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Review

Current Status and Future Prospects of Sustainable Hydrogen Production from Food Industry Waste by Aqueous Phase Reforming

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Abstract

Aqueous phase reforming has been posed as a promising technology for renewable hydrogen production in the framework of the transition to a sustainable energy economy. Since the use of chemical compounds as process feedstock has proven to be one of the major constraints to its potential scalability, several cost-free residual biomasses have been investigated as alternative substrates. This also allows for the recovery of residues, offsetting the significant costs of waste management through conventional treatment. In recent years, different wastes from the food processing industry such as brewery, fish canning, dairy industries, fruit juice extraction, and corn production wastewaters, have taken the attention of scientific community due to their composition, favorable to this process, and its high-water content. However, few and heterogeneous results can be found within the literature, suggesting that the research into this application is now at a stage of development which will require further investigation. Therefore, this work is focused on compiling and discussing the reported studies, aiming to present a critical reflection on the potential of aqueous phase reforming as a means for the valorization of this kind of residue.

Keywords: aqueous phase reforming; renewable hydrogen; waste biomass; food industry wastes

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1. Introduction

For a long time, H_2 has been one of the most important raw materials in the chemical industry. Currently, it is used for a wide variety of applications such as refining, petrochemistry, the synthesis of ammonia, methanol, fertilizers, and many other products, and, recently, even steel production (as a substitute for coal) [1–3]. Additionally, H_2 is poised to play a pivotal role in the transition to a sustainable energy model as an efficient energy carrier which can be used not only in the industrial sector, but also as a direct fuel and in fuel cell electric vehicles such as trucks, trains, or ships [1,4].

Globally, more than 95% of H_2 is currently produced from fossil sources, mainly by Steam Methane Reforming (SMR), resulting in high CO_2 emissions [1]. However, for a green H_2-based energy model, the research and development into sustainable production processes aimed at reducing the carbon footprint and complying with the Paris Agreement (achieving net-zero CO_2 emissions by 2050) is needed [5]. The utilization of the excess energy generated from solar or wind sources for its production via electrolysis, storage, and subsequent conversion back to electricity through fuel cells constitutes the main option [1],

but complementary technologies must be available, particularly for the H_2 supply in local communities with difficulties connecting to general distribution networks.

One of these emerging technologies is Aqueous Phase Reforming (APR). This is a thermochemical catalytic process that is able to convert aqueous solutions of sugars, alcohols, aldehydes, and other substrates (in the form of different types of biomasses) into H_2. The process is operated under relatively mild conditions (200–270 °C and 15–50 bar) and in the liquid phase, resulting in lower energy consumption compared to SMR, since the water vaporization involves a high heat demand [6–8]. Essentially, APR is a reforming process (i.e., the conversion of oxygenated organic compounds into CO and H_2), but, unlike SMR, under APR operating conditions, the water–gas shift reaction is highly favored. For that reason, C is mainly found in the form of CO_2, with negligible presence of CO, and the overall process can be expressed as shown in (1):

$$C_nH_{2n}O_n + n\ H_2O \leftrightarrow n\ CO_2 + 2n\ H_2 \quad (1)$$

In addition, several side reactions usually take place, leading to the formation of light alkanes (C1–C3) which account for ca. 5–10% of gas products and different by-products (mainly ketones and acids) in the liquid phase as a result of hydrogenolysis, hydrolysis, dehydrogenation, or Fischer–Tropsch reactions [8,9].

The substrate conversion, H_2 production, and the qualitative and quantitative composition of the gas and liquid APR product streams depend mainly on temperature, being 200–250 °C the optimal operating range [8,10]. For example, an increase in the operating temperature can favor endothermic reactions, such as C-C bond cleavage and dehydrogenation, and hinder exothermic ones, such as methanation, resulting in higher H_2 production and concentration in the gas product and a decrease in methane concentration and the formation of water-soluble by-products (e.g., Fischer–Tropsch products) [11,12]. However, an operating temperature above this range increases CO selectivity, probably due to an inhibition of WGS, as it is exothermic and therefore favored at mild temperatures [13]. On the contrary, the H_2 production was observed to dramatically drop below 200 °C [14], corroborating the narrow optimal range of temperatures in the APR process.

Regarding the pressure, many reported works have been carried out in batch mode using a stirred tank reactor and operating under autogenous pressure (corresponding to the water vapor pressure at operating temperature). However, several authors found that increased pressure favors CO/CO_2 methanation reactions, leading to a decrease in H_2 selectivity [15,16], which could be minimized by dilution with N_2 or other inert gases [8].

Although several metals have been explored as active phase for the APR catalyst, an overwhelming consensus can be found in the literature that Pt is the most suitable on the basis of its ability to cleavage C-C bonds and promote WGS, resulting in high activity and high thermal and chemical stability [8,17,18]. At the same time, it is an expensive metal and tends to exhibit relatively moderate H_2 selectivity, whereby other noble (Pd, Ru) and no-noble (Ni, Co, Cu) metals have been tested. However, these metals have been revealed to be non-competitive with Pt due to high selectivity to undesirable side reactions, low stability, etc. [8,10,18,19]. To face this challenge, different Pt-based bimetallic catalysts have been additionally investigated. Pt-Pd, Pt-Fe, Pt-Co, Pt-Rh, Pt-Ru, and Pt-Re allowed for a high substrate conversion and WGS extension, but showed low H_2 selectivity due to side-reaction promotion [16,20].

On the other hand, different materials have also been tested as catalytic supports. As long as it has enough stability in the hydrothermal conditions of the APR process, the textural characteristics and surface chemistry are decisive for the catalytic performance of the material [16,21,22]. Metal oxides, both individually and in combination, have received much attention. On the basis of its low cost, high metal dispersion, thermal stability, and

mechanical resistance, γ-Al_2O_3 has been the most studied among these materials. However, it has a high tendency to become boehmite (AlOOH) under hydrothermal conditions of APR [23,24]. ZrO_2, CeO_2, La_2O_3, and their combinations showed high stability and H_2 production in the APR of methanol and other substrates [24,25]. TiO_2, SiO_2, and MgO have also been studied, but a good balance between catalytic activity and hydrothermal stability was difficult to find [16,17,26–28]. Nevertheless, the best results have been found using carbonaceous supports. Many different materials, such as mesoporous and activated carbons, carbon blacks, carbon nanotubes, or graphene oxides, among others, have been successfully tested due to their high stability under APR operating conditions, tunable surface chemistry, and variable textural characteristics [22,29].

APR was initially developed using individual model compounds with chemical characteristics favorable to this process; alcohols such as glycerol, methanol, ethylene glycol, or ethanol [30–32] have been the most studied. Later, more complex substrates were faced, such as model compounds of biorefinery waste streams [14,33–36] or sugar alcohols such as sorbitol and xylitol [37–39]. However, several works demonstrate that the economic feasibility of the process depends directly on the substrate cost [40,41], whereby the current research interest focuses on wastewater streams as potential APR feedstocks [42]. As an additional advantage, if waste streams, particularly residual biomass, are used as APR feedstock, the obtained H_2 can be considered as renewable/bio H_2, being a very interesting approach to circular energy economy. For example, interesting results have been reported for waste streams from pilot-scale Fischer–Tropsch processes [43], hydrothermal carbonization [44,45], and water-soluble fractions of pyrolysis bio-oil [27,46–48]. However, the higher complexity of these streams compared to model compounds, both individually and mixed, constitutes a handicap and a research niche at the same time.

Solid biomasses and other organic fractions have also been explored for valorization by APR, but, since it is a process that takes place strictly in the liquid phase, the solubilization of the organic matter into this phase is required, which is particularly difficult for lignocellulosic and other types of biomasses. The potential feeding of solid substrate, usually known as direct APR, shows several disadvantages, such as the optimal conditions for APR not necessarily being suitable for the organic matter solubilization and the catalyst being mixed with the solid residue at the end of the process [42]. Therefore, the most studied alternative is a combined process with a pretreatment aimed at solubilizing organic matter in the process water, which was subsequently submitted to APR [49–52].

Finally, in recent years, significant attention has been paid by the scientific community to waste streams from the food processing industry (i.e., any kind of waste stream or fraction, solid or liquid, of a biomass nature, produced at any step of various food production processes, which can be valorized by APR), such as milk whey or wastewater from breweries, fish canning, or fruit juice extraction, among others, due to their significant production and their potentially suitable characteristics as substrates for APR [53]. Global production of milk whey exceeds 200 million tons per year [54], but much greater than this is the production of wastewater during the brewing and fruit juice extraction processes, taking into account that up to 10 L of wastewater are produced per liter of beer or fruit juice (with beer production amounting 1800 million hectoliters worldwide or 1.4 million tons of orange juice alone) [55,56]. On the other hand, these residues usually consist of suitable compounds for this process (mono- and polysaccharides, among others), frequently as aqueous solutions or, in the case of solids fractions, with a high moisture content. Although some review works reported in the literature in recent years include the use of waste biomass from the agriculture and, especially, food industries, none of them are particularly focused on this topic. However, the results are highly variable and are beginning to reveal certain limitations that must be overcome for their potential large-scale

exploitation. With that, the objective of the current work is to review the state of the art related to the APR of food industry waste streams and discuss the potential and future perspectives of this application.

2. Bibliometric Analysis

A search of the reported literature on APR was performed using the Web of Science database. Figure 1a shows the reported works containing "aqueous phase reforming" in the title. As can be seen, APR has received sustained interest in the research community. Since the publication of the work by Cortright and co-workers in 2002 [57], which is usually considered to be the first reported research on this process, the number of published papers has grown year by year up to reach ca. 450 to date, with an average annual publication rate of 27 works.

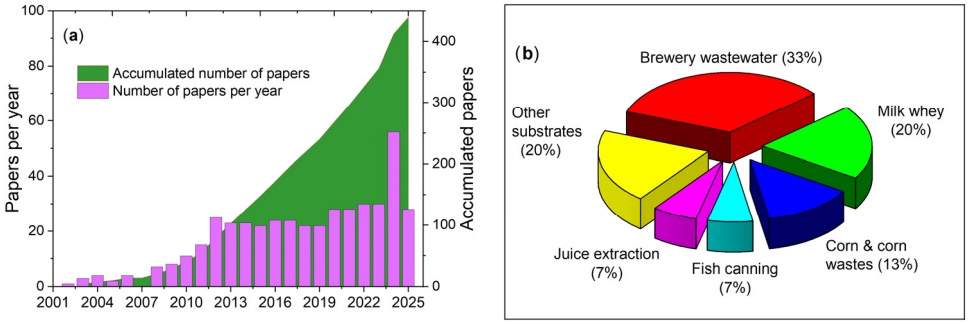


Figure 1. (**a**) Scientific papers published on the APR process and (**b**) on the use of food industry wastes as an APR substrate.

This preliminary bibliographic search was refined by an additional search in the "Topic" field using the following keywords: "waste biomass", "food industry waste", "food industry residue", "brewery wastewater", "fruit juice wastewater", "cheese whey", "milk whey", "corn residues", "pulp waste", and "fish-canning industry". It was found that the use of biomass, both raw and waste, related to food industry was first reported in 2013, and the number of published papers remains low, totaling 17 (Figure 1b). In addition, considering the origin of the waste biomass studied, it can be observed that, so far, many different types and sources of biomass have been tested in just one or a few papers. This suggests that the research into this application is now at a stage of development focused on screening potential substrates, which will need further investigation.

3. Reported Literature Analysis

The review of the reported works has been structured according to the type of the biomass or the food industry from which the waste originates. Five of them can be found: wastewaters from breweries, fish canning, dairy industries, fruit juice extraction, and corn processing. In addition, an additional miscellaneous group has been considered, including different works related more or less directly to the food industry, but which did not corresponding to any of the listed areas. In this bibliographic analysis, prior review articles have been excluded; only original research contributions pertinent to the addressed topic have been considered.

3.1. Breweries

The brewing industry generates significant volumes of wastewater rich in organic compounds from raw materials such as yeast, maltose, and other fermentable sugars. Its use as APR substrate has been deeply investigated by Oliveira and co-workers.

Oliveira et al. [29] studied synthetic brewery wastewater (TOC ≈ 1968 mg/L, COD ≈ 6229 mg/L). Later, Oliveira et al. [21] compared this synthetic substrate with real brewery wastewater (1646–1871 mgTOC/L, 4764–5846 mgCOD/L) using similar APR conditions. Due to the alkaline pH of real wastewater, subsequent work used synthetic solutions at pH 10 and similar organic loads [58,59]. Finally, Oliveira et al. also tested pre-hydrogenated maltose (as maltitol) as a substrate [60].

Experiments used Pt or Pt-based bimetallic catalysts on carbon supports in batch or continuous reactors (200–225 °C, 24–30 bar). High TOC removal (up to 99%) and gas conversion (up to 93%) were achieved, with H_2 being the main product (over 70% of gas phase). Higher organic loads slightly increased H_2 yield, but reduced the substrate conversion. Bimetallic Pt-Re catalysts improved H_2 production, but suffered deactivation. Pre-hydrogenation to maltitol increased H_2 selectivity and the catalyst stability.

3.2. Dairy Industry

A waste stream from the dairy industry that has been explored as an APR substrate is milk whey. It is a liquid by-product generated during cheese and dairy production as a result of casein coagulation and separation. On average, 9 kg of whey are produced per kilogram of cheese, representing a significant global waste stream. Whey contains a high organic load, with lactose concentrations between 4.5 and 6%, as well as proteins, organic acids, and salts, resulting in elevated COD values. Untreated disposal can cause severe environmental impacts, prompting the development of valorization technologies. However, direct use of whey in APR is challenging due to the presence of solids, proteins, and salts, which promotes catalyst fouling and reduce system stability.

Remón et al. [61] used real whey generated during cow cheese production. The tests were carried out in continuous tubular reactors operating at 220 °C and 44 bar, with a Ni-La/Al_2O_3 catalyst and different space velocities. Under these conditions, complete lactose conversion and 25–35% carbon-to-gas (CCgas) yield, mainly composed of H_2 and CO_2 (up to 32.6% H_2), were achieved.

On the other hand, the same authors [62], Joshi and Vaidya [63], and Pipitone et al. [63] employed lactose as a whey model compound to avoid interference from complex whey components in 1–10, 1–5, and 2.5–10 wt%, respectively. Remón et al. used the continuous reactor described above, while Joshi and Vaidya and Pipitone et al. operated in batch mode. In addition, the works reported by Remón et al. and Joshi and Vaidya [62,63] were carried out at lower temperatures (220 and 215–245 °C, respectively), and using Ni-La/Al_2O_3 and Pt-Ni/hydrotalcite catalysts, while 230–270 °C and commercial Pt/C and Ru/C catalysts were used by Pipitone et al. [64], with different metal concentrations in all cases. Finally, the potential improvement in H_2 production by pre-hydrogenation of the substrate, previously proposed by Oliveira et al. [60] for maltose, was also tested by Pipitone et al. [64]. These studies showed very heterogeneous results; for example, C conversion to gas products ranged between 5–41, 33–85, and 5–20% [62–64], respectively. These works corroborated the marked dependence of the H_2 production and selectivity on lactose concentration, with a maximum value of 73% reported by Joshi and Vaidya [63]. Finally, lactose pre-hydrogenation resulted in an improvement in APR performance, with a noticeably increase of H_2 selectivity from 2.5 to 70% [64], corroborating the interest in this pre-treatment to improve APR yield using disaccharides as substrates.

3.3. Corn Production and Processing

In this case, no waste fraction was used, but rather raw corn; the high production surplus in some countries, such as the USA, allows us to consider its potential valorization into H_2 by APR, since this seed is mainly composed of starch [65,66].

Tiryaki and Irmak initially studied field corn, focusing on the effect of hydrolysate concentration (2430; 4050; 5670 mgTOC/L) on H_2 production [65]. Later, they examined the influence of corn variety (field corn, non-genetically modified—non-GMO—field corn, yellow field corn, white field corn, and popcorn) under uniform cultivation conditions to minimize external variability [66]. In all cases, corn was ground and sieved (<1 mm particles) to facilitate subcritical water solubilization, producing a hydrolysate suitable for APR. Both studies were carried out in batch mode and using 5 wt% Pt on activated carbon as catalyst. Hydrolysates were prepared from 6 g corn in 350 mL water at 200 °C for 1 h under subcritical conditions (13.79 MPa CO_2). The resulting hydrolysate was then processed via APR with 0.2 g catalyst at 250 °C, autogenous pressure, for 90 min.

The results show that the H_2 yield was similar across all corn varieties, averaging 130 mL H_2/g corn. The initial C in the hydrolysate mainly yielded a solid fraction, with only 1.5–1.9% converted to gas. The highest H_2 yield was achieved with the lowest hydrolysate concentration, while higher concentrations reduced H_2 yield and increased CO_2 due to solid formation and catalyst center saturation.

3.4. Fish Canning Process

Oliveira et al. reported the first application of APR to valorize a wastewater generated during the tuna cooking, a typical effluent from the fish canning industry [67]. The substrate was a synthetic solution mimicking real waste, characterized by a high organic content (TOC ≈ 1895 mg/L, COD ≈ 4996 mg/L) and a complex ionic composition with high chloride, acetate, and phosphate content, and smaller amounts of formate and sulfate. The experiments used 3 wt% Pt catalysts supported on three commercial carbon materials: two activated carbons (Norit CAPSUPER, Norit SXPLUS) and a carbon black (ENSACO 350G). APR tests were performed in stainless steel reactors operating in both batch and semi-continuous modes using 20 mL of effluent and 0.4 g of catalyst at 200 °C for 4 h. In the semi-continuous mode, a constant Ar flow (1 NmL/min) was maintained.

TOC and COD removing of 45–60% was achieved, and total gas production ranged from 63 to 366 µmol, with the highest values for carbon black support and in semi-continuous mode. However, CO_2 was the main gas product, while H_2 and alkanes accounted for up to 18% of the gas volume. ENSACO 350G yielded the highest H_2 fraction (up to 9% in batch mode). Semi-continuous operation reduced the alkane content and slightly increased H_2 yield. The catalyst stability was higher for activated carbons, while carbon black showed progressive deactivation.

3.5. Fruit Juice Extraction

Saenz de Miera et al. [68] investigated the feasibility of using APR for the treatment of wastewater from the fruit juice extraction industry, which generates large volumes of effluents with high organic content. The feedstock was a synthetic effluent representative of juice industry wastewater, rich in simple sugars (glucose, fructose) and organic acids (citric, ascorbic, galacturonic). The effect of salinity was also evaluated by adding typical inorganic salts. The experiments used a 3 wt% Pt/ENSACO 250G catalyst in a 50 mL stainless steel batch reactor operating at 220 °C with 0.3 g/15 mL solution for 4 h. The influence of initial pH (2, 7, 10, 12), organic load (2873–11,592 mg/L), and salinity (low: 150 mg/L $(NH_4)_2SO_4$, 13 mg/L KH_2PO_4, 329 mg/L NaCl; high: 300 mg/L $(NH_4)_2SO_4$, 27 mg/L KH_2PO_4, 640 mg/L NaCl) was assessed.

High carbon removal was achieved, especially under acidic conditions, reaching up to 91.7% TOC reduction. The average H_2 yield was ca. 7 mmol H_2/g initial COD, peaking at 7.8 mmol H_2/g COD at pH 10. The increase in organic load slightly reduced

gas conversion due to enhanced solid formation. High salinity did not significantly affect carbon conversion, but decreased H_2 yield.

3.6. Other Waste

In other works, a wide range of substrates has been employed, from polysaccharides such as starch to disaccharides like sucrose, as well as mixtures containing carbohydrates, proteins, and lipids. Although these substrates cannot always be attributed to a specific food industry, they represent different stages within the life cycle of food industry processes [18,20,69].

Oliveira et al. used both synthetic and real starch-rich wastewater from food processing (rice, potato, sweet potato, cassava), with starch as the main component [20]. Pt and Pt-Ru, Pt-Pd, Pt-Re, and Pt-Rh supported on ENSACO250G were used as catalysts at 220 °C in a 100 mL batch reactor, loaded with 20 mL of wastewater and 0.4 g of catalyst. The H_2 yield reached maximum values of 51 mmol H_2/g initial TOC with starch-rich synthetic wastewater and Pt/C or Pt-Ru/C catalysts, while results decreased significantly for the real substrate (28.5 mmol H_2/g initial TOC).

Joshi et al. employed a synthetic mixture simulating food waste, including starch, glucose, meat peptone, and sunflower oil [18]. In this case, commercial Pt/C and Ru/C catalysts were used also in batch mode, studying the influence of operating temperature (180–235 °C), time (1.5–6 h), and initial pH (5–9). In addition, a substrate pretreatment under hydrothermal conditions was evaluated. A highest H_2 yield of 2.13 mmol H_2/g COD and 60% COD reduction were reported. The hydrothermal pretreatment was revealed to improve results, probably due to the removal of inhibiting compounds or the organic matter fractionation.

Finally, Godina et al. utilized technical sucrose hydrogenated to produce a sorbitol-mannitol mixture [69]. The experiments were generally performed in a continuous operating mode reactor at 180–235 °C using a 2.5 wt% Pt/C catalyst and different space velocities (0.5 and 1.1 h^{-1}) for more than 100 h. A maximum H_2 selectivity of 62% was achieved by the APR of polyols derived from sucrose, with similar yields observed for both commercial sorbitol and the sorbitol–mannitol mixture obtained by hydrogenation.

4. Discussion

Table 1 summarizes the essential information obtained from the reported works described above. Most studies published to date on the application of APR to food industry waste used aqueous fractions, such as milk whey or wastewater from juice extraction, fish canning, and other industries, since no pretreatment is needed to solubilize the organic matter. The best results were obtained for milk whey and wastewater from breweries, with high organic matter conversion and H_2 production for both cases, but a significant catalyst deactivation was observed for whey. On the contrary, lower H_2 production and high conversion of C to solid fraction was found for wastewater from fish canning and corn processing. It was assumed that the modest results obtained with fish caning wastewater could be due to the high protein and fat content, but the good results obtained using milk whey (which also has a high content of these components) suggest that pH and high salinity could impair reforming.

Table 1. Essential information from reported works on the APR of food industry wastes.

Substrate		Experimental	Outcomes	Reference
Brewery wastewater	Synthetic	Pt on different supports 220–225 °C, batch mode	99% TOC removal 93% CCgas, 70% H_2 Best support: CB ENSACO250G (15 mmolH_2/gCOD)	[29]
	Synthetic and real		TOC removal and H_2 production over 80% and 9 mmolH_2/gCOD	[21]
	Synthetic	Pt-Re (1:1,2:1,1:2)/AC catalyst 225 °C, continuous mode WHSV 0.03–0.48 h^{-1} Ar velocity 0.1–0.8 cm/s	0.18 mmol H_2/min (Pt:Re 1:1) CCgas up to 41% Best results for 0.03 h^{-1} and 0.8 cm/s	[58,59]
Milk whey	Synthetic and real	Ni-La/Al_2O_3 catalyst, 220 °C Continuous (60–130 g/min·gC)	100% lactose conversion CCgas 25–35% Up to 36.5% H_2	[61]
	Synthetic	Ni-La/Al_2O_3 catalyst 220 °C 10–40 g catalyst/g lactose	100% lactose conversion CCgas 5–41% 8–58% H_2	[62]
		Pt-Ni/hydrotalcite 215-245 °C, 1.5-6 h 1-5 wt% lactose	↑ lactose conc. > ↓ H_2 prod. Max. H_2 selectivity: 73% ↑ T > ↓ CH_4 generation	[63]
		Pt/C and Ru/C catalyst 230–270 and 180–220 °C 2.5–10 wt% lactose Lactose pre-hydrogenation	↑ lactose conc. > ↓ H_2 prod. Up to 20% CCgas and H_2 conc. Increased to ca. 60 and 30% by lactose pre-hydrogenation	[64]
Corn	Field	5 wt% Pt/C catalyst 250 °C, 90 min Pretreat. subcritical cond. 2430–5670 mgTOC_o/L	↑ initial conc. > ↓ H_2 prod. Up to 130 mL H_2/g corn	[65]
	Field, non-GMO, yellow, white and pop corn	5 wt% Pt/C catalyst 250 °C, 90 min Pretreat. subcritical cond. 2430 mgTOC_o/L	ca. 130 mL H_2/g corn for all cases CCgas 1.5–1.9%	[66]
Fish canning wastewater	Synthetic	3 wt% Pt on Capsuper, SXPlus, Ensaco350G Batch and Semi-cont. 200 °C, 4 h	↓ TOC/COD 45–60% ENSACO: ↑ prod. H_2, ↓ stability ca. 366 µmolH_2, 18% in gas	[67]
Fruit juice extraction wastewater	Synthetic	3 wt% Pt/C catalyst Batch, 220 °C Initial pH 2–12 2873–11,592 mgCOD_o/L Low (50 mg/L) and high (300 mg/L) salinity levels	92% max. TOC removing (↓ pH) 8 mmolH_2/gCOD max. (↑ pH) ↑ salinity > ~TOC conv. ↓ H_2 production	[68]
Others	Synthetic and real starch-rich waste	Pt, Pt-(Pd,Re,Ru,Rh)/CB Batch, 220 °C	max. H_2 prod. > Pt, Pt-Ru synthetic: 51, real: 29 (mmol/gCOD)	[20]
	Synthetic food waste	Pt and Ru/C catalysts Batch, 180–235 °C pH 5–9, pre-HTC	60% COD removal ca. 2 mmolH_2/gCOD max. Pt/C best catalyst	[18]
	Commercial sorbitol Sorbitol/mannitol mixture from sucrose hydrogenate	2.5 wt% Pt/C catalyst Continuous, 225 °C, 100 h WSHV 2.5–1.1 h^{-1}	max. 62% H_2 selectivity similar results for both substrates	[69]

Regarding the operating conditions, optimum values were found to cluster within a narrow range, despite the different types of residues employed. The process must be operated in the 200–250 °C interval, with 225 °C being the most frequently used value, which favors both conversion and catalyst stability. Pressure varies between 24 and 45 bar, with a frequent use of autogenous pressure in batch systems. Additionally, batch experiments typically employ a reaction time of 4 h after the heating period from room temperature. In contrast, continuous studies often explore different space velocities or residence times, with lower values being found to enhance gas conversion. Higher organic loads generally reduce H_2 yield and promote the formation of by-products. This homogeneity in optimal operating conditions suggests that the variability in results depends mainly on the substrate and the catalyst rather than on the operating parameters that have already been established in multiple studies of the APR process.

Based on the reported results, future research on the valorization of food industry waste by APR should focus on milk whey and brewery wastewater to determine the optimal chemical pretreatment (hydrogenation, etc.) of substrates in order to increase hydrogen production. To date, many of these reported studies were carried out using synthetic substrates, so it could be interesting to conduct further research feeding real wastewater into the process. In addition, solid residues from the food industry with high moisture content would be promising substrates for the APR process. In this case, since a previous solubilization stage is required, the main objective should be to optimize the operating conditions of the overall process, not to achieve maximum solubilization of the organic matter, but rather to obtain the liquid phase composition that achieves the best performance as APR substrate.

On the other hand, although several alternatives have been explored (both mono- and bimetallic catalysts), the best catalytic performance was exhibited, by far, by Pt on different supports, with a particular predominance of carbon materials. However, it is often affected by a lack of long-term stability. For this reason, the most interesting future direction for research on APR catalysts could be related to the development of an effective procedure for catalyst regeneration, since improving stability through the modification of the metal and/or support has not yet been completely successful.

5. Conclusions

Considering the body of available studies, it can be affirmed that APR is a promising technology for the energetic valorization of food industry waste, particularly when in liquid form. However, there remains a critical dependence on both the type of waste and the catalyst employed. Furthermore, there are currently very few studies on this topic, and none have yet been conducted on an industrial scale. Many works still rely on model solutions or pretreated matrices, which significantly limits the real-world validity of the results.

From a practical standpoint, waste that offers greater ease of operation, good hydrogen yield, and lower formation of solid by-products appears to be the most suitable candidate for further development in the short-term, as is the case with wastewater from the brewing industry. Nevertheless, whey, owing to its high availability and lactose content, could emerge as a valuable feedstock if effective pretreatment strategies and more robust catalysts that are resistant to deactivation are developed.

With regard to the future of APR using food industry residues, the results indicate that the highest research priority should be the development of more resilient and selective catalytic systems tailored to the impurities present in real waste streams. Additionally, it will be essential to implement continuous systems that more accurately represent industrial environments and to conduct economic and scale-up studies to assess the commercial

viability of the process. Should these objectives be achieved, it is reasonable to assume that APR of food waste could become established as a key technology in the production of renewable hydrogen.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

APR	Aqueous phase reforming
COD	Chemical Oxygen Demand
TOC	Total Organic Carbon
SMR	Steam Methane Reforming
WGS	Water-Gas Shift reaction

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