Valorization of Agro-Food Industrial Residues by Solid-State Fermentation

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Agro-food industrial residues (AFIRs) are generated in large quantities all over the world. The vast majority of these wastes are lignocellulosic wastes that are a source of value-added products. Technologies such as solid-state fermentation (SSF) for bioconversion of lignocellulosic waste, based on the production of a wide range of bioproducts, offer both economic and environmental benefits. The versatility of application and interest in applying the principles of the circular bioeconomy make SSF one of the valorization strategies for AFIRs that can have a significant impact on the environment of the wider community.

lignocellulosic biomass | value-added products | bioactive compounds | biofuels | feed

1. Introduction

The term “residue” includes materials that are not intentionally generated in the production process but are not necessarily considered waste [1]. Significant amounts of residues are generated during the processing of plant raw materials that are transformed into final products, e.g., in wine production and grain processing, residues account for about 30% of the processed raw material mass [2][3]. They are treated as waste and their unregulated disposal into the environment can cause serious environmental problems [4]. AFIRs are a wide variety of biomass, including pomace, fruit, and vegetable peels; husks, bran, and germ of cereals; pods; stalks; and pomace left over after oil production. Due to their chemical composition, they are rich sources of high value components such as polysaccharides, proteins (including enzymes), dietary fibers, fatty acids, flavors and aromas, and bioactive compounds [5][6][7][8]. High-value components refer to components that have health-promoting properties and a wide range of potential industrial applications (pharmaceutical, food, and cosmetic industries) due to their biological activity or nutritional value [5][6].

AFIRs are mostly lignocellulosic materials composed of three polymers: cellulose (40–50%), hemicellulose (20–30%), and lignin (20–35%). Lignin is the main component of the cell wall [9][10]. According to numerous studies, SSF is one of the most suitable techniques to obtain the desired biomolecules from lignocellulosic materials. SSF has been extensively studied for potential applications in fuel, food, and feed, as well as in the chemical and pharmaceutical industries [11][12].

By-products from lignocellulosic biomass are an important alternative energy source, playing an important role in the circular bioeconomy. The management of this resource promotes the reuse of raw materials, high industrial production yields, and the generation of minimal waste [13]. The efficient use of natural resources, the development of new technologies, and the improvement of existing ones increase the value of agricultural waste. Their use in the production of biogas, biofuels, biofertilizers, bioactive compounds, and pharmaceuticals is consistent with sustainable development and the business model of using agricultural waste in the bioeconomy. SSF can be applied as a technique of biological processing of different lignocellulosic materials to obtain different products following the concept of the 3-R approach “reduce, reuse, recycle” [14], thus contributing to the circular bioeconomy [15].

2. Agro-Food Industrial Residues

The increasing expansion of agro-industrial activities in recent decades has resulted in the accumulation of a large amount of lignocellulosic residues (wastes or by-products) worldwide, which are not properly disposed of and thus contribute to climate change, as well as soil, water, and air pollution [16][17]. An estimated one-third (≈1.3 billion tonnes) of food produced for human consumption is wasted annually worldwide [18]. Although some of these residues are used as animal feed, large quantities are disposed of in landfills or incinerated [17]. In general, AFIRs can be divided into agricultural residues and food industry residues (**Figure 1**). This research does not cover food waste, which includes unsold food, leftovers, and uneaten food from households and restaurants, as well as from large-scale producers such as caterers and supermarkets [19].

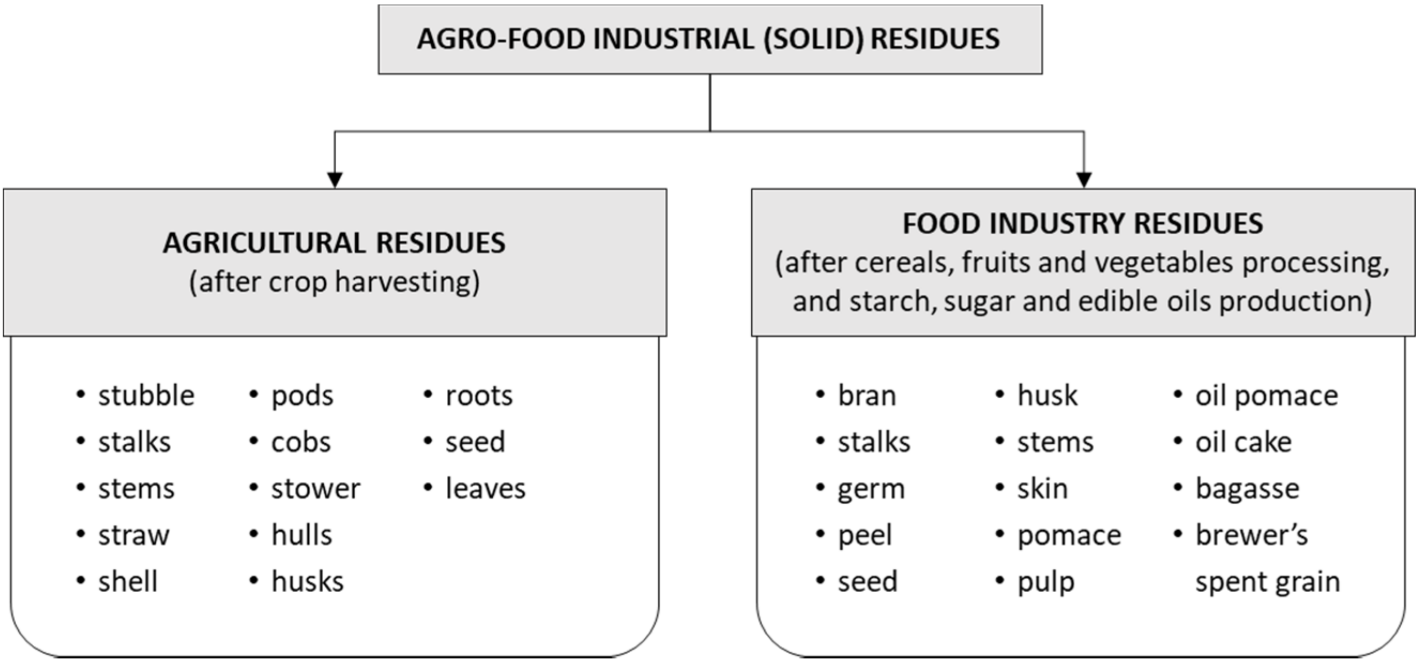

Figure 1. Classification of AFIRs.

3. Solid-State Fermentation (SSF)

3.1. General

SSF is a fermentation process in which microorganisms grow on moist, solid material under controlled conditions, without the presence of free water or with a minimal amount of free water. Inert or non-inert materials can be used as solid substrate in SSF processes [20]. AFIRs are among the non-inert solid substrates that serve as nutrients for microbial growth and metabolite production. After fermentation, they can also be the product of fermentation, used for feed or biofuel production.

The microorganisms used in SSF are filamentous fungi, yeasts, and bacteria. Due to their physiological, biochemical, and enzymatic properties, filamentous fungi (multicellular organisms) are the most commonly used, especially those from fungal kingdom sub-division *Basidiomycota* and *Ascomycota* [21][22]. In order to develop a reliable and repeatable SSF process, researchers should carry out this process in specific types of bioreactors under controlled conditions, such as tray bioreactors, rotating disc reactors, fixed bed bioreactors, column bioreactors, air pressure pulsation solid state bioreactors, rotating horizontal drum bioreactors, stirred drum bioreactors, fluidized bed bioreactors, air-lift bioreactors, and immersion bioreactors [8][23]. The most suitable type of bioreactor for SSF scale-up is the tray bioreactor. It is a traditional type of bioreactor used in SSF, most commonly in laboratory research for enzyme production [24], for lignin degradation [23][24] for the application of biologically pretreated material in the process of biogas production [25]. It is also used in commercial processes in various industries, such as the production of fermented foods such as tempeh [8] and the production of various enzymes [26]. This is due to its simple design and ease of use [27].

The advantages of SSF over submerged fermentation (SmF) are its similarity to the natural habitat of microorganisms, higher productivity, lower cost (due to the use of cheap agro-industrial residues as substrates), lower water consumption, lower use of chemicals, lower generation of waste streams, and lower energy consumption [21][28][29][30]. Despite these advantages, there are some important technological concerns that need to be considered in order to improve the overall SSF process. Some of them are problems caused by the heterogeneity of the system, such as heat and mass transfer resistance; separation of the microorganisms from the substrate; and sampling problems during fermentation for continuous monitoring of the chemical composition of the substrate and/or product accumulation [31].

The most important factors that have effect on the efficiency of SSF process are substrate (chemical composition, humidity, and particle size), inoculum (concentration, age, and morphology), external carbon and/or nitrogen addition, addition of a specific enzyme's inducers for microorganism's growth and/or desired metabolite production, mixing, temperature, pH, and oxygen concentration.

The basic information of substrates and microorganisms that are commonly used in SSF are provided further here.

3.2. Substrates Used in SSF

The choice of substrate is usually determined by its cost and availability, its chemical composition, and its suitability to be converted into a particular product via biochemical pathways. Depending on the objective (production of the

desired enzyme, production of the desired phenolic compounds, organic acids or other valuable product, use for biofuel production, use for feed processing), it is important to know the chemical composition of the substrate and to select a suitable microorganism. If the substrate does not contain the required amounts of nutrients, some macro- and micronutrients are added for optimal growth of the microorganisms [32][33]. Macronutrients (carbon, nitrogen, oxygen, hydrogen, sulfur, phosphorus, Mg^{2+}, and K^{+}) are needed in concentrations greater than 10^{-4} M, whereas carbon in the growth medium is the main source of energy. Microelements (Mo^{2+}, Zn^{2+}, Cu^{2+}, Mn^{2+}, Ca^{2+}, Na^{+}) and vitamins, growth hormones, and metabolic precursors are needed in concentration less than 10^{-4} M [21]. AFIRs are a source of carbon, nitrogen, and nutrients and can therefore serve as solid carriers suitable for nutrient absorption and biomass growth [33]. Sometimes it is necessary to combine several different residues according to their chemical composition and to use such a mixture as a substrate to ensure sufficient nutrients for the optimal growth of microorganisms [11]. For SSF, the moisture content of the substrate is one of the most important operating parameters that affects the whole fermentation process. If the moisture content is too high, the interstitial spaces of the solid material will be filled with water and gas diffusion will be restricted. On the other hand, if the moisture content is too low, the growth of microorganisms will be impaired. The optimum moisture content depends on the substrate and the microorganism and changes during the fermentation process [34]. The final water content is the sum of the initial water content and the water produced by the metabolism of the microorganisms minus the water removed by evaporation. Water partially evaporates, and at the same time it is produced by the metabolism of the microorganisms [35]. If the production of metabolic water is greater than the evaporated water, then the water content is reduced [23].

Particle size and shape of the substrate can affect the accessibility of nutrients to the microorganism. Smaller particle size causes the smaller inter-particle spaces and the greater pressure drops when air flows through the substrate mass. The particles with a larger surface area tend to be contiguous with the flat surfaces and thus actually exclude oxygen, limiting the growth of microorganisms [36]. The particle size range of the substrate used in SSF is usually between 0.25 and 7.5 mm, depending on the type of substrate used [37][38][39][40][41].

3.3. Microorganisms Used in SSF

Filamentous fungi, yeasts, and bacteria can be used in the SSF process to produce value-added compounds. Unicellular organisms such as bacteria and yeast grow as a biofilm, while multicellular filamentous organisms grow in the form of a mycelium, which is comprised of aerial and penetrative hyphae. If the layer of hyphae is thick, then water moves by capillary action from the substrate, resulting with layer into a moist biofilm. A biofilm can also be formed in the case when the bed is mixed, since mixing causes squashing of aerial hyphae onto the surface of the substrate [8]. The most commonly used microorganisms in SSF are filamentous fungi. The choice of microorganism depends on the desired end product, while the choice of substrate is an important parameter for the successful growth of the selected microorganism [42]. The microorganisms can be used as single cultures, as identifiable mixed cultures, or as a consortium of mixed indigenous microorganisms. Many factors can affect the growth of microorganisms, such as the moisture content and properties of the substrate (chemical composition, particle size, height of the substrate layer), temperature, aeration, mixing, initial concentration, and age of the microorganisms [42][43]. Microbial growth usually results in the release of metabolic heat. High temperatures can lead to denaturation

of enzymes and affect metabolite production. Since SSF occurs in the absence of free water, it is difficult to dissipate the heat generated during microbial growth due to the limited thermal conductivity of the solid substrate and the low heat capacity of the air. The difficulties in controlling the temperature in SSF can become even more pronounced when the process is carried out on a large scale.

4. Enzyme Production by SSF

During the biotransformation process of AFIRs for the purpose of producing various value-added products, biofuels, or animal feeds, the conversion of lignocellulosic biomass into fermentable sugars, sugar acids, and/or phenols is carried out by a complex enzymatic system of selected microorganisms [44].

SSF can offer significant benefits in the economic aspects of the enzyme production compared to SmF, since it uses low-cost and easily available substrates, such as lignocellulosic substrates, especially AFIRs [45]. Selection of substrate, microorganism, and process conditions has influence on desired enzyme(s) production. This section describes the catalytic activities of the most investigated enzymes produced by SSF by different microorganisms, and possible industrial application are given.

4.1. Lignocellulolytic Enzymes

Lignin is a complex, aromatic, and optically inert hydrophobic amorphous three-dimensional polymer consisting mainly of three different phenylpropane alcohols: *p*-coumaryl, coniferyl, and sinapyl. Their quantities depend on various factors, such as plant species, maturity, and the space localization in the cell [46]. Lignin is responsible for the structural rigidity of plants, their impermeability, and their resistance to microbial attacks and oxidative stress. Due to its properties, lignin is a major obstacle in the AFIR bioconversion process into valuable compounds [18]. The enzymatic system responsible for the fungal degradation of lignin is comprised of ligninases: phenol oxidases (laccase, EC 1.10.3.2) and peroxidases (manganese peroxidase (MnP), EC 1.11.1.13, lignin peroxidase (LiP), EC 1.11.1.7) [47].

Laccases are multi-copper glycoproteins that use molecular oxygen to oxidize various aromatic and non-aromatic compounds by a radical-catalyzed reaction mechanism. Laccase can be used in food and beverage industries for modification of color appearance, in the pulp and paper industry for delignification, and in the textile industry for textile bleaching or dye synthesis, as well as for many other purposes such as soil bioremediation, herbicide degradation, synthetic chemistry, cosmetics, and biosensors [48][49]. Laccases are found in higher plants, insects, prokaryotes, and fungi, but the most commonly used microorganisms in SSF for laccase production are white-rot fungi such as *T. versicolor*, *T. pubescens*, *Ganoderma lucidum*, and *Pleurotus eryngii*. Osma et al. [37] showed that banana peels can be a good substrate for the cultivation of *T. pubescens* under SSF conditions for laccase production. They indicated that by using this type of non-expensive substrates, it is possible to produce enzymes with higher activities at lower production costs. Produced laccase had a maximum activity of 1500 U/L and was found to be more efficient in decolorization of anthraquinone dyes compared to commercial laccase. Potato peel

waste, pretreated with distilled water, is also one of the examples of economical substrates for the production of highly active laccase (6708.3 U/L) under SSF conditions with *P. ostreatus* [50].

Manganese peroxidase (MnP) belongs to the peroxidase family. It is an extracellular glycosylated heme enzyme that uses H_2O_2 to oxidize Mn^{II} to Mn^{III}–chelate. This enzyme is mostly produced by numerous species of fungi (*Basidiomycetes*), especially white-rot fungi, and bacteria (*Actinomycetes*). It belongs to group of enzymes that have a significant role in efficient bioconversion of plant residues. MnP finds its use in various industries—paper, food, dye, textile, cosmetics, and many others [50].

Lignin peroxidase (LiP) is a water-soluble glycosylated enzyme that also uses H_2O_2 for catalysis. LiP is enzyme capable of producing radical cations through one-electron oxidation of nonphenolic aromatic compounds as well as phenolic aromatic compounds such as veratryl alcohol or 1,4-dimethoxybenzene [51]. This enzyme, like MnP, is produced mostly by filamentous fungi and participates in lignin degradation, having many applications in different industries [48][50]. The white-rot fungus *Inonotus obliquus* produces all three of the above ligninolytic enzymes (laccase, MnP, and Lip) under SSF conditions. Xu et al. [52] optimized process parameters such as pH, temperature, substrate moisture ratio, and inoculum level. Various lignocellulosic materials have been used as substrates (wheat bran, wheat straw, rice straw, peanut shell, sugarcane bagasse, cassava peel, birch branch, beech branch). Under optimal conditions, laccase, MnP, and LiP enzyme activities of 81.94 ± 7.55, 1603 ± 7.76, and 1500 ± 21.44 IU/g were obtained, respectively.

4.2. Cellulolytic Enzymes

Cellulose is an unbranched long polymer of β-D-glucose units linked by (1→4) glycosidic bonds to form cellobiose-repeating units in the cellulose chain. A numerous hydroxyl groups SSF can offer significant benefits on the inner and outer surface of cellulose-forming hydrogen bonds, while cellulose chains are interlinked by hydrogen bonds and Van der Waals forces. Owing to different orientations throughout the structure, cellulose molecules have different levels of crystallinity—low crystallinity (amorphous regions) and high crystallinity (crystalline regions) [46].

Cellulases are enzymes that have the ability to break cellulose and convert it into simple sugars. They include endoglucanases (1,4-β-D-glucan glucohydrolase), exoglucanases or cellobiohydrolases (1,4-β-D-glucan cellobiohydrolase), and β-glucosidases or cellobiases (β-D-glucoside glucohydrolase). Endoglucanases (EC 3.2.1.4) randomly hydrolyze internal glycosidic linkages (β-1,4 glucosidic bonds), resulting in shorter polymer chains and an increase of released number of reducing ends.

Endoglucanases find their application in the formulation of detergent compositions for increasing the production yield. They can also be used for improving the nutritive quality of products obtained in different food industry sectors (fruit processing industry; beer, oil, and bakery industries). They are known to be used in feed production [53] and in the textile and pharmaceutical industries as well [19].

Endoglucanases are mainly produced by fungi and bacteria cultivated on AFIRs. The most important producers of endoglucanases are given in **Table 1**. Exoglucanases (EC 3.2.1.91) or cellobiohydrolases (CBHs) catalyze

cellulose hydrolysis to cellobiose units by acting on reducing and non-reducing end of the cellulose. Furthermore, released cellobiose units can be converted to glucose by β-glucosidase [54]. CBHs have tunnel-shaped active sites that accept only a substrate chain via its end terminal regions. It works by stinging the cellulose chain through the tunnel, removing the cellobiose units in a sequential manner [55]. The most important producers of exoglucanases are given in **Table 1**.

Table 1. Valorization of different AFIRs for the production of enzymes from different microorganisms.

Enzymes		Microorganism	Substrate	Reference
Lignolytic	laccase	*Trametes versicolor*	corn silage, brewers' spent grain, barley husk	[56][57][58]
		Trametes pubescens	banana skin	[37]
		Pleurotus eryngii	peach waste	[59]
		Aspergillus flavus PUF5	dried ridge gourd peel	[60]
		Ganoderma lucidum	wheat bran	[61]
		Lysinibacillus sp.	wheat bran	[62]
	manganese peroxidase lignin peroxidase	*Inonotus obliquus*	birch branch, beech branch, rice straw, wheat straw, wheat bran, sugarcane bagasse, cassava peel, peanut shell	[52]
Cellulolytic	cellulase	*Trichoderma* sp.	corn cob, wheat bran	[63]
	endoglucanase exoglucanase	*Penicillium roqueforti*	rice husk	[64]
		Aspergilus fumigatus	wheat straw	[65]

Enzymes		Microorganism	Substrate	Reference
		Thermoascus aurantiacus	Jatropha deoiled seed cake	[53]
		Aspergillus fumigatus	wheat straw	[66]
		Trichoderma viride *Ganoderma lucidum*	corn stover	[67]
	cellobiase	*Humicola insolens*	paddy straw, soybean pod husk, sugarcane bagasse, groundnut shells, corn stalks and pigeonpea pod husk	[68]
	β-glucosidase	*Lichtheimia ramosa*	wheat bran, soy bran, corn cob, corn straw, rice peel, sugar cane bagasse	[69]
		Thermoascus aurantiacus *Aureobasidium pullulans*	wheat bran, soy bran, soy peel, corn cob, corn straw	[70]
		Trichoderma viride *Ganoderma lucidum*	corn stover	[67]
Hemicellulolytic	xylanase	*Aspergillus oryzae*	wheat bran	[34]

Enzymes	Microorganism	Substrate	Reference
	Aspergillus tubingensis	wheat straw, sorghum straw	[71]
	Bacillus stearothermophilus	wheat bran	[72]
	Aspergillus niger	rice straw	[73]
	Aspergillus awamori	tomato pomace	[74]
	Thermomyces lanuginosus	wheat bran	[75]
	Humicola insolens	paddy straw, soybean pod husk, sugarcane bagasse, groundnut shells, corn stalks and pigeonpea pod husk	[68]

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antioxidant potential since the availability of free hydroxyl groups on the phenol ring affects the resonance stabilization of free radicals. The reduced antioxidant activity has a direct impact on the weaker health functionality during the ingestion of these compounds in the body [76]. It has been recognized that bioaccessibility of high-molecular weight polyphenols (e.g., hydrolysable, condensed tannins), complex flavonoids conjugated with sugars and acetylated with hydroxycinnamic acids, are lower compared to aglycones (units without sugar) and low-molecular weight polyphenols [77]. Therefore, liberation of free phenolic compounds may improve their effect on the health functionality. The most important producers of β-glucosidases are given in **Table 1**. In addition to ligninolytic enzymes, the previously mentioned white-rot fungus *I. obliquus* also produces cellulolytic enzymes under SSF conditions. The maximum activities of the enzymes carboxymethylcellulase, filter paper cellulase, and β-glucosidase obtained under optimal process conditions using wheat bran as substrate at 40% inoculum, pH 6.0, and substrate/moisture ratio of 1:2.5 were 27.15, 3.16, and 2.53 IU/g, respectively [52].

Trichoderma is one of the microorganisms that have been extensively studied for the production of various industrially important enzymes, mainly cellulase, exoglucanase, and β-glucosidase under SSF conditions using different AFIRs as substrates. The studies conducted by Shazhadi et al. [67] aimed at hyperproduction of exoglucanase and β-glucosidase using a low-cost and readily available corn stover substrate. Optimization of process conditions (substrate amount 15 g; 50% *w*/*w* moisture, 6 mL inoculum, pH 6.0, 35 °C) for successful growth of co-culture of *T. viride* and *G. lucidum* on corn stover resulted in production of exoglucanase and β-

glucosidase enzymes and their activities of 485 ± 6.5 U/mL and 255 ± 3.3 U/mL after 5 days of incubation, respectively. They also investigated the influence of additional carbon and nitrogen sources regulating enzyme synthesis during growth of white-rot fungi, and the combination of glucose and ammonium sulfate proved to be the best in the production of exoglucanase and β-glucosidase.

4.3. Hemicellulolytic Enzymes

Hemicellulose is a complex of polysaccharide matrixes composed of different units of sugars (xylans, glucans, xyloglucans, callose, mannans, and glucomannans). It is the second most abundant polysaccharide in plant cell wall. Xylan is the most abundant hemicellulose polymer, constituting around 70% of hemicelluloses. Galcto(gluco)mannans and xyloglucans are another two major hemicelluloses in plant cell wall. In order to degrade such a complex material, microorganisms should have ability to produce a large set of hemicellulases, which act in interaction.

Hemicellulases include xylanases (EC 3.2.1.8), β-mannanases (EC 3.2.1.78), arabinofuranosidases (EC 3.2.1.55), and β-xylosidases (EC 3.2.1.37). Endo-1,4-β-xylanases (also called xylanases, endoxylanases, 1,4-D-xylan-xylanohydrolases, endo-1,4-β-D-xylanases, β-1,4-xylanases, and β-xylanases) belong to the glycosil hydrolase family. They catalyze the hydrolysis of 1,4-glycosidic linkages between xylose residues in the backbone of xylans [78]. Since xylan is the major part of hemicellulose, xylanase is the key enzyme for depolymerization of hemicellulose components [34][79]. For complete hydrolysis of xylan to be achieved, the following enzymes are required: α-arabinofuranosidase, α-glucuronidase, acetylxylan esterase, and hydroxycinnamic acid esterase split side residues from the xylan backbone. Xylanases find their application in the food industry (brewing, wine production, juice clarification, baking), textile industry, and bioremediation [72][73][78]. Additionally, they can be applied in the pulp and paper industry, which results in a reduced amount of chlorine and chlorine dioxide commonly used for bleaching paper pulp. The most important producers of xylanases are given in **Table 1**.

Tomato pomace is a waste available in large quantities, and its chemical composition contains proteins, lipids, carbohydrates, amino acids, carotenoids, and minerals. Umsza-Guez et al. [74] used this waste as a substrate in SSF for xylanase production. Fermentation was carried out in a conical flask and a laboratory scale plate-type SSF reactor by *A. awamori*. In conical flasks, the maximum activity of xylanase was reached between the fourth and eighth day of fermentation (about 100 IU/g_{ds}), while in the plate-type SSF reactor, the maximum activity was reached on the fifth day of fermentation (195.92 ± 11.0 IU/g_{ds}).

Some studies have shown that co-cultivation of compatible microorganisms can enhance enzyme biosynthesis. Gupta et al. [80] studied the co-cultivation of SSF bacteria (*Bacillus* sp. and *B. halodurans* FNP135) producing xylanase and laccase. They used wheat bran as substrate. Under optimized conditions (pH 10.5, inoculum size 10+10%, moisture/substrate ratio 0.8:1), a significant increase in the production of xylanase (1685 IU/g) and laccase (2270 IU/g) was obtained. The mixed enzyme preparation was found to be effective in bio-bleaching of craft pulp.

Some researchers are concerned with the purification and characterization of the enzymes produced, as this is an important step that provides insight into enzyme properties and helps determine potential applications. David et al. [81] optimized the production of mannanase and protease using *Bacillus nealsonii* under SSF conditions on wheat bran as substrate. The protease was purified by standard protein purification procedures that include methods such as ammonium sulfate precipitation, gel filtration chromatography, and ion exchange chromatography. Each step was performed at 4 °C, and enzyme volume, protease activity, and protein content were determined after each step. The combination of mannanase and protease from *B. nealsonii* was found to be effective in removing various stains when used as detergent additive.

5. Production of Phenolic Compounds and Other Value-Added Compounds

AFIRs are rich in nutrients and bioactive compounds. Therefore, these residues have potential applications in SSF processes for the obtainment of beneficial compounds such as phenolic compounds, organic acids, flavor, and aroma compounds, which possess antioxidative, anti-inflammatory, antiallergic, antiviral, anticancer, antimicrobial, and antimutagenic properties [2]. A trend is to enrich food products with AFIRs, primarily because of the high content of dietary fibers and bioactive polyphenolic compounds, which increase the nutritional value and help in diseases prevention, but also positively affect stability, organoleptic properties, and technological properties of the final product [82].

Phenolic compounds represent the important group of bioactive compounds from plant material. They are the most abundant antioxidants in the human diet. Their structure consists of an aromatic ring, containing one or more hydroxyl substituents. The number and position of the hydroxyl groups, and the nature of substituents on the aromatic rings, affect the physiological properties of phenolic compounds. These compounds show a broad spectrum of physiological properties, such as anti-allergenic, anti-artherogenic, anti-inflammatory, anti-microbial, antioxidant, anti-thrombotic, cardioprotective, and vasodilatory effects [83]. Generally, they can be divided into three main groups, namely, phenolic acids (hydroxycinnamic acids, hydroxybenzoic acids), flavonoids (flavones, flavonols, flavanols, anthocyanins), and tannins (hydrolysable and nonhydrolyzable or condensed tannins) [84]. AFIRs are a cheap and rich source of potentially functional ingredients, such as phenolic compounds, thus promoting a circular economy concept. For example, after the processing of apples, it is estimated that 82–99% of the original polyphenols remain in apple pomace [85].

Lignin fraction of AFIRs contains various phenolic compounds, mainly phenolic acids such as ferulic, *p*-coumaric, syringic, vanillic, and *p*-hydroxybenzoic [86]. Simple phenolic compounds from biological materials can usually be isolated by extraction with organic solvents, while non-extractable highly polymerized proanthocyanidins and phenol complexes with proteins, fibers, and polysaccharides have to be hydrolyzed or degraded beforehand. The methods used for this are acid hydrolysis, which is environmentally unacceptable, and enzymatic hydrolysis, which is economically inconvenient [56]. On the other hand, phenolic compounds can be recovered by SSF, during which the microorganisms synthesize enzymes involved in breakdown of complex lignocellulosic material and release of these valuable compounds [11] (**Table 2**).

Table 2. Production of phenolic compounds from some food industry residues by SSF.

Products	Conditions	Remarks	Reference
Total polyphenolic compounds from apple pomace	Substrate: apple pomace, treated with inducers: copper sulphate (2 mM), veratryl alcohol (2 mM) and Tween-80 (0.1%); pH 4.5; autoclaved (121 °C, 30 min), moisture content 72% *w*/*v*. Microorganism: *P. chrysosporium*, inoculation with spore suspension (2.5×10^6 spores/g of solid). SSF: carried out in flasks, in controlled environment at 37 ± 1 °C for 14 days. Extraction (optimization): - type: UAE (in ultrasonication bath,) or MAE (in sealed green chem Teflon reactor vessel, pressure of 692 kPa, power 400 W). - solvent: water, or 60%, 70%, or 80% ethanol; acetone; or methanol. - temperature: 30, 40, 50, 60, 70, or 80 °C. - interval: 20, 30, or 40 min (UAE); 5, 10, or 15 min (MAE). - effect of surfactant: different concentrations of Tween-20 (0.1%, 1%, 2%, and 5% in *v*/*v* with water).	The phenol content was higher in the fermented apple pomace, and the antioxidant activity correlated with the increase in polyphenol content, with both values depending on the type of solvent, extraction temperature, extraction time, and method used.	[87]

Products	Conditions	Remarks	Reference
	After the extraction, sample mixture was centrifuged at 9268× *g* for 20 min to obtain the supernatant for further determination of total phenolic content (at 725 nm) and free radical scavenging activity (DPPH method at 517 nm).		
Individual polyphenolic compound from grape pomace	Substrate: corn silage, particle size 1.0–2.0 cm; autoclaved (121 °C, 20 min). Microorganism: *T. versicolor* TV-6, cultivated on PDA medium for 7 days at 27 °C; five mycelial plugs (diameter 1 cm) suspended in 10 cm^3 of sterile water (inoculum). SSF: performed in laboratory jars at 27 °C for 5, 9, 13, and 20 days. Extraction: milled dry substrate after SSF was extracted by 50% ethanol with solid/liquid ratio 1:40, in a shaking-water bath at 80 °C by (200 rpm) for 120 min. After the extraction, samples were centrifuged for 10 min at 10,000× *g* in order to obtain liquid extracts for further UHPLC analysis of phenolic acids.	After 20 days of corn silage treatment with *T. versicolor*, 10.4-, 3.4-, 3.0-, and 1.8-fold increments in extraction yield of syringic acid, vanillic acid, *p*-hydroxybenzoic acid, and caffeic acid, respectively, were reached.	[56]
Phenolic antioxidants	Substrate: grape waste, dehydrated at 60 °C/24 h, pulverized (30-mesh), stored at 22 °C.	The extracts of grape waste enhanced their free radical scavenging and	[88]

Products	Conditions	Remarks	Reference
from grape waste	Microorganism: different fungal strains: *A. niger* GH1, PSH, Aa-20, ESH; *Penicillium pinophilum* ESH2, ESH3; *Penicillium purpurogenum* GH2; inoculation with 2×10^7 fungal spores per gram of solid support. SSF: performed in tray reactor at 30 °C/60 h. Assay: total antioxidant activity of the extracts was tested by two different free radical (DPPH· and ABTS·+) inhibitions; free gallic acid content was estimated by HPLC.	preserved the capacity to avoid the lipid peroxidation after SSF. Gallic acid is not the only phenolic compound related to the free radical scavenging and antioxidant properties of the fermented samples.	
Phenolic antioxidants from pomegranate peels	Substrate: pomegranate peels, cleaned, dried at 60 °C/48 h, pulverized, stored at room temperature in black bags. Microorganism: *A. niger* GH1; inoculation with 2×10^7 spores/g of plant material, or substrate impregnated with culture broth. SSF: carried out in flasks at 30 °C for 96 h. Assay: tannins were analyzed using a spectrophometric method; concentration of gallic and ellagic acids was determined by HPLC.	The ellagic acid was accumulated considerably in pomegranate peels after fungal fermentation, which demonstrated that the high level of hydrolysable tannins in pomegrante peel tannins are mainly ellagitannins.	[89]
Phenolic antioxidants	Substrate: chokeberry (cultivar “Nero”) pomace, dried < 40 °C,	The extractable phenolics increased more than 1.7-fold during both fermentation	[90]

Products	Conditions	Remarks	Reference
from chokeberry pomace	ground (0.5–1 mm), stored at 18 °C; moisturized (65%) with a nutrient solution (containing yeast extract and glucose), pH 5.5; autoclaved at 121 °C/30 min. Microorganism: *A. niger* ATCC-6275 and *R. oligosporus* ATCC-22959; inoculating cultures were produced by growing the strains on fresh PDA at 27 °C for 10 days, and spore inoculum was prepared by washing the agar surface with sterile distilled water. SSF: was carried out in in Erlenmeyer flasks at 30 °C for 12 days; substrate was inoculated with spore suspension 2×10^7 spores/g of solid. Extraction: in an ultrasonic bath for 30 min at 40 °C with solvent mixture (hydrochloric acid: methanol: water in the ratio 1: 80: 19). The mixtures were centrifuged (4000× *g* for 10 min); supernatants were filtered and evaporated under vacuum and then stored in methanol (4 °C) until analysis (total phenolics, flavonoids, and anthocyanins; individual phenolics; antioxidant activities).	processes, and a similar trend was observed for total flavonoids. The free radical scavenging ability of phenolic extracts were significantly enhanced during the SSFs. The amounts of flavonols and cinnamic acids increased while the concentrations of glycosylated anthocyanins decreased substantially.	

Products	Conditions	Remarks	Reference
Water-soluble phenolic antioxidants from cranberry pomace	Substrate: freshly pressed cranberry pomace, vacuum-dried and stored in a refrigerator. Microorganism: *Lentinus edodes* was maintained on PDA slants and Petri plates at 4 °C and sub-cultured. The fungus was resuscitated by transferring onto a PDA plate and cultured at room temperature 20 days before use. SSF: carried out in in Erlenmeyer flasks at 28 °C for 25 days (cranberry pomace + calcium carbonate + water + ammonium nitrate or fish protein hydrolysate was autoclaved at 121 °C for 20 min and the vegetative mycelia from one PDA plate were inoculated into flasks). Extraction: distilled water or 95% ethanol was added to fungus–pomace flask and the culture was homogenized for 1 min and then centrifuged at 15,000× *g* at 4 °C for 20 min and then filtered.	There was an increase in the extractable phenolic content. Both phenolics and antioxidant capacity correlated with the increase in the β-glucosidase activity, showing that the enzyme may play an important role in the release of phenolic aglycones from cranberry pomace and, therefore, increase the antioxidant capacity.	[76]

UAE—ultrasonic-assisted extraction; MAE—microwave-assisted extraction; PDA—potato dextrose agar; DPPH—1,1-diphenyl-2-picrylhydrazyl radical; ABTS·+—2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) radical; UHPLC—ultra-high-performance liquid chromatography; HPLC—high-performance liquid chromatography.

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