbial competitors

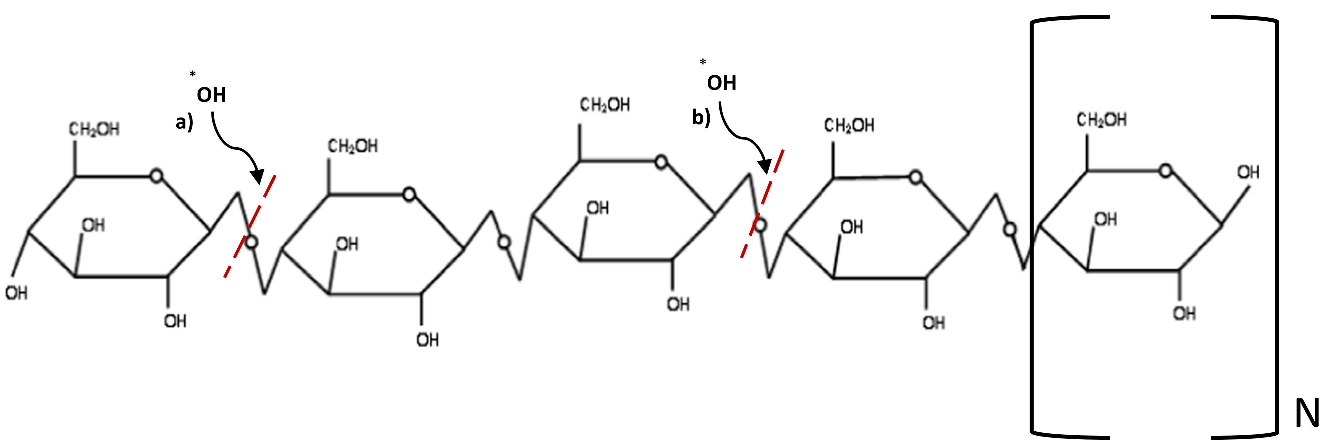

Fig. 5 Solubilization of cellulose after attack by hydroxy radicals

Table 1 Application of AOPs in pretreatment of biomass substrates for biohydrogen production

S. No	Substrate	AOP	Effects on H_2 production	Reference
1	Palm olive mill effluent (POME)	Ozone	60% increase in yield	[72]
2	POME	Ozone	50% increase in H_2 yield	[42]
3	Biomethanated DWW	Ozone	More than twofold increase in H_2 yield	[73]
4	POME	Ozone	4% increase in H_2 yield	[74]
5	Glucose	Temp. controlled sonication	45% increase in H_2 yield	[75]
6	Food wastes	Sonication H_2 reactor	62% H_2 increase in H_2 yield	[76]
7	Food wastes	sonication	75% increase in H_2 yield	[77]
8	Distillery WW	Sonication	96% increase in H_2 yield	[78]
9	Complex dairy WW	Sonication	27% increase in H_2 yield	[79]
10	Palm oil, pulp and paper mills effluent	Sonication	87% increase in H_2 yield	[80]
11	Waste sludge	Photocatalysis	17-fold increase in H_2 yield	[81]
12	Waste-activated sludge protein	Photocatalysis	1.2-fold increase in H_2 yield	[82]
13	Glucose	Photoelectrocatalytic oxidation	30% increase in H_2 yield	[83]
14	Waste-activated sludge	Photocatalysis	220% increase in H_2 yield	[84]
15	Poultry slaughterhouse sludge	Microwave-enhanced	70-fold increase in H_2 yield	[85]
16	Cornstalk	Microwave assisted alkali pretreatment	55% increase in H_2	[86]
17	Corn	Microwave assisted acid pretreatment	Increase in H_2 yield	[87]
18	Wheat straw	Ozonation	100–150% increase in H_2 yield	[88]
19	Sewage sludge	Sonication-ozonation	35% increase in biodegradability index	[89]
20	Food waste	Sonication	145% increase in H_2 production rate	[76]
21	POME	Ozonation	60% decrease in H_2 yield	[90]
22	Algae biomass	Microwave-acid method	190% increase in H_2 yield	[91]
23	Pulp and paper	Microwave-alkaline	26% decrease in H_2 yield	[92]
24	Waste sludge	Sonication	> 3-fold increase in short chain fatty acids	[93]
25	Grass waste	Ionization radiation	30% increase in H_2 yield	[94]

The low yields in biohydrogen fermentation are mainly due to competition for substrates by biohydrogen specialists and other microbes like surfate-reducing bacteria, methanogens, and acidogens bacteria. Equations 15, 16, and 17 demonstrate the scavenging effect of hydrogen by these microbes:

Methanogens

$$4H_2 + CO_2 \rightarrow_{\text{Methanogens}} CH_4 + 2H_2O \quad \Delta G^\circ{-}165KJ \tag{15}$$

$$5H_2 + SO_4^{2-} \rightarrow_{\text{Sulfate-reducing microbes}} H_2S + 4H_2O \, \Delta G^\circ + 30KJ \tag{16}$$

$$4H_2 + 2CO_2 \rightarrow_{\text{Acidogens}} CH_3COOH + 2H_2O \, \Delta G^\circ{-}75.2KJ \tag{17}$$

One method of enhancing biohydrogen yields is by enriching hydrogen-producing specialists in the inoculum. This entails suppression of the competing microbes including methanogens [95]. Various methods have been reported to suppress methanogens mixed culture inoculum like pretreatment with waste frying oil [96]. Heat treatment is the most applied method of inoculum pretreatment to enhance biohydrogen production [97]. The other AOP methods that have been investigated in biohydrogen inoculum enrichment include electric shock, ionization irradiation, and ultraviolet irradiation [98, 99]. Figure 6 illustrates the elimination of the competitors by AOPs enables higher energy recovery.

b Charged organic particles

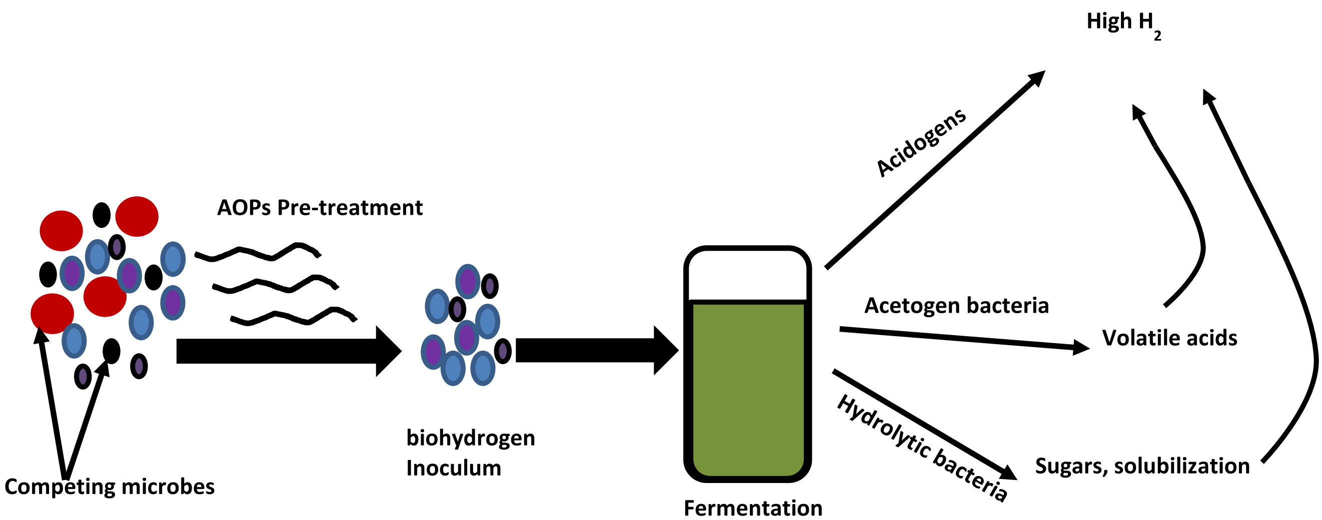

Fig. 6 Illustration of how elimination of competitors by AOP pretreatment increases biohydrogen yields

The process of dark hydrogen fermentation employs mixed acid metabolic pathway. The end product of the process depends on the metabolic pathway followed as shown in Fig. 7. The acetate pathway is the most preferable because it produces four molecules of hydrogen from one hexose sugar. The butyrate pathway produces two moles of hydrogen per mole hexose while no hydrogen is produced in ethanol and lactate pathways. The dark fermentation reaction pathways are limited in that when the redox reaction by pyruvate ferredoxin oxidoreductase is less than the rate of pyruvate formation, more substrate is converted to lactate and therefore less hydrogen is produced. In addition to breakdown and solubilization of the substrates, the AOPs oxidize part of substrates to produce charged particles that act as a conduit for electrons in conversion of pyruvate to acetyl-CoA as shown in Fig. 7. The charged particles ensure that the pyruvate is not converted to lactate by providing a fast pathway for electron flow to hydrogenase enzyme. Similar effects have been reported of increasing biohydrogen production by the use of organic and inorganic nanoparticles that boasted the electrons flow [2, 3].

There are reports suggesting that up to 10% of biohydrogen inoculum enrichment is done using microwave irradiation treatment with the bulk of application choosing heat treatment [100, 101]. The low effectiveness of using ultraviolet and sonication compared to heat pretreatment of microflora for biohydrogen has been reported [102]. However, other results indicated higher performance in chemical pretreatment using 2-bromoethane sulphonic acid sodium salt than heat shock pretreatment at 100 °C [103]. More comparative studies of the effectiveness of different methods of inoculum pretreatments are required especially with processes like microwave-enhanced AOPs to establish the effectiveness of the same. In addition, there is need to investigate the effect of combining different methods. For instance, a combination of ultrasonication and alkaline treatment of sludge substrate produced highest hydrogen yields compared to the two individual processes and heat treatment [23]. A correlation between acetic acid production during fermentation process and hydrogen yield after enrichment of the inoculum with AOPs has been reported [104]. Table 2 gives a summary of application of AOPs in pretreatment of inoculum for biohydrogen production. The studies clearly indicate that the pretreatment of biohydrogen inoculum using AOPs is effective in enhancing the yields. Some of the methods which have been tried in this regard include microwave heating, sonication, electrochemical, and gamma radiation.

4.3 Application of AOPs in treatment of the effluent from bioenergy fermentation processes

Most bioenergy processes including hydrogen fermentation release effluents that require remediation before they are discharged to the receiving bodies. The quality of the effluent is dependent on the bioenergy substrate used and the production processes. The processes utilizing molasses-related substrates release very dark colored effluent containing high remnant COD [119]. The recalcitrant in the effluent include colorants like phenolics, melanoidins, and caramel compounds [120]. The processes utilizing winery distillery effluent have high polyphenols, dark color, and high nutrients [121, 122]. There is high concentration of polyphenols and COD in the effluent from processes utilizing olive mill-related substrates [123]. Also, effluents from dairy industries or industries dealing with dairy-related products like cheese contain high presence of nutrients especially total nitrogen and fats which result to high COD [124]. The current stringent environmental regulations worldwide require that these pollutants are eliminated before the effluents are discharged to the receiving bodies.

The use of conventional treatment methods for remediation of bioenergy effluents include physical-chemical processes

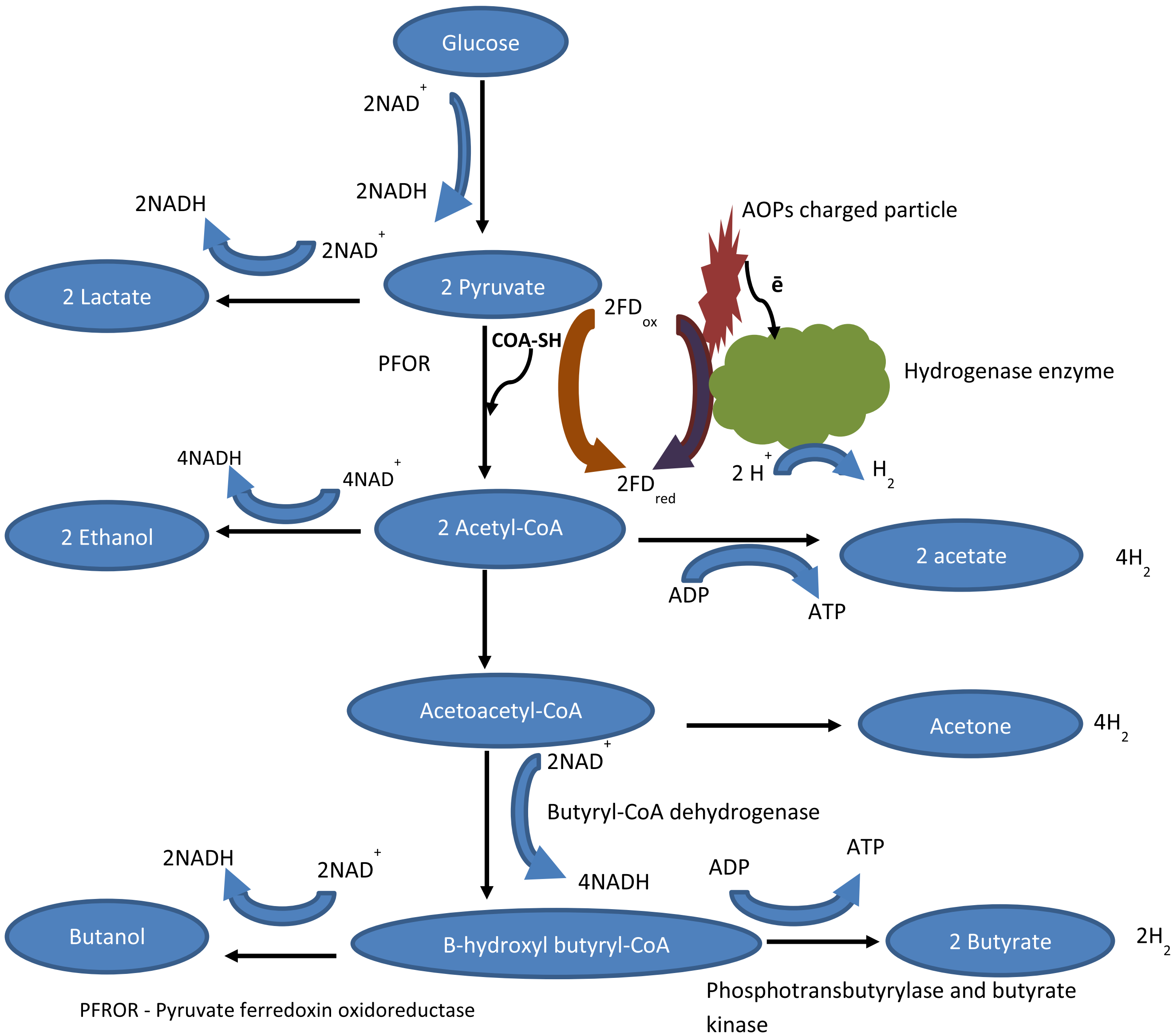

Fig. 7 Biohydrogen dark fermentation process and mechanism of AOPs pretreatment of biohydrogen inoculum

like coagulation [125], adsorption [126], membrane filtration [127], and biological processes like aerobic digestion [128], anaerobic digestion [129, 130], and membrane separation [131]. The methods are only effective when used as primary treatment to remove the bulk of COD. However, they are limited in removing the recalcitrant in the effluents which is essential to produce polished effluent that meets the required standards for discharge to receiving bodies. Advanced oxidation processes through the radicals which they produce are able to mineralize the recalcitrant in the effluent to achieve the final polishing treatment. Some of the AOP processes that are commonly employed to remediate these effluents include Fenton [132], ozonation [133], ultrasonication [134], electrochemical oxidation [135], photocatalysis [136], and wet air oxidation [137]. The application of photocatalytic AOPs in remediation of colored effluent from biohydrogen fermentation is illustrated in Fig. 8. The electrons and holes produced by illuminating photo-catalytic surfaces with light energy are used to produce radicals from oxygen, water, or hydroxide by photo-reduction or oxidation. The radicals produced react with unstable bonds like aromatic linkages in some colorant substances. The reaction results in decolorization of the substrate through breakages of these bonds, formation of other linkages, or mineralization of the substrates.

In addition to reducing the COD, the AOPs have the ability to increase the biodegradability of the bioenergy effluent [138, 139]. This makes them appropriate for application as an intermediate step before biological treatment is done on recalcitrant effluent. The effect of applying various AOPs in treatment of bioenergy effluent is summarized in Table 3. Some of the AOPs applied to treat bioenergy effluent include ozonation, Fenton, photocatalysis, and electrochemical oxidation.

Table 2 Pretreatment with AOPs of innoculum for biohydrogen fermentation

S.no	Inoculums	Substrate	AOP	Effect	Ref
1	Cow dung inoculum	Mixed culture waste	Microwave-enhanced AOP	10-fold increase in H_2 yield	[105]
2	Cow dung compost	Corn	Microwave-enhanced AOP	85% increase in H_2 productivity	[106]
3	Sewage sludge	Glucose	Gamma irradiation	200% increase in H_2 productivity	[107]
4	Primary anaerobic digester sludge	Waste-activated sludge	Gamma irradiation	200% increase in H_2 productivity	[108]
5	Waste-activated sludge	Pretreated sludge	Sonication	18% increase in H_2 yield	[109]
6	Sludge	Organic wastewater	Electrochemical	fivefold increase in H_2 productivity	[110]
7	Wastewater treatment plant sludge	Glucose	Electrochemical	130% increase in H_2 yield	[111]
8	*Rhodopseudomonas palustris*	Glucose	Ultrasonic treatment	twofold increase in energy conversion	[112]
9	Activated sludge	Apple pomace	UV and Sonication	80% increase in hydrogen yield	[113]
10	Mixed anaerobic culture	Sludge	Sonication	2.5-fold increase in power density	[114]
11	Mixed microflora	Corn stover hydrolysate	Ultrasonication	8% increase in hydrogen yield	[102]
12	Cow dung microflora	Kitchen waste	Sonication	twofold increase in hydrogen yield on 40-min exposure	[115]
13	Domestic biogas fermenter sludge	Soluble starch	Microwave-enhanced AOP	15-fold curing time for elimination of methanogens compered to heat	[116]
14	Anaerobic sludge	Citrus wastewater	Electroporation	64% increase in hydrogen yield over heat treatment	[117]
15	Anaerobic sludge	Citrus wastewater	Sonication	24% increase in hydrogen yield over heat treatment	[117]
16	Anaerobic digested sludge	Rice and lettuce	Sonication	3% increase in hydrogen yield compared to raw	[118]

The results indicate that AOPs are effective in decolorization of bioenergy effluent with reports of more than 90% removal.

4.4 Integration of AOPs with other processes to enhance performance

The technical and cost-effectiveness of oxidation processes can be enhanced by applying them in integration with other treatment methods. Various groups have investigated the use of AOPs in combination with conventional treatment methods including the physical-chemical processes like biological [163] and coagulation [164]. It is also possible to combine more than one oxidation method to enhance process performance [165]. The hydrolysis of lignocellulosic rice straw substrate for biogas production with the Fenton process increased the enzymatic saccharification 1.5-fold. However, a combination of Fenton and ultrasound treatment improved the saccharification by fourfold [166]. Comparative studies involving AOPs and other methods are necessary to optimize biohydrogen production processes. A study which investigated a combination of heat, AOPs, and biological treatment of recalcitrant COD from distillery wastewater observed that ozone was more effective than sonolysis in the remediation [167].

The integration of wet air oxidation with a biological process in bioenergy effluent treatment has been reviewed [168]. The process is limited in that a biological process can take high volume effluents that would require a large WAO reactor which would increase the process costs. However, the problem can be solved by optimizing the flow-rate in the two reactors factoring in the short HRT for WAO and long HRT for biological processes. Similarly, a study coupling Fenton oxidation and biological processes found that maximum treatment capacity could be achieved by optimization of mineralization rate and hydraulic retention time [169]. Other than biological and physical-chemical processes, it is possible to integrate two or more AOPs for better results. An integration of wet oxidation with heterogeneous Fenton using Fe_2O_3 nanocomposite catalysts was able to increase the biodegradability (BOD_5/COD ratio) of industrial effluent from 0.2 to 0.3 [170]. The possibility of integrating sonolysis and photocatalysis for enhancement of biomass pretreatment has been reviewed [171]. Hence, there is a high potential for increasing the technical efficiency of the process by combining several AOP processes but more studies on the same are required especially on optimizing the combined operations.

The reason for integrating several operations is to increase the process's effectiveness in terms of the yield achieved and the process costs reduction. The suitability of different AOPs for various applications depends on the substrate. A simple method of establishing the most suitable oxidation process should entail comparing the output and the costs involved. Despite all the literature on applications of oxidation processes, there is limited documentation on the cost-effectiveness of

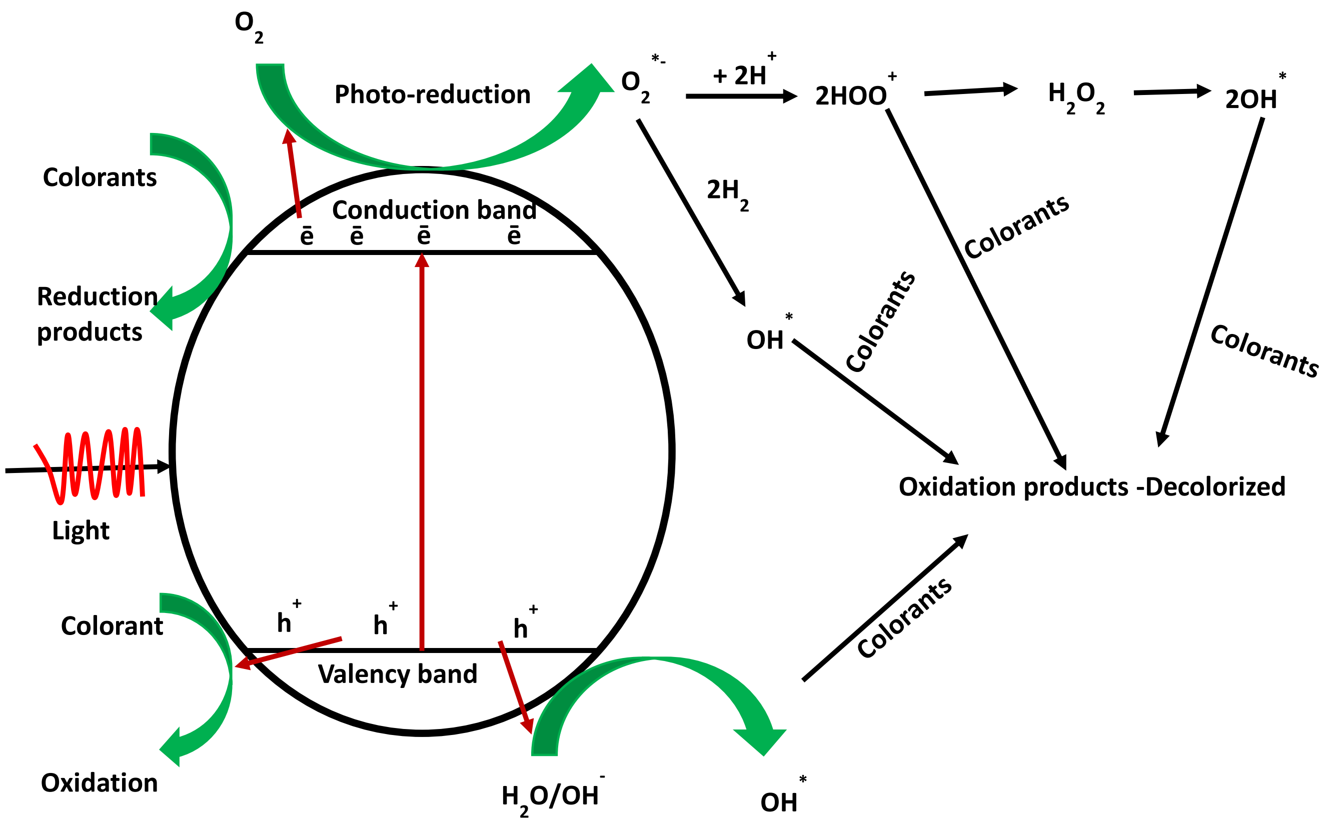

Fig. 8 Illustration on decolorization of colored effluent from biohydrogen fermentation process by photocatalysis

different AOPs. Among the documentations available is a study combining the Fenton and cavitation process that observed the process to be more efficient and cost-effective compared to the use of either process separately [172]. Another comparison study where biological treatment of leachate was coupled with either solar photo-Fenton or ozonation observed that the former was more cost-effective but the latter was better in the reduction of the substrate toxicity [173]. Also, a study on the removal of micro pollutants from municipal wastewater reported that solar photo-Fenton was more efficient and cost-effective compared to ozonation and photocatalysis [174].

The application of photocatalysis only was least effective in the removal of micro pollutants [174]. However, more studies are necessary to shed light on the cost-effectiveness of using AOPs on various stages of bioenergy production.

The integration of AOPs with physical-chemical and biological processes can help in maximizing on COD removal during final effluent treatment [144, 175]. Most AOPs are poor in bulk COD elimination compared to conventional processes like coagulation, filtration, adsorption, and biological digestion. The purpose of these processes, when applied in this integration, is to reduce the chemicals or energy required by using AOPs. This helps in the enhancement of cost-effectiveness for the entire process. The AOPs can selectively target the recalcitrant like refractory COD or colorants which cannot be eliminated by conventional methods. When integrated with biological methods, AOPs mineralize the recalcitrant COD to more biodegradable compounds which are subsequently eliminated through bio-digestion.

Integration of AOPs with conventional methods in the pretreatment of bioenergy substrate can help in enhancement of energy yields. Sonolysis is one of the methods used in bioenergy substrate pretreatment because the process has low COD elimination. However, the high energy requirement by the process can be reduced by integrating it with other AOPs like Fenton oxidation [176], ozonation [177], and microwave heating [178]. The integration of AOPs with thermochemical processes ensures that the treatment process is faster, more effective, and cheaper [179, 180]. The treatment by hydrogen peroxide or ultraviolet radiation helps to fasten other processes like ozonation and Fenton processes [181].

4.5 Biorefinery production concept and AOPs

The biorefinery concept in bioenergy production refers to the production of several biofuels and bioproducts from a common organic substrate feed to improve on the process of energy output and cost-effectiveness [182, 183]. The production of biodiesel and biogas from algae substrates was reported to yield higher energy than when single products were produced [184]. The possible products from the process include

Table 3 Application of AOPs in treatment of bioenergy effluent

S No.	Wastewater	AOP Process	Effects	Ref
1	OMW	Fenton oxidation	90% color removal	[140]
2	Distillery spent wash	Electro-Fenton and Fenton	66% and 79% color removal by Fenton and electrocoagulation respectively	[141]
3	Cellulose fermentation wastewaters	Continuous flow microbial fuel cell	76% COD removal	[142]
4	OMW	Combined UV/O_3 and biological	91% COD removal	[143]
5	Distillery effluent	Ozonation and electrocoagulation	83% COD removal	[144]
6	Acidogenic food waste effluent	Microbial electrochemical cell	59% COD removal	[145]
7	Municipal waste liquor	Microbial fuel cell	92% COD removal	[146]
8	Distillery wastewater	Nano-catalytic ozonation	60% COD removal	[147]
9	Vinasse	Electrochemical	50% COD removal	[148]
10	Leachate	Combined electrochemical and anaerobic	87% COD and 100% NH_3-N removal	[149]
11	Ricotta cheese whey	Combined electrochemical and dark fermentation	79% COD removal	[150]
12	Domestic wastewater	Microbial electrolysis cell	86% COD removal	[151]
13	OMW	Catalytic ozonation, ultrasound and H_2O_2	85% removal of COD	[152]
14	Crude glycerol from biodiesel industry	Microbial fuel cell	50% COD removal	[153]
15	Distillery effluent	Electrocoagulation	58% COD removal	[154]
16	Cellubiose fermentation wastewater	Microbial fuel cell	75% COD removal	[155]
17	OMW	Photocatalysis	92% COD removal	[156]
18	Biomethanated distillery effluent	Combined ozonation and anaerobic	98% colorants and 55% COD removal	[157]
19	Vinasse	Electrochemical	61% COD removal	[158]
20	Molasses wastewater	Ozonation	93% color removal	[159]
21	Dairy wastewater	Electrochemical	95%, 78%, and 99% removal of COD, proteins, and turbidity respectively	[160]
22	Distillery effluent	Electrochemical	62% COD and 98% color removal	[161]
23	OMW	Combined microwave and Fenton-like CU(II)H_2O_2	98% color and 82% phenolic removal	[162]

biopharma, yeast, bioethanol, biohydrogen, biogas, and bio-oil.

The AOPs can be applied to enhance biorefinery production, both as substrate pretreatment and as final treatment of bioenergy effluent and sludge. Some bioenergy effluents like distillery and wastewater though biodegradable, have highly recalcitrant COD (> 1.5 g/L) which is very dark in color due to melanoidins and related substances. These recalcitrant are resistant to biological digestion and remain in the treated effluent causing environmental pollution. The environmental regulating bodies require that the effluent must meet the set standards before it is discharged. The conventional method of bioremediation which entails the use of chemicals and membranes is not sufficient to remove the recalcitrant. This necessitates further treatment by application of alternative methods like AOPs. Advanced oxidation methods are used as the final treatment of the effluent to remove recalcitrant like colorants. Also, they can be applied in pretreatment of substrate and sludge before biological treatment. The pretreatment enhances generation of energy and removal of COD or color from the effluent by subsequent processes. Figure 9 demonstrates the integration of AOPs with other processes in a biorefinery set-up to produce various bioproducts and biofuels from molasses as a common substrate. Furthermore, it demonstrates the application of AOPs in pretreatment of sludge for biofuel production and final removal of recalcitrant in the effluent before discharge.

5 Electrochemical Hydrogen Production and Microbial Fuel Cells (MFC)

One of the recent technologies on bioenergy production entails the use of electrochemical processes in a setup known as microbial fuel cell [185]. These cells can, in addition to the generation of electricity, produce other biofuels like

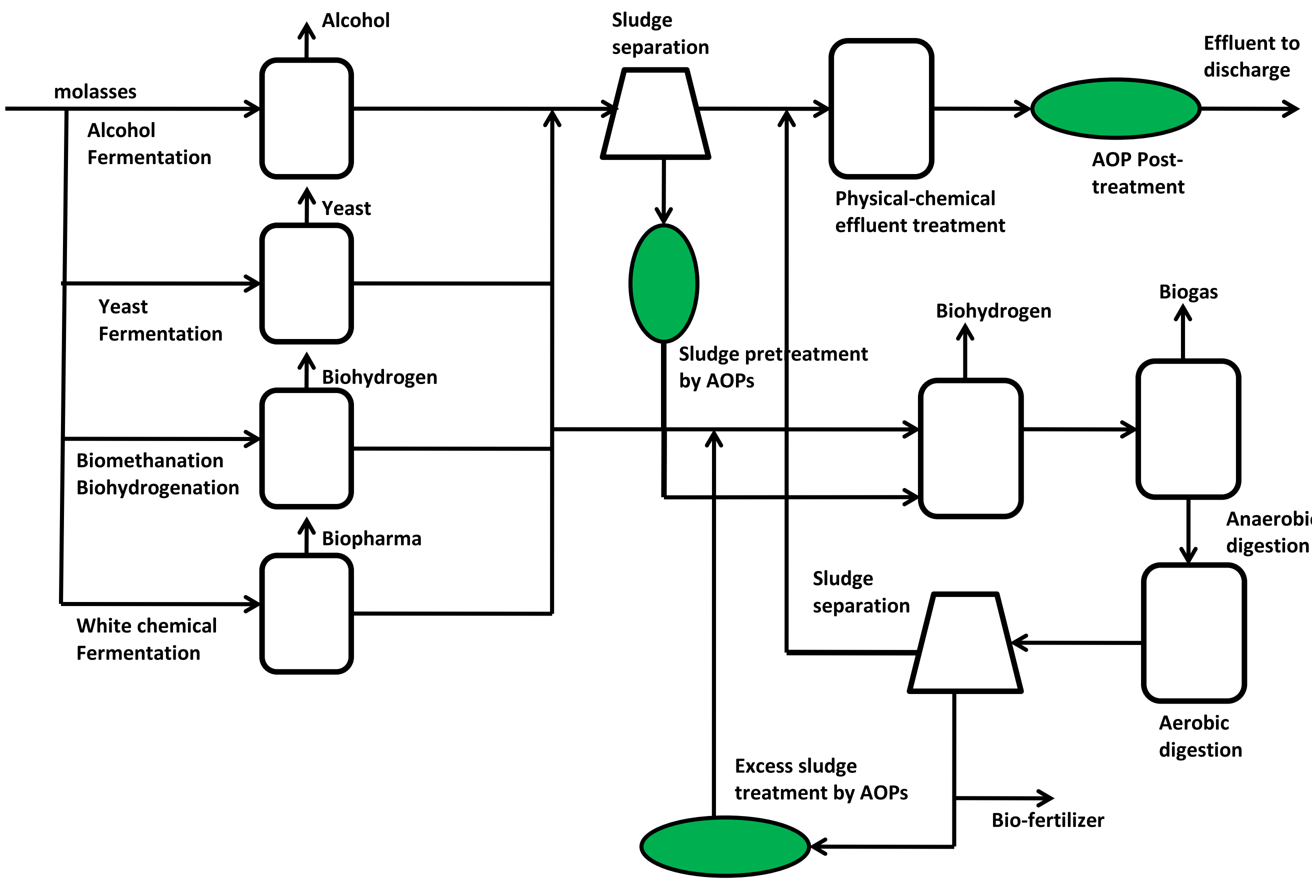

Fig. 9 Illustration of usage of AOP in biorefinery production using molasses as substrate to enhance the process technical and cost-effectiveness

biohydrogen and bioethanol through electrochemical oxidation [186, 187]. The fuel cell uses the principle of electrolyte, cathode, and anode to generate electrochemical energy. There are investigations on the usage of sludge as an electrolyte to produce electricity using microbial fuel cells [188]. The organic acid in the sludge acts as the electrolyte. There are documentations indicating that formation of electroactive biofilm in MFC is a possible mechanism for electron transfer [189]. The two reactions in both electrodes are demonstrated below by Eqs. 18 and 19 using acetate.

$$\text{Anode}: CH_3COO^- + 2H_2O \rightarrow 2CO_2 + 7H^+ + 8e- \quad (18)$$

$$\text{Cathode}: O_2 + 4e^- + 4H^+ \rightarrow 2H_2O \quad (19)$$

The two reactions are only possible when utilizing anaerobic anode chamber where oxygen is kept off from the electrode. Though the microbial fuel cell can be successfully used to produce energy from different organic substrates by catalytic reactions of specialized microbes, the low energy density output remains the main limitation [190]. One strategy of enhancing the energy yields from MFC is coproduction of several energy types like biohydrogen, biomethane, and bioelectricity. Figure 10 illustrates of how biohydrogen production in MFC can be coproduced with several energy forms from organic effluent substrate. The dark hydrogen production step results in hydrogen production and biochemical byproducts rich in volatile acids like acetate, propionates, butyrate, and methanoates. These are either applied to produce more hydrogen in MFC. The anode electrode contains microbes that are able to metabolize volatile acids by oxidization to generate carbon dioxide, electrons, and hydrogen ions in anode of MFC by electrochemical oxidation. The same oxidation can be achieved by electrochemical reaction at the anode. The hydrogen ions generated are transported to cathode via proton exchange membrane (PEM). The ions are then reduced at the cathode to produce hydrogen gas in the absence of oxygen. The anode and cathode electrodes are connected and the current generated is used as electricity. The volatile acids produced in the dark fermentation can be used as substrate for more hydrogen production through photo-fermentation by specialists' microbes. The effluent from photo-fermentation is used as substrate for hydrogen production in MFC or biomethane production in anaerobic digestion by methanogens. The effluent from MFC should be subjected to biomethanation process so that more energy is recovered as demonstrated in Fig. 10.

An investigation with the soya edible oil refinery effluent produced both biomethane and bioelectricity using microbial fuel cells and microbial electrolysis cells [191]. The possibility of producing bioenergy and some useful chemicals from

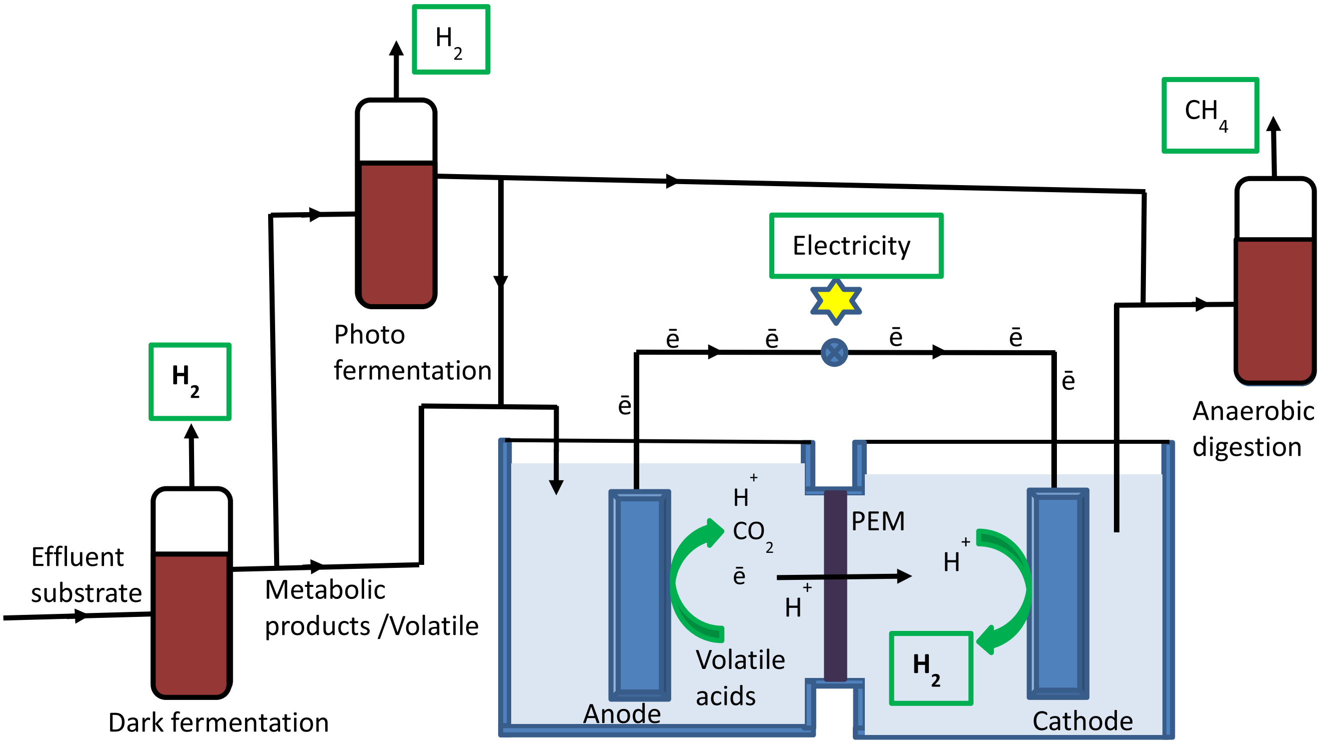

Fig. 10 Use of MFC to coproduce biohydrogen with other bioenergy types

biorefinery waste by the use of microbial fuel cells have been reviewed [192]. It has also been observed that coupling of hydrogen fermentation with microbial electrolysis drastically increased hydrogen production by several folds [193].

In addition to electricity, biomethane, and biohydrogen, other useful products like minerals, heavy metals, industrial chemicals, and nutrients can be recovered from wastewater through electrochemical processes [194]. A process incorporating dark hydrogen fermentation, anaerobic fermentation, and MFC observed that the dark hydrogen fermentation had the highest energy recovery per COD but MFC had the highest COD removal [146]. A separate study with 3 common volatile acid products from biohydrogen fermentation showed that acetate had highest current density compared to butyrate and propionate with the latter having the least current potential [195].

The effectiveness of MFC is affected by factors like foulants and substrates characteristics [196]. The problem is even more intense when the substrate is complex organic wastewater where foulants are abundant. Therefore, more studies to overcome this challenge are required. Another short-coming of MFC is up-scaling for industrial use and process optimization. The factors to consider for optimization and scale-up have been reviewed [55]. Thus, it is fruitful to optimize the fermentation conditions so that the reaction pathway that produces acetate and not the other byproducts is followed. Other areas of research which future research can embark on to make MFC economically viable include pretreatment of electrodes, addition of chemicals, and bio-augmentation to enhance microbial electro-activity. The MFC technology is still at the research stage, and more investigations are necessary to enable commercial application.

6 Discussion

The conventional methods of enhancing bioenergy productivity and yield from biomass include reactor choice and their modifications, substrate choice, process optimization and microbial culture selection. These approaches are limited in inducing a breakthrough in the cost-effectiveness of the biohydrogen production process in order to compete with fossil fuels. The use of advances in biotechnology which include metabolic engineering where metabolic pathways are modified to produce more hydrogen from the substrate has a good potential of creating a revolution in biohydrogen fermentation. However, these investigations are in the infant stage which implies that more research on the same is required [197]. The limitations of low energy yields and high production costs indicate that multi-facial investigations are required so that the process achieves the required cost-effectiveness.

The application of AOPs in enhancement of biohydrogen fermentation is a new development used in the enhancement of biodegradability of substrate for higher productivity and yields [198]. This application is most appropriate where the biomass substrate contains recalcitrant, toxicants, or inhibitors to fermentation processes. In this regard, the main advantage of using AOPs is their selectivity to target the recalcitrant or inhibitors for removal by mineralization or enhancement of their biodegradability. Ultimately, this ensures that the other portion of the substrate remains unaffected and is available for subsequent biohydrogen production. There are reports indicating that physical-chemical pretreatment of lignocellulosic biomass substrates produces compounds that are inhibitory to biohydrogen production [199]. However, more studies are required especially with AOP pretreatments.

Another application of AOPs is in enrichment of biohydrogen inoculum to eliminate competing microbes including methanogens, acidogens, and sulfur-reducing bacteria. These competitors not only consume the substrate at the expense of biohydrogen microbes but also scavenge on hydrogen generated to produce other byproducts. The use of microwave-enhanced AOPs, ultrasonication, and gamma irradiation are among the AOPs successfully applied for selection of biohydrogen specialist [102, 105, 107]. Ultrasonication is among the most promising AOPs in pretreatment of biohydrogen inoculum [200, 201]. Optimization of inoculum pretreatment time is necessary to achieve high hydrogen yields. Use of intermittent ultrasonication was found to enhance yields while excessive ultrasonication reduced the yields [200]. The effectiveness of inoculum pretreatment by AOPs may also be affected by the temperature [201]. Therefore, more studies are required on enhancement of biohydrogen specialists and elimination of competing microbes by the use of AOPs.

The AOPs can also be applied in the treatment of bioenergy effluents so that it meets the disposal standards [202]. Most bioenergy effluents like distillery wastewater and molasses wastewater are dark-colored due to recalcitrant substances that cannot be removed by conventional methods [202]. A promising method of remediating this type of effluent is by the use of AOPs to selectively remove these refractory substances before biological remediation of the effluent is done [203].

The choice of appropriate AOP process for either substrate pre-treatment or remediation of bioenergy effluent is highly determined by the substrates. For substrates with huge recalcitrant like bio-solids and solid wastes, sonolysis is the most promising technology because of its ability to solubilize solid particles which helps increase their biodegradability [204]. Moreover, the process results in negligible COD loss which ensures that maximum bioenergy is recovered. However, the process is energy-intensive and has not been optimized for large scale applications. The application of AOPs as pretreatment for biohydrogen production is dose dependent. Over dosage or over exposure can produce substances that may be more toxic to biodigestion compared to the original substrate. Each AOP type and process should be optimized for maximum energy yields and production.

The biorefinery concept entails the use of common substrates to produce several products. The low substrate conversion in biohydrogen production means that the semi-converted substrate can be utilized to produce other products. Some of the products that can be co-produced with biohydrogen include biomethane [205], polyhydroxyalkanoate [206], and biobutanol [207]. In biorefinery, the use of AOPs can enhance biodegradability of the semi-converted substrate for processing into other useful products. In addition, they can be used to treat the effluent from the processes to attain effluent disposal regulations. The technical and economic effectiveness of AOPs in various stages of bioenergy production can be enhanced by appropriately integrating them with other AOP processes [208]. It is also possible to integrate AOPs with conventional processes like biological and physical-chemical processes. Whereas these other treatment processes may not substantially increase biodegradability or remove the toxicants and inhibitors like AOPs, their ability to remove bulk COD at reasonably low costs makes their integration with AOPs plausible. Furthermore, the use of biological treatment of effluent after enhancement of biodegradability by AOPs enables maximum removal of contaminants at reduced energy costs [209]. In addition, more studies on integration of various treatment processes with AOPs for maximization of the bioenergy yields while minimizing the process costs need to be carried out.

The MFC is among the technologies which employ the principle of electrolysis to generate bioenergy. In addition to biohydrogen, MFC can generate other energy types like electricity [210] and bioethanol [211]. However, the process is in research stage and therefore more studies are required; especially on its scale-up. In addition, production of biohydrogen and other energy types, MFC can be used to remediate bioenergy effluent [212]. The production of biohydrogen from complex organic effluent by MFC can achieve twin objectives of energy production and wastewater treatment. However, the main limitations are low energy production and presence of foulants that requires further investigation.

The main limitation to application of AOPs is the high process costs due to oxidant chemicals in most processes [52, 139]. Ozone and hydrogen peroxide are among the oxidant chemicals required in some processes. Another cost results from the high energy demand in some processes including sonication, microwave-enhanced AOPs, electrochemical oxidation, photocatalysis, and ultra-violet processes [213, 214]. The use of catalysts in processes like heterogeneous Fenton, wet air oxidation, and catalyzed microwave AOPs enhance the process effectiveness. However, they too increase the process costs. It is therefore important to identify cheap catalysts to improve the process cost-effectiveness. The cost of using AOPs depends on the AOP type used, the dosage used, and the exposure time for energy-consuming processes. In electrochemical processes, the initial cost of the electrode and electricity forms part of the major costs. A study on the treatment of wastewater from sugar industry using electrochemical oxidation with aluminum electrode removed 79%

COD and 78% color. The process cost was \$6.22/m^3 and the energy consumption was 58 Kwh/m^3 [215]. Another study on the use of sonoelectrochemical process to degrade ofloxacin, a pharmaceutical recalcitrant pollutant observed 70% COD removal with 11.92 kWwh/g-$COD_{removed}$ and 343 Kwh/m^3 energy consumption [216]. An economic analysis of three AOPs in wastewater treatment observed that electrochemical oxidation was most technically effective but Fenton process was cost-effective, while ozonation had the highest investment cost [217]. The study observed the operation costs for removing 70% COD to be 2.4–4, 0.7–3, and 8.5–10 \$/equivalent O_2 for electrochemical, Fenton, and ozonation respectively [217]. Identification of the right process and optimization of the process are necessary to reduce the process costs.

7 Future direction

One of the limitations to application of AOPs in biohydrogen fermentation is that most processes are not optimized for industrial applications. In the pretreatment of biohydrogen substrate, over dosage or too much exposure of biohydrogen substrate may produce compounds that are toxic to subsequent microbial activity. This not only reduces the biodegradability of the substrate but increases the cost of production due to AOPs chemicals used or energy demand. The same applies to the pretreatment of biohydrogen inoculum with AOPs. Too much exposure destroys the cell membranes and genetic materials of the microbes. It is therefore important that the processes are optimized. Some AOPs like sonication, electrochemical oxidation, and microwave-enhanced AOPs are limited to laboratory scale and pilot studies. Studies on scale up are required to enable commercial application. Integration of AOPs with other physical-chemical and biological processes can be useful in reducing the costs and enhancing the effectiveness of AOPs. In pretreatment of biohydrogen substrates and inoculum, heat and chemical treatment has been widely applied. Studies on how to integrate AOPs with these processes are required. In treatment of final effluent from biohydrogen production, integrating AOPs with processes like coagulation and biological treatment can eliminate COD and color to achieve the standards required by the environmental bodies at much reduced costs. This is because AOPs are more effective in color elimination and enhancement of biodegradability but poor in COD removal. The coagulation and biological processes integrated with AOPs would remove the COD and therefore improve the process technical and cost-effectiveness.

8 Conclusion

The technical feasibility and effectiveness of using AOPs treatment on various stages of bioenergy production processes have been demonstrated by many investigations. There is however little documentation on the cost-effectiveness and economic feasibility of advanced oxidation processes. Most AOPs are generally costly due to high energy requirements like sonolysis, electrochemical oxidation, wet air oxidation, and microwave-enhanced AOPs. Moreover, some processes employ expensive chemicals like ozonation, Fenton oxidation, and catalytic processes. More studies should aim at establishing the economic feasibility of these processes for commercial application.

Some advanced oxidation processes like photocatalysis, microwave irradiation, and microbial fuel cell have not been scaled up for industrial applications. Therefore, investigations on process scale-up ought to be intensified. The integration of appropriate AOPs with conventional treatments like biological and physical-chemical processes can enhance bioenergy substrate pretreatment and final remediation of bioenergy effluent. In biorefinery production, AOPs can be used at various production stages in pretreatment of biohydrogen substrate, excess sludge for production of other energy like biogas, and inoculum to remove competing microbes. Furthermore, they can be used in final remediation of bioenergy effluent's color or recalcitrant COD. The future of application of AOPs in biohydrogen production lies in integrating them with conventional processes like physical-chemical and biological methods for enhancement of their technical and cost-effectiveness.

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