Valorization of Non-Edible Oilseed Residues

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The sustainable development of biodiesel and oleochemical industries requires optimal recycling and reuse strategies for all the generated residues and by-products. The main residues from non-edible oilseeds are either lignocellulosic materials, such as fruit shells, pods, hulls, branches, and leaves, generated before oil extraction or a protein-rich material, e.g., the press cake or de-oiled meal, generated after oil extraction. Both lignocellulosic- and protein-rich materials have huge economic potential. However, since using non-edible oils for biodiesel production is still emerging, the valorization of non-edible oilseed residues is still underdeveloped compared to that of edible oil production residues. The utilization potential of non-edible oilseed residues goes far beyond the traditional energetic approaches. Thermochemical, biochemical, physico-chemical, and chemical approaches provide different utilization routes. Thermochemical approaches, such as gasification and pyrolysis, result in syngas, biochar, and biooil, which can then be converted into advanced biofuels or serve as raw materials for the chemical industry. In the biochemical conversion approach, by either anaerobic digestion, sugar-platform processes, or solid-state fermentation, microorganisms convert the starting substrates into gaseous or liquid biofuels, enzymes, or other compounds.

Keywords: non-edible oils ; bioconversion ; pretreatment ; anaerobic digestion ; enzymatic saccharification

1. Bioconversion Processes for Valorization of Non-Edible Oilseed Residues

Biochemical conversion can be used for valorizing different agro-based bioresources, including non-edible oilseed residues. Bioconversion processes consist of deconstructing the complex structure of plant biomass by enzymes or microorganisms into simpler compounds that are further processed by microbial fermentation or chemical conversion. Overall, bioconversion processes include several connected steps operating at room temperature and atmospheric pressure. Bioconversion might also include preparatory steps at high temperatures and pressure and using chemicals. Careful optimization of each step is required to achieve efficient and cost-effective conversion of bioresources into valuable products.

In anaerobic digestion (AD), microorganisms break down biomass materials in the absence of oxygen by a sequence of hydrolysis, acidogenesis, acetogenesis, and methanogenesis processes (**Figure 1**). The AD results in a methane-rich gas mixture known as biogas and a nitrogen-rich wet slurry known as digestate. The high calorific value of methane makes biogas a valuable fuel, which can be transformed into electricity and heat, used in domestic applications [1], or upgraded to transportation fuel [2]. The AD digestate, due to its high nitrogen content, can be used as a biofertilizer, soil conditioner, or a source for the recovery of nutrients [3].

In sugar-platform conversion (**Figure 1**), saccharification, often referred to as hydrolysis, is applied to generate sugars from the biomass polysaccharides, i.e., cellulose and hemicelluloses. The sugar-platform processes include four main steps, namely pretreatment, saccharification, fermentation, and product recovery. A pretreatment is usually required as the first step for ensuring efficient saccharification. Pretreatment, typically performed with heat, chemicals, or enzymes, removes lignin and/or hemicelluloses and enhances the accessibility of cellulose to enzymes and microorganisms [4]. After pretreatment, the pretreated materials are subjected to saccharification, performed by enzyme consortia or chemicals [5]. Enzyme preparations containing cellulases and hemicellulases of fungal origin are commonly used. Saccharification breaks down cellulose and hemicelluloses into simple sugars transferred into the liquid phase, i.e., the hydrolysate. Lignin remains relatively untouched by the saccharifying agents and is separated from the hydrolysate by filtration. When the saccharification is completed, the generated sugars are used as substrates for microbial fermentations resulting in valuable products, such as biofuels, biomaterials, and platform chemicals. Ethanol is a common fermentation product. Yeasts, e.g., *Saccharomyces cerevisiae*, or bacteria, e.g., *Zymomonas mobilis*, are ethanol-producing microorganisms [6]. Other fermentation products are lactic acid and biobutanol, produced via LAB (lactic acid bacteria) and ABE (acetone-butanol-ethanol) fermentation, respectively. The saccharification and fermentation steps can be integrated into different

configurations, such as separate hydrolysis and fermentation (SHF), simultaneous saccharification and fermentation (SiSF) [7], and consolidated bioprocessing [8]. In the recovery step, the products are isolated from the fermentation broth by separation techniques, such as filtration, distillation, or centrifugation, and purified until reaching the quality standards required for commercial use.

Another bioconversion approach produces enzymes (**Figure 1**) by cultivating enzyme-producing microorganisms directly on biomass residues [9]. Solid-state fermentation (SSF) techniques with various microorganisms are used [10].

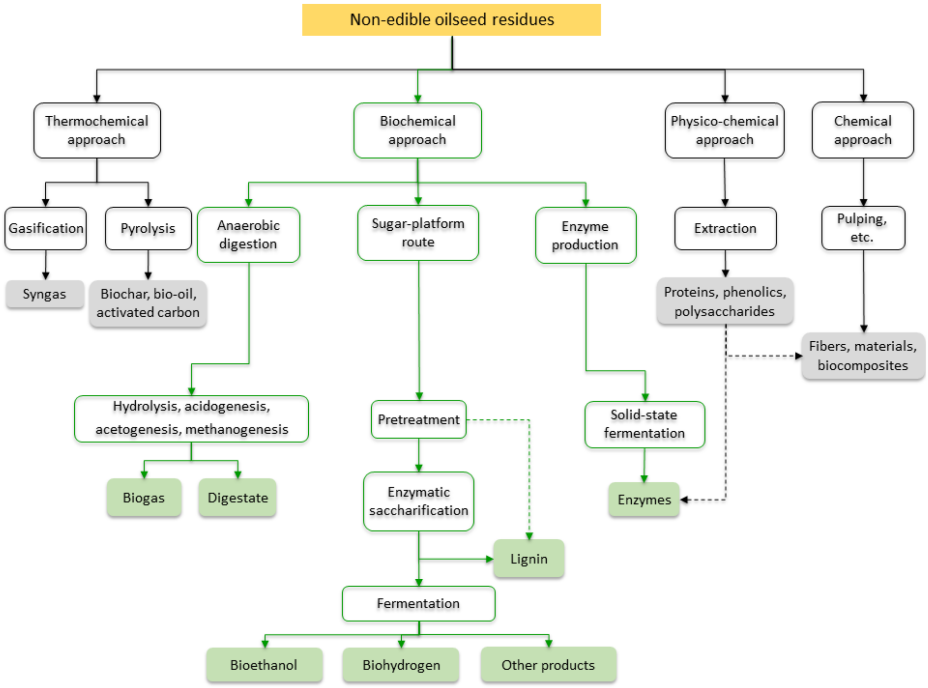

Figure 1. General scheme of valorization approaches for non-edible oilseed residues. Bioconversion routes are shown with green lines. Bioconversion products are in green blocks.

The bioconversion processes applied to non-edible oilseed residues include primarily anaerobic digestion of press cakes, sugar-platform processes of lignocellulosic streams, i.e., hulls, shells, pods, pruning residues, and production of enzymes by SSF of press cakes. The bioconversion approach for non-edible oilseed residues should be selected to fit the characteristics of each addressed material well. It would be unrealistic to expect high biogas yields from highly-recalcitrant lignocellulosic residues or high sugar yields from press cakes with low carbohydrate content.

1.1. Anaerobic Digestion

Due to the increasing interest in non-edible oils for biodiesel production, the generation of press cakes and other related residues is continuously increasing. The main use of the cakes from the extraction of edible oils is as cattle feed [11]. However, cakes from non-edible oilseeds are unsuitable for feed because they contain toxins, e.g., phorbol esters (jatropha), ricin (castor), or chromenoflavones (karanja), or have strong odors or other anti-nutritional factors [1]. Those barriers and the need to find economic uses for the escalating amounts of non-edible oil cakes have increased the emphasis on using them in anaerobic digestion for producing biogas.

Anaerobic digestion of press cakes is the most investigated application for non-edible oilseed residues. Some examples are shown in **Table 1**. Regardless, the research interest shown in the application of residues has, so far, been considerably lower than the interest shown in biodiesel production. An advanced *Web of Science* search for the three most investigated non-edible oilseed-bearing plants, using the query combination ((“jatropha” OR “castor” OR “karanja”) AND (“biogas” OR “anaerobic digestion”)) performed in April 2023 resulted in 135 hits, which is considerably lower than the around 5000 results for a related search for the same plant species using “biodiesel” instead of “biogas” OR “anaerobic digestion”.

Jatropha cakes account for most of the literature reports on the anaerobic digestion of non-edible oilseed residues. The first scientific article showing the suitability of jatropha press cake for biogas production was published in 1997 [12], and the second one came only in 2008 [13]. After that, the interest in the anaerobic digestion of jatropha press cakes continuously increased, as shown by the 38 scientific papers indexed in the *Web of Science* during the last five years.

Table 1. Examples of anaerobic digestion of press cakes and other residues of non-edible oilseeds.

Material	Conditions	Results	Ref.
Jatropha seed cake	AD of a 1:20 cake/water slurry in a 5-L batch reactor at 30 °C for 60 days.	Methane yield: 156 L/kg of seed cake; COD removal: 52%.	[14]
Jatropha seed cake	Semi-continuous flow at 30 °C; COD range: 1.25–5 kg/m^3 day.	Highest methane yield (340 L/kg COD degraded) was obtained at an OLR of 1.25 kg COD/m^3 day.	[15]
Jatropha seed cake	AD of cow dung alone and mixed with jatropha cake in 2-L plastic jars for 40 days.	Biogas yield of jatropha cake (0.170 m^3/kg) was higher than that of cow dung (0.166 m^3/kg). The digestate was a suitable fertilizer for maize and tomato.	[16]
Jatropha seed cake	Jatropha cake alone or combined with cattle dung, 37 °C, 5-L glass fermenter	Biogas yield: 265 L/kg biomass; methane concentration: 65%	[17]
Jatropha seed cake	Co-digestion of jatropha cake and cattle dung in a 6-m^3 floating-type digester for 60 days.	Methane concentration: 62.3–69.2% under mesophilic conditions and 65.2–69.2% for psychrophilic conditions.	[18]
Jatropha seed cake	Pilot-scale continuous 40-m^3 stirred digester; co-digestion with cow dung (3:1) for 120 days	Within 5 days, the reactor started producing 20 m^3 of biogas per day.	[19]
Jatropha seed cake	Co-digestion with sugarcane bagasse and addition of Fe^{2+} ions in 120-mL serum vials as digesters.	Co-digestion of jatropha cake (10% (*w*/*v*)) and bagasse (5% (*w*/*v*)) gave higher BPR than experiments with jatropha cake alone. Adding 10 mM of Fe^{2+} ions led to further improvement.	[20]
Jatropha seed cake	AD in the presence of an iron additive	H_2S content in biogas was reduced.	[21]
Jatropha and karanja cakes	AD in a 20 m^3/d floating drum under mesophilic temperature	Methane potential: 0.39 (for jatropha cake) and 0.43 m^3/kg TS (for karanja cake); average methane concentration: 66.6% (for jatropha) and 62.5% (for karanja); higher methane concentration than in biogas from cattle dung.	[22]
Jatropha and karanja cakes, pods, and glycerol	Serum glass bottles (125 mL) fitted with rubber airtight stoppers were used as digesters.	The biogas potential of residues of karanja and jatropha was, respectively, 3.07 and 1.83 m^3 per kg of produced biodiesel.	[23]
Karanja oil cake	Karanja cake mixed with cow dung in 75:25, 50:50, 25:75 and 0:100 (*w*/*w*) proportions	The 25:75 mixture gave the best results. Methane content was 73%, and the slurry had a higher fertilizer value.	[24]
Mahua and hingan cakes	A 20-L plastic bottle was used as single-phase digestion system	Biogas yield: 198–233 L/kg seedcake. The digestates had high fertilizer value due to high nitrogen content.	[25]
Castor cake	AD in 5-L capacity single-stage fermenters at 30 and 37 °C	Particle size 2.0–1.4 mm was favorable for BPR. High temperature resulted in higher yield. Conversion of the feed: 30–35% TS.	[26]
Castor cake, stem, and leaves	AD in 118-mL bottles	Seed cakes and leaves were suitable substrates for AD, but stems were unsuitable without pretreatment. The combined biogas yield from cake, stem, and leaves was 131 g/kg of initial plant biomass. Biodiesel yield is 155 g/kg, and ethanol yield is 85 g/kg.	[27]

AD, anaerobic digestion; COD, chemical oxygen demand; OLR, organic loading rate; BPR, biogas production rate; TS, total solids.

Sinbuathong et al. [14] showed that *J. curcas* seed cake is a good source of methane by anaerobic digestion. Methane yields of up to 156 L/kg cake can be achieved, and the optimal cake-to-water ratio is in the range of 1:10–1:20. The same group reported an evaluation of the effect of the organic loading rate (OLR) on biogas production during the AD of jatropha seed cakes in a semi-continuous flow at 30 °C [15]. The highest methane yield (340 L/kg COD degraded) was obtained at the OLR of 1.25 kg COD/m^3 day.

Raheman and Mondal [16] showed that maximum biogas production could be achieved by AD at a total solids load of 15–20% with C:N ratios between 22:1 and 27:1. The biogas yield of jatropha cake (0.170 m^3/kg) was higher than that of cow

dung (0.166 m^3/kg). The digestate of jatropha cake was an effective biofertilizer for improving the growth of maize and tomato.

Co-digestion of jatropha cake with cattle dung or other animal manure has often been reported. The AD of jatropha cake combined with cattle dung in a lab-scale fermenter operating at 37 °C resulted in 265 L/kg biomass with a methane concentration of around 65% [17]. AD in a 6-m^3 floating-type digester for 60 days resulted in a methane concentration of 62.3–69.2% under mesophilic conditions and 65.2–69.2% for psychrophilic conditions [18]. The study concluded that jatropha cake is a better solution for improving biogas quality and composition and getting a valuable digestate. Singhal et al. [19] designed a pilot-scale continuous stirred tank reactor for co-digesting jatropha de-oiled cake and cow dung. The reactor produced 20 m^3 of biogas daily during 120 days of continuous operation.

Co-digestion of jatropha cake with various plant residues, including press cakes of other oilseeds, has also been reported. Sen et al. [20] showed that co-digestion of jatropha cake with bagasse in the presence of a low amount of Fe^{2+} ions leads to high biogas yield within a short digestion time. Iron additives have also been used to reduce the H_2S content in the biogas and to facilitate the anaerobic digestion of jatropha cake [21].

Karanja is another non-edible oilseed thoroughly investigated as a biogas source. A total of 26 results were found in a *Web of Science* advanced search related to using karanja cakes for anaerobic digestion. Anaerobic digestion of press cakes and other karanja residues at different scales, both alone or combined with other materials, has been reported. An industrial-scale study revealed that biogas produced from jatropha and karanja cakes had a 15–20% higher methane content than biogas produced from cattle dung [22]. Khuntia et al. [23] assessed the biological methane potential of karanja and jatropha cakes and pods and that of the residual glycerol from biodiesel production. The study revealed that the biogas potential of the residues of karanja and jatropha is, respectively, 3.07 and 1.83 m^3 per kg of produced biodiesel. Barik and Murugan [24] reported the characterization of the biogas and the digestate resulting from the co-digestion of karanja cake and cattle dung. The biogas contained 73% methane, and the digestate showed good characteristics as a nontoxic and environmentally friendly biofertilizer.

Mahua cake also has a high biogas production potential. A 50:50 combination of hot water-detoxified mahua cake and cattle dung resulted in a biogas output of 442 L/kg of total solids (TS) with a methane concentration of 58.5–60% [28]. A lower output (198–233 L/kg) was reported for co-digestion of mahua and hingan press cakes. However, a concomitant production of digestates with high fertilizing value was achieved [25].

For the AD of castor oil cakes, the effect of operational factors on biogas production has been investigated [26]. Optimal particle size, temperature, loading rate, and stirring have been established. The yield of various biofuels from different castor streams was shown by Bateni et al. [27]. The study showed that 1 kg of castor plant could yield 155 g biodiesel from the oil and 131 g biogas or 85 g ethanol from the press cake, stem, and leaves.

The AD of other non-edible oilseed residues has also been investigated, although the number of reports is lower than for the above-discussed species. For example, the biogas potential of jojoba cake [29], neem leaf litter [30], and moringa leaves and branches [31] has been assessed.

Research results on the anaerobic digestion of some non-edible oilseeds, mainly jatropha, have already been developed to pilot scale [19]. Some projects on incorporating jatropha-derived biogas into the energy matrix in rural areas for providing different energy services [32] and biofuel-based decentralized power [33] have been successfully implemented.

1.2. Sugar-Platform Processes

In the sugar-platform conversion processes, the sugars generated by saccharification are used by microorganisms, e.g., bacteria, fungi, or yeasts, to yield various useful products, e.g., ethanol, lactic acid, hydrogen, or butanol. Lignin, either generated as a saccharification residue or separated during pretreatment (**Figure 1**), can be upgraded to novel materials, diesel-like advanced biofuels, or commercially relevant chemicals [34].

The interest in saccharification of non-edible oilseed residues has so far been low, as indicated by the number of indexed articles (59) in the *Web of Science*, which is considerably lower than the number of anaerobic digestion-related papers (135) for the three most relevant species (jatropha, castor, and karanja). Despite the low number of published reports, sugar-platform processing is a relevant bioconversion route for valorizing residues of non-edible oilseeds. Jatropha residues, including shells [35], fruit hulls [36], husks [37], press cakes [38], and de-oiled waste [39], are the most investigated materials in the studies reported in the literature. Other substrates, such as castor plant residues [40] and press cakes [41],

karanja defatted kernel [42] and hull [43], moringa empty pods [44], stems and branches [45], and bladderpod press cakes [46], have also been investigated.

Some studies on the sugar-platform conversion of non-edible oilseed residues focus production of sugars [35] without stressing a specific end product to be obtained from the sugars. Other studies are focused on producing biofuels, such as cellulosic ethanol [47] and hydrogen [48]. Both ethanol and hydrogen are produced by fermentation of the sugars resulting from saccharification of the lignocellulosic parts of the residues. Itaconic acid, succinic acid, butanol, 2,3-butanediol, and lignin are other products that can be produced from residues of non-edible oilseeds following sugar-platform conversion. Hydrolysates of bladderpod press cakes have been investigated for microbial fermentations for producing succinic acid [46] and butanol [49]. Production of itaconic and succinic acids has been reported by fermentation of hydrolysates of jatropha press cakes [50]. Hydrolysates of jatropha hulls were suitable for producing 2,3-butanediol [51]. High recovery of lignin was reported by alkaline processing of jatropha press cake [52]. The recovered lignin was characterized using ^{1}H NMR, FTIR, and nitrobenzene oxidation.

Most reports on producing sugars and ethanol from cellulose contained in residues of non-edible oilseeds use enzymatic saccharification, but some studies apply acid hydrolysis. Muktkham et al. [53] investigated the effects of various acids at different concentrations on the formation of glucose from karanja seed residues. Among the investigated acids, HCl led to the highest glucose formation (173.4 g/kg seed residue). The fermentation of the produced hydrolysate with *Saccharomyces cerevisiae* gave 88.6 g ethanol per kg of initial biomass. In another approach, karanja seed cake was extracted with ethanol, and the extractive-free material was submitted to acid hydrolysis with H_2SO_4 [54]. Optimization of the operational conditions for maximizing sugar release revealed that the maximum glucose formation (245 g/kg of extractive-free cake) could be obtained for hydrolysis at 120 °C, with 7.5% H_2SO_4, for 1 h, and with a liquid-to-solid ratio of 15. García et al. [37] investigated the dilute-sulfuric acid hydrolysis of the xylan fraction of a mixture of jatropha shells and husks under H_2SO_4 concentrations in the range between 0.5 to 4.5% at 170–220 °C and for 10–20 min. Low H_2SO_4 concentrations, low temperatures, and reaction times below 10 min favored xylan hydrolytic conversion and minimized xylose degradation.

1.3. Production of Enzymes from Residues of Non-Edible Oilseeds

Since press cakes are rich in C and N in the form of proteins and carbohydrates [55], they are suitable substrates for enzyme-secreting microorganisms. Producing enzymes of industrial importance using edible oilseed residues has been reported in several studies [56], while production from non-edible oil residues has been less investigated. However, with the increase in the relevance of non-edible oils for biodiesel production, the interest in valorizing their residues for enzyme production has also increased.

Table 2 summarizes studies published over the last 15 years using non-edible oilseed residues as substrates for enzyme production. The table gives the used substrates, cultivated microorganisms, the produced enzymes, and their anticipated applications. Press cakes of jatropha [57] and castor bean [58] have attracted the most research interest. Jatropha husks [59] and press cakes of other species, such as jojoba [58], karanja [42], moringa [60], and mahua [9], have also been the object of study.

Table 2. Examples of enzyme production from residues of non-edible oilseed species.

Source	Microorganism(s)	Enzyme(s)	Application	Ref.
Jatropha seed cake	***Pseudomonas aeruginosa***	**Protease, lipase**	**Industrial enzyme production**	[61]
	Aspergillus niger*, *Rhizomucor miehei	**Lipase**	**Enzyme production**	[57]
	Paecilomyces variotii	**Cellulases**	**Biofuel production**	[9]
	Scytadilium thermophilum	**Xylanase**	**Biobleaching of paper pulp**	[62]
	Thermoascus aurantiacus	**Cellulases**	**Saccharification of sugarcane bagasse**	[63]
	A. niger	**Cellulase, xylanase**	**Biofuel production**	[64]
Jatropha seed husk	***Bjerkandera adusta*** ***Pycnoporus sanguineus***	**Cellulases, xylanases**	**Screening of inducible enzyme activity on lignocellulosic residues**	[59]

Source	Microorganism(s)	Enzyme(s)	Application	Ref.
Castor bean waste	*Penicillium simplicissimum*	Lipases	Ricin detoxification and biodiesel enzyme production	[65]
	Penicillium simplicissimum	Lipases	Biodiesel enzyme production	[66]
	Aspergillus spp., *Emericela* spp., *Rhodotorula* spp.	CMCase, FPase, β-glucosidase	Screening of fungal isolates for cellulase activity	[67]
	Pa. varoitii	Tannase, phytase	Ricin detoxification, phytate phosphate release	[68]
Jojoba meal	*Aspergillus* spp.	Extracellular β-glucosidase	Biofuel production, fortification of *T. reesei* cellulases	[58]
Karanja seed residue	*Spingomonas echinoides, Iprex lacteus*	Endo- and exoglucanases, xylanase, laccase	Biofuel production	[42]
	A. niger, Bacillus licheniformis, Acinetobacter pittii	Proteases	Enzyme production, gelatin film breakdown	[69]
Moringa straw	*Penicillium funiculosum, Fusarium verticillioides, Cladosporium cladosporoides*	CMCase, FPase, β-glucosidase	Screening of fungal isolates for cellulase activity	[60]
Mahua seed cake	*A. niger*	Proteases	ANF detoxification	[9]

Filamentous fungi are the typical microorganisms used for enzyme production on agro-industrial wastes, as they are heterotrophic decomposers that grow readily on the surface of organic material under suitable moisture and temperature conditions. For example, *Aspergillus* spp. strains are well-known producers of several hydrolytic enzymes [70]. In this context, they secrete lipases [57], proteases [69], and β-glucosidase [58]. The mesophilic fungus *Penicillium simplicissimum*, a producer of many secondary metabolites, has also been investigated for detoxifying castor press cakes [65] and producing lipase [66]. Furthermore, the fungi *Scytadilium thermophilum* [62], *Thermosaceus aurantiacus* [63], and *Paecilomyces variotii* [9] were cultivated on non-edible oilseed residues for their ability to produce different lignocellulolytic enzymes. Apart from the valorizing aspect, non-edible oilseed residues were in some studies primarily only used as a cellulose substrate to induce lignocellulolytic enzymes from fungal isolates, including white-rot fungi [60], to characterize their saccharification potential [59]. Hydrolytic enzymes may also be produced by bacteria, such as *Pseudomonas aeruginosa* [61], *Spingomonas echinoides* [42], *Bacillus licheniformis*, and *Acinetobacter pittii* [69], but that is less frequently reported.

Notably, fungal fermentation can offer a value-added effect for castor residue. After the oil recovery, the highly toxic protein ricin remains in the press cake, making it unsuitable as animal feed. Biodetoxification of ricin was reported in two cases by the coproduction of lipase [65] and tannase and phytase [68]. However, none of these enzymes are proteolytic; thus, the degradation of the proteinaceous toxin must have been caused by proteases that were also secreted during fungal cultivation. In both cases, complete ricin removal after three days was detected by SDS gel electrophoresis [68] and gel filtration chromatography [65].

An overview of the production of hydrolytic enzymes by solid-state fermentation (SSF) of various fungi on non-edible oilseed residues is presented in **Table 3**. In SSF, which is carried out in static mode, the moldlike growth on a solid substrate essentially requires the absence of free water. The moisture content should not exceed the maximum water retention capacity of the particulate matter but just enough to keep it moist [9]. Too much water reduces the particle porosity and the microbial respiration and thus reduces substrate digestion and stimulates aerial growth of mycelia. Typically, the moisture level in SSF is about fifty percent, obtained by mixing sterilized particulate solids with a minimum volume of liquid and the inoculum culture. In the SSF studies presented in **Table 3**, the fermentation processes ensued for at least two days but continued up to 7–9 days. Maximum enzyme yield usually peaks after 3–5 days and then drops on prolonged cultivation, likely due to inactivation or degradation [9][61][62][64][66].

Table 3. Overview of enzymes produced by solid-state fermentation (SSF) on non-edible oilseed residues. Enzyme classification numbers are provided where applicable.

Enzyme	Microorganism	Substrate	SSF Length	Max. Activity (U/g Substrate)	Ref.
Lipase (EC 3.1.1.3)	*P. aeruginosa*	Jatropha seed cake	120 h	620	[61]
	P. simplicissimum	Castor cake	96 h	44.8	[65]
	P. simplicissimum	Castor cake	120 h	155	[66]
Tannase (EC 3.1.1.20)	*Pa. varoitii*	Castor cake	48 h	2600	[67]
Phytase (EC 3.1.3.8/.26)	*Pa. varoitii*	Castor cake	72 h	260	[68]
Cellulase (FPase [1]) (EC 3.2.x.x)	*Th. aurantiacus*	Jatropha seed cake	6 days	4.9	[63]
	Pa. variotii	Jatropha seed cake	4 days	27.3	[9]
Endoglucanse (CMCase [2]) (EC 3.2.1.4)	*Th. aurantiacus*	Jatropha seed cake	6 days	124.4	[63]
	Aspergillus niger FGSCA733	Jatropha seed cake	120 h	3974	[63]
	Spingomonas echinoides	Karanja seed residue	8 days	16.2	[42]
	Iprex lacteus	Karanja seed residue	8 days	49.2	[42]
Exoglucanase (EC 3.2.1.9)	*S. echinoides*	Karanja seed residue	8 days	23.4	[42]
	Iprex lacteus	Karanja seed residue	8 days	31.2	[42]
β-glucosidase (EC 3.2.1.21)	*Th. aurantiacus*	Jatropha seed cake	6 days	28.9	[63]
	Aspergillus sp. *DHE7*	Jojoba meal	72 h	153	[71]
Xylanase (EC 3.2.1.8)	*Scytadilium thermophilum*	Jatropha seed cake	9 days	1455	[62]
	A. niger FGSCA733	Jatropha seed cake	48 h	6087	[63]
	S. echinoides	Karanja seed residue	8 days	4.8	[42]
	I. lacteus	Karanja seed residue	8 days	16.2	[42]
	P. aeruginosa PseA	Jatropha seed cake	72 h	1800	[61]
Protease (EC 3.4.x.x)	*A. niger*	Mahua deoiled seed cake	2 days	52.5	[9]
	A. niger	Karanja seed residue	7 days	3.7	[69]
	Acinetobacter pittii	Karanja seed residue	7 days	1.8	[69]
	B. licheniformis	Karanja seed residue	48 h	2.1	[69]

[1] Filter paper as substrate; [2] carboxymethyl cellulose as substrate.

Given the concern about the high cost of commercial enzymes needed for bioethanol production, many studies using SSF have focused on expressing lignocellulolytic enzymes [42]. Cellulolytic enzymes comprise endoglucanase, detected using carboxymethyl cellulose as a substrate (i.e., CMCase), exoglucanase [63], and β-glucosidase [58]. Hemicellulases, such as xylanases, are also included in many studies [62]. The total cellulase activity can be assessed by the digestion of filter paper (i.e., FPase). Radhakumari et al. [42] also observed the activity of lignin-degrading laccases from *S. echinoides* grown on karanya seed residue but at far lower levels than the cellulase activities.

Although standard enzyme assays are used, the reported activity yields of the same enzymes vary greatly and thus are difficult to compare (**Table 3**). For example, CMCase, xylanase, and protease activities vary over three orders of magnitude. The differences can arise biologically from the type of microorganism and substrate used, or they can be of technical origin related to the SSF methodology and analytical performance. The enzyme expression levels, though, are not economically sustainable compared to commercial production. When optimizing an SSF process, the moisture content, pH, nutrient supplements, and inoculum size are critical factors. Thus, approaches for maximizing the enzyme yields varying such factors are reported for lipase from *P. simplicissimum* [66], tannase and phytase from *Pa. variotii* [68], β-glucosidase from *Aspergillus* sp. [58], and cellulase from *T. aurantiacus* [63].

2. Other Valorization Routes for Non-Edible Oilseed Residues

Production of bio-oils, biochar, and activated carbon by thermochemical conversion is a valorization approach for non-edible oilseed residues. There is documented research on the thermochemical conversion of jatropha de-oiled cake [72] and husks [73], castor husks [74], stems and leaves [75], and residues of other non-edible oilseeds. Pyrolysis of jatropha press cake has been reported to result in bio-oil and biochar yields of up to 45 and 36% (*w*/*w*), respectively [76]. Gasification of jatropha and moringa husks can cover the energy needs of a biodiesel facility with a capacity of 800 L/day, as shown in a simulation study reported by Pfeil et al. [73]. The effect of alkaline pretreatment on hydrothermal liquefaction (HTL) of castor stems and leaves was reported by Kaur et al. [75]. The study included a thorough characterization of the HTL products (bio-oil and biochar), the determination of their maximum heating value, including an assessment of the application potential of the phenolic compounds contained in the produced bio-oil. Pyrolysis of rubber-seed shells results in high yields of activated carbons displaying high specific area and other properties as adsorbents [77]. Pyrolysis of tobacco seed cakes resulted in biochar with high potential as an adsorbent and soil amendment [78]. Neem bark has been reported for bio-oil production using pyrolysis [79].

Press cakes of castor, jatropha, karanja, and neem as well as castor stems and moringa empty pods have been investigated for biocomposites and other material applications. Cellulose fibers produced from castor stems with an alkaline pulping process exhibit good properties for composites and textile applications [80]. Castor press cake is also useful for producing green composites by combining it with wood nanocellulose [81]. The production of an eco-friendly polymeric resin from jatropha cake reinforced with microfibrillated cellulose has been reported [82]. Patil et al. [83] reported using karanja cake for developing green resins with modified sisal fibers. The produced composites exhibit improved tensile properties compared with those made with as-received sisal fibers. Cellulose extracted from neem press cake was shown to be suitable to be incorporated as a biofiller in polymer matrices for manufacturing eco-friendly composites [84]. Cellulose nanofibers prepared by acid hydrolysis of moringa empty pods were shown to be a good natural reinforcing material for fiber-reinforced polymer composites [85].

The production of particleboards from castor stalks and jatropha press cakes has also been investigated. Grigoriou and Ntalos [86] mixed chipped castor stalks with industrial wood particles to produce the middle layer of three-layer particleboards. The produced materials meet most of the relevant European and American standard requirements for interior boards. Evon et al. [87] manufactured renewable and biodegradable particleboards by thermo-pressing jatropha press cakes. The assessment of the mechanical properties revealed that the particleboards are suitable for being used as an interlayer sheet for pallets, furniture, or building materials.

Following a lignin-first strategy, valuable products can be obtained from lignocellulosic residues of some oilseeds. A recently discovered “ideal lignin”, which is a benzodioxane homopolymer termed catechyl lignin (C-lignin), can be extracted from jatropha and castor seed coats [88] and from candlenut shells [89]. Liu et al. [88] applied catalytic hydrogenolysis in deep eutectic solvents to castor seed husks for extracting C-lignin and depolymerizing it to catechol.

Press cakes can be sources for the extraction of protein and bioactive compounds. Protein yields between 53 and 82% have been reported after extraction and recovery from jatropha cake [90]. Jatropha press cake protein is of interest for non-food uses, e.g., in producing coatings and adhesives. Biswal et al. [91] found that proteins from mahua de-oiled cake display comparable functional properties to proteins from other plants. Protein from de-oiled karanja cake was shown to be suitable for fabricating low-cost, fully “green” biocomposites [83]. The potential of a protein extract from moringa seed residue for inducing the separation of microalgae from their aqueous medium has been shown [92]. Microwave-assisted extraction of proteins and polyphenols with antioxidant activity from jojoba seed cake was reported [93]. Polyphenols and flavonoids obtained from jatropha de-oiled meal exhibited antioxidant activities comparable to that of β-carotene [94].

Some of the valorization routes mentioned above can be applied to bioconversion residues. For example, saccharification residues and the spent substrate after enzyme production by SSF can be upgraded by thermochemical-conversion technologies or used for manufacturing particleboards, composites, and other materials.

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