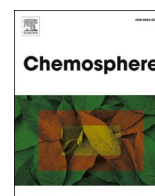




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Agricultural waste biomass for sustainable bioenergy production: Feedstock, characterization and pre-treatment methodologies

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HIGHLIGHTS

- A promising route for valorizing agro waste biomass is bioenergy.
- Feedstock analysis, usage, and pre-treatment methods have been assessed.
- Pre-treatment of agricultural trash to boost bioenergy output was brought under light.
- Healthy strategies to promote bioenergy and address ecological issues were discussed.

GRAPHICAL ABSTRACT



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ABSTRACT

The worldwide trend in energy production is moving toward circular economy systems and sustainable availability of sources. Some advanced methods support the economic development of energy production by the utilization of waste biomass, while limiting ecological effects. The use of agro waste biomass is viewed as a major alternative energy source that expressively lowers greenhouse gas emissions. Agricultural residues produced as wastes after each step of agricultural production are used as sustainable biomass assets for bioenergy production. Nevertheless, agro waste biomass needs to go through a few cyclic changes, among which biomass pre-treatment contributes to the removal of lignin and has a significant role in the efficiency and yield of bioenergy production. As a result of rapid innovation in the utilization of agro waste for biomass-derived bioenergy, a comprehensive overview of the thrilling highlights and necessary advancements, in addition to a detailed analysis of feedstock, characterization, bioconversion, and contemporary pre-treatment procedures, appear to be vital. To this end, the

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current status in the generation of bioenergy from agro biomass through various pre-treatment procedures was examined in this study, along with presenting relevant challenges and a perspective for future investigations.

1. Introduction

Nobody is questioning the fact that energy is of the most important parameters for global prosperity. Interconnection of rapid population expansion and advancement in industrialization and modernization has led to a hike in the demand of energy in recent years (Paramati et al., 2022). Therefore, the depletion of conventional resources such as oil, natural gas and coal have started and, on the other hand, a drastic ecological pollution, specifically the pollution of air and water resources, has been resulted, along with increase in global temperature and ozone layer depletion (Xu et al., 2022). Today, most of the energy is obtained from fossil fuels that cannot be reproduced again and pose negative impact on ecosystem owing to high emission of carbon dioxide (Baz et al., 2021; Anyaoha and Zhang, 2022). The realised CO₂ can alter permeability of atmosphere, allowing radiations to pass through easily, which causes changes in climatic conditions to occur at higher rates. Hence, developed and developing nations are focusing on minimizing the utilization of fossil fuels and increasing the production of renewable energy from available sources (Fathi et al., 2021).

A crucial component of producing renewable energy while keeping sustainability in mind for utilizing waste from the agricultural sector can serve as a suitable, environmentally-benign alternative to fossil fuels (Saleem, 2022; Yana et al., 2022). Depending on their origin, character, and heterogeneity, biomass and feedstock differ from one another and all organic substance that is obtained directly or indirectly through the process of photosynthesis is biomass. Rice straw, rice husk, wood, sugarcane, sugar beet, municipal solid waste, sewage, manure, and other materials are examples of them (Macri et al., 2020; Wang et al., 2021). Biomass resources are organic matter that is obtained from four major types: First-generation biomass feedstock is crop plants. However, due to interference with the food chain and food supply the focus has been moved to second-generation biomass feedstock for bioenergy production, which includes lignocellulosic materials (Esmaili et al., 2020). Subsequently, the drawbacks of second generation resulted in exploitation of third and fourth-generation biomass feedstock that utilizes microbial community (Miret et al., 2016; Low and Yee, 2021; Ebhodaghe et al., 2022). By putting biomass through a variety of analytical methods, one can provide extensive data on the features of biomass for enhancing production and recovery (Hoang et al., 2021). Characterizing biomass both before and after pre-treatment is essential to understand more about the conversion process, mechanism, and design (Zhou and Tian, 2022). Identifying and locating the mechanism involved in the conversion process continue to be difficult tasks. The inability of the technologies employed to evaluate and identify the components of biomass that are relevant to the production of bioenergy and value-added products continues to be a significant obstacle to studying the impact of pre-treating biomass and the factors that affect the chosen method (Yiin et al., 2018; Ha et al., 2020).

This review focuses on importance of agricultural biomass for producing renewable energy production. The various types of agricultural biomass and their utilization methods for bioenergy production are discussed in detail. Importance of biomass pre-treatment and their types, i.e. physical, chemical and biological methods, are explained and brought to under scrutiny. In addition, characterization of biomass using different analytical tools is described in brief. The challenges and difficulties in the way of using agricultural biomass as a potential substance for producing various bioenergy and value-added products, by appropriate pre-treatment methods, are implied and a perspective for the future studies is presented.

2. Agro-waste biomass

Agricultural waste indicates the leftover residues from agricultural activities such as production and processing of agricultural products such as crops, vegetables, fruits, dairy products, etc. Biomass of plant wastes and their components such as cellulose, hemicellulose and lignin are included in agrowaste biomass that can be differentiated into paper waste, wood residues, grasses etc. (Fermanelli et al., 2020; Mukhopadhyay et al., 2022). Polymeric materials such as polypropylene and polyester are also included in agrowaste. Research have been focused on production of biofuels and other forms of bioenergy from agro-waste biomass. Nevertheless, owing to increased demand for food crops, restrictions have been made in utilizing food crops for being utilized as alternative source for producing energy (Zheng et al., 2022). Due to the copious availability, it is significant to connect waste biomass to biomass economy, since most of the biomass can be spent as waste in pre-treatment and energy-production processes. Interest has been developed particularly in utilizing lignocellulosic biomass for producing various biofuels and other value-added products (Kumar et al., 2022; Saravanan et al., 2022). Generally agricultural waste biomass can be classified into crop residues, fruits and vegetable wastes, and livestock wastes.

The waste residues that are obtained directly from the agricultural field during production process are termed under crop residues and include leaves, straws, seed pods, etc., and Fig. 1 shows different types of agro waste. These crop residues are considered as the most abundant and cheapest organic waste materials that can be utilized for producing valuable products (Anand et al., 2022). Wheat straw, corn straw, and rice straw are commonly available crop residues which can be utilized for biofuel production (Panagiotopoulos et al., 2013; Qureshi et al., 2013). These residues are readily available all over the year and, till now, a minimum portion of them have been used for biofuel production, while remaining portions are left unused.

2.1. Primary food crop residue

Rice straw is a value-added promising biomass available worldwide. Rice is an essential and important agricultural product grown in many area of the world. Husk, bran and straw are by-products which are released during rice production. Amongst them, rice straw is a rich source of cellulose material that could be effectively utilized for producing value added products such as biofuels, biomaterials and other biological products (Ranjan and Moholkar, 2013; Tsegaye et al., 2020). Rice straw consists of cellulose, hemicellulose and lignin components. Producing biofuels from rice straw has been challenging due to presence of cellulose and lignin that possess high rate of polymerization.

Rice straw can be utilized for milk production by treating with either calcium hydroxide or urea (Dev et al., 2022; Singh and Patel, 2022). The removal of lignin and hemicellulose were done by breaking the ester bonds present in their structure through chemical and physical pre-treatment approaches. Rice straw was also utilized for producing ceramic materials. Rice husk and bran can be separated from rice straw and can be used as food supplements that bestow another source of income. Rice husk comprises of a majority of volatile substances, carbon and ash content (Elimbinzi et al., 2020; Halim et al., 2022). Rice husk can be used for biofuels production. Rice husk must be hydrolysed prior to its application in biofuel generation. It has been found that, using rice straw as raw material, biofuel production was more, compared to using rice husk as the raw material. Rice bran is a source of vitamins B and E and is natural source of oryzanol.

Next to rice, wheat is another important crop and nearly half of

population consume wheat equally to rice worldwide. Wheat straw is a by-product obtained after removal of scrap from the grain. Wheat straw is rich source of carbohydrates, i.e., cellulose (10–20%), hemicellulose (30–40%), and lignin (10–20%), and some proteins, silica, minerals, and ash contents are also present in the straw structure. It is a wide source of phytosterols which is obtained with noncellulosic compounds and, additionally, ferulic acids and bioactive components are also extracted from this agro waste (Gao and Goldfarb, 2019; Rani et al., 2022). Phytosterols are highly noticed in cell wall region as temperature stabilizer, as they bring about greater physical strength, and used as animal feed and in the biomedical field (Tufail et al., 2021). Few enzymes have also been isolated, using wheat straw as raw material catalysed by fungal species. Wheat straw can also be utilized as adsorbent materials for removal of environmental pollutants. Owing to presence of enormous amount of fibre in it, wheat bran has found a valuable application in animal supplements. Enzyme production and biocomposite synthesis is also possible using wheat bran (Chancharoonpong et al., 2012; Ma et al., 2022). Wheat bran is also loaded with lipids that are essential source of phytochemicals for prevention of various diseases.

2.2. Maize and vegetable residues

Maize is regarded as a wonderful and paramount staple crop from the global nutrition point of view. Corn stover is the leftover that remain after harvesting maize. This biomass is readily available and doesn't call for additional investment. The bare minimum is left on the earth to stop soil erosion. By biochemical and thermochemical conversion procedures, corn wastes can also be employed for the production of energy and the average yield is 20–25 MJ per kg (García-Condado et al., 2019). Corn stover is a main source of organic carbon in soil for maintaining soil quality and productivity (Croce et al., 2016; Yang et al., 2020). It is loaded with hemicellulose and cellulose and its biodegradability is limited by small portion of lignin. Maize straw, chaff and cobs are by-products of maize. Maize straw consists of cellulose, lignin and hemicellulose. Maize straw can be digested through various pretreatment processes such as chemical, heat and mechanical methods (Li et al., 2022). A fungal treatment that increases the digestion of protein and minimizes fibre can enhance the nutritional value of maize husk. Maize husk is a viable and affordable precursor that can be utilized to make adsorbents that remove pollutants from effluents. *Auricularia auricula* is cultivated separately and it is used in the pre-treatment method and is great in lignin digestion. it removes almost 29% of the lignin for producing pure biomass (Intani et al., 2016; Lu et al., 2022).

Another food crop that is grown in great quantities and that produces a lot of trash in the preparation is cassava. Cassava's peel is unusable waste that can be used as manure, and its stem can be used as a base for the generation of bioenergy where 253L biogas is obtained from 10 tons of waste (Cruz et al., 2021; Padi et al., 2022). Cassava peel is a rich source of carbohydrates and annually several billion tons of it are thrown away as waste. It can also be used as a spawn for mushroom cultivation that can be prepared using either as a mixture or as a product isolated from cassava peel. Cassava peel requires pre-treatment processes in which hydrolysis is important for producing bioethanol (Adekunle et al., 2016; Amalia et al., 2021). It can also be utilized as adsorbent material for the removal of heavy metals and other contaminants from polluted water. Cassava stalks can be used for producing boards, polymeric products and plastic composites (Olukanni and Olatunji, 2018).

Groundnuts are filled with carbohydrates that comprise an outer shell. These shells are thrown away as waste after the consumption of inner seeds which is the main food. These shells are thrown away and a small percentage is dried and powdered for their utilization as adsorbent material and as a carrier for fungal inoculation. Groundnut shells can be used as soil stabilizers in maintaining soil moisture and strength and is used as wonderful biomass for biofuels energy of 120–140 kJ/m³ (Yalappa et al., 2017; Deivajothi et al., 2018).

Sugarcane bagasse is rich in polysaccharides and is composed of cellulose, hemicellulose and lignin. It can be used for producing valuable products and electricity generation. Pre-treating bagasse will help in maximum conversion of sugar molecules to bioethanol (H. Huang et al., 2020).

Another important crop is soybean that is directly consumed and is infused with protein. Pod husk and straw are by-products from soybean. Soybean straw comprises of dried leaves, stems, protected by hairs. It is cost-effective and considered as renewable source for producing energy and chemicals. Soybean is loaded with cellulose, hemicellulose, ash, lignin and proteins. Soybean straw is considered as a promising material for biofuel production (Vedovatto et al., 2021).

Next category of using agro-waste biomass includes the usage of obtained wastes from agriculture industrial processing. It comprises of residues and by-products that are obtained from food processing sectors such as vegetables and fruit peels, meat eggs, animal fat, sugarcane bagasse, starch obtained from starch producing industries, molasses from sugar manufacturing industries, etc. Sugarcane bagasse is the commonly and abundantly available agro-industrial waste (Chatterjee and Mohan, 2021; Siddiqui et al., 2022). It is the dry fibrous material



Fig. 1. Different types of agro waste biomass.

obtained after complete extraction of juice. Peels of fruits such as orange, pomegranate and other fruits are also categorized under agro-industrial biomass. Cultivating crops and livestock production provide income to farmers worldwide. Vegetable and fruit waste comprises of unprocessed vegetables and fruits including mango, orange, banana, pineapple, tomatoes, etc. Fruits and vegetable waste generated from food processing industries are numerous. These organic wastes pose serious threat to ecological systems by causing pollution and contamination, due to their perishable nature (Kebaili et al., 2021; Sharma et al., 2022). Enormous waste has been generated from processing, packaging, distributing and consuming fruits and vegetables.

In the past years, agro waste resulted in over 935 million tonnes and they are from variant forms like those mentioned above. When thrown away on land, these wastes cause pollutants and their adverse effects bring about harms to living organisms and ecosystems, either directly or indirectly. Naturally, these are greater sources of agro waste and their exploitation can lead to a stunning yield of bioenergy production. The waste is converted into zero waste and wealth is regenerated by pre-treatment and conversion to give quality biomass for energy production.

3. Feedstock for bioenergy production

Biomass from agricultural field can be utilized as feedstock for producing value-added products. This comprises of biomass from plants, field crops, fruits, vegetables and biomass from fisheries and livestock (Li et al., 2018; Cai et al., 2021). Terrestrial and aquatic plants can be utilized as feedstock for producing energy. Unused biomass that is disposed as waste from agricultural sector, forest and municipal waste also acts as valuable feedstock for bioenergy production (Zhang et al., 2022). Agricultural feedstock can be divided as food and non-food raw materials, depending on whether they are utilized as animal feed. Feedstock can also be divided based on their yield or products obtained from them (Srinivasan et al., 2015). Sugars, wheat, corn, sorghum, sugar beet, barley, etc. can be utilized as feedstock for producing biofuels such as ethanol. In addition, oil producing crops such as palm oil, mustard, coconut, hemp, flax seeds, sunflower, etc. can also be used as feedstock

materials. Few plant species show adaptability to various ecological conditions and those plants suit more for utilizing as feedstock (Liao et al., 2013). Few plants include *Panicum turgidum*, *Phragmites karta*, *Halopyrum mucronatum*, etc. Waste biomasses and agricultural residues from oil, starch and sugar producing crops also can be utilized as feedstock. In addition, waste biomasses from rice, corn, wheat, sugar beet and sugar cane are potential and suitable sources for bioenergy production. Table 1 displays a summary of various agricultural biomasses for bioenergy production.

For producing biofuels such as methanol and ethanol, corn oil and hybrid sugarcane are gaining attention, owing to their vast potency and productivity. Microorganisms and plants liberate lignocellulose continuously in huge quantities. Glucose is produced from cellulose through the action of enzymes and can be utilized in feed or food sectors. Lignocellulosic energy crops such as *Miscanthus* sp produces high yield of biomass and conditions for cultivation of those crops are very limited. Similarly, red giant grass is another crop family with high and long-term yield (Zhou and Tian, 2022). Though, municipal solid waste, animal manures, sewage and poultry manures can also be transformed for energy production, residues from agricultural sectors such as husk, stalk, stem, shells, etc., and from forest such as leaves, timbers, dry leaves, branches, etc. are widely utilized for conversion and production of biofuels and bioenergy (Alam et al., 2022; Pocha et al., 2022). Apart from plant species, aquatic plants and weed varieties are another group which possess potency for utilizing as feedstock for bioenergy generation. High adaptability, more biomass amount and yield of oil produces benefits of using these materials as feedstock in agricultural industries. The microbes present in the field and nearby water sources are helping and can be captured for conversion of biomass into energy resources (Koul et al., 2022; Zhang et al., 2021; Wu et al., 2022). The feedstock that is obtained from agricultural sectors possesses various features. Few demerits of agricultural biomass include collection, transportation and handling. Though there are few issues and problems related to the utilizations of microbial species and agricultural residue, there are benefits, necessities and demands which raise the importance of their usages as feedstock in bioenergy production (Kumari and Singh, 2018). Advanced

Table 1
Various agricultural biomass for bioenergy production.

Feedstock	Residues	Process	Ash content (%)	Parameters	Energy	Yield product	References
Plant & Tree	Crop & Pruning	Anaerobic digestion	–	–	Bioelectricity & Thermal	619 GWh & 895 GWh	Moustakas et al. (2020)
Rice	Straw & husk	Direct combustion	0.22	400 kg/h of steam, 95% thermal efficiency	Bioelectricity	3.8 billion (kW)h per year	Matsumura et al. (2005)
Corn	Stalk	Hydrolysis & Gasification	–	–	Bio-jet fuel	107.7 kg/t-bio	Zhang et al. (2022)
Pine tree	Saw dust	Microwave plasma gasification	–	Air supply: 100 sL/min, Power: 3–6 kW, Temp: 621–1024 °C	Biohydrogen	0.1–0.4%	Sanlisoy and Carpinlioglu (2019)
Micro algae	Dry spirulina algae	Microwave plasma system	–	Power: 700–1100 W, Temp: 700–900 °C	Hydrogen gas	31–36%	Chang et al. (2020)
<i>Jatropha curcas</i>	Dried sludge - Pruning	Stirred tank reactor	–	pH: 5.5, Temp: 37 °C for 4 h,	Hydrogen	66.6 mL H ₂ ·g ⁻¹	Serrato-Nerio et al. (2020)
<i>Chlorella</i> sp	Granular sludge	Dark fermentation	5.0	pH: 5.0–7.0; Feedstock to inoculum ratio: 3.0 in 60 mL	Hydrogen	47.2 mL g-VS ⁻¹	Phanduang et al. (2019)
<i>Chlorella vulgaris</i>	–	Anaerobic membrane bioreactor	–	Temp: 35 °C for 24 h, pH: 7.0, Volume: 2 L, Flow rate: 1.4 mL/min	Biogas	2.4 kWh. kg ⁻¹ COD _(feed) ·d ⁻¹	Vu et al. (2020)
Corn	Cob residue	Anaerobic co-digestion	2.5	pH: 9.0; Temp: 35 °C; Shaking at 180 rpm for 20 days	Methane	126.02 Nm ³ ton ⁻¹ residue	de Oliveira et al., 2020
Kernel	Shell	Pyrolysis gas combustion	2.4	Load capacity: 30 kg/h; Temp: 450 °C; Residence time: 15 min	Thermal energy	197 mg/Nm ³	Salgado et al. (2020)
<i>Silybum marianum</i>	Seeds	Anaerobic digestion	–	–	Biogas	194–223 mL g ⁻¹	Hunce et al. (2019)
Sugar beet	Leaf	Anaerobic digestion	–	Temp: 37 °C, Incubation: 25 days	Methane	1200 Nm ³ ha ⁻¹	Shilpi et al. (2019)
Microalgal	Seashell	Direct transesterification	–	Temp: 80 °C for 2 h	Biodiesel	95%	Karpagam et al. (2020)
Napier grass	Bagasse	Pyrolysis, gasification	–	Pyrolytic oil at 480 °C	Liquid biofuel	5.35 kg/h	Mohammed et al. (2019)
Cocoa waste	–	Feb batch reactor	–	pH: 7.0, Temp: 4 °C	Methane	107 L kg ⁻¹	Acosta et al. (2018)

approaches are needed to be developed for increasing the biomass productivity and yield in producing bioenergy.

The presence of high nutritional components in agricultural waste materials also compels the researchers more to focus for maximum utility. Food industry also liberates enormous amount of food waste and is considered as one of major reasons for ecological pollution (Mohanty et al., 2022). There are various sectors available generating huge quantities of waste residues possessing distinct characteristics and those residues have to be categorized according to their nature and properties for better understanding and utilization. Agricultural residues can be classified as process and field residues. Field residues are those that are left unused or unutilized in the agricultural fields after crop harvesting. Those materials include stems, seeds, dry leaves and stalks. The quantity of field residues are so high such that when left unnoticed or no managed properly causes serious environmental issues and pollution and making the agricultural land unfit for subsequent cultivation. Process residue includes the left over crops after extracting valuable resources. One example is rice cultivation where husk and paddy are process residues. The process residues can be utilized for producing biofertilizer, improving soil characteristics, as feed for animals, etc., (Wang et al., 2015; Kumar et al., 2022). The residues of other industries, such as food industry, contains wastes that are loaded with components such as cellulose, lignin, moisture, ash, hemicellulose, etc. These rich-value components serve more benefits when are utilized for producing biofuels and bioenergy. Fruit and vegetable processing sectors has grown with increasing demand with liberation of enormous residues such as seeds, pulps, peels, leaves, etc. Peels of citrus fruits from citrus processing sector can serve as an efficient substrate during any fermentation process, since they are rich of bioactive components. Studies have successfully shown the feasibility of production of biofuels utilizing various fruit peels (Santos et al., 2015; Lin et al., 2021).

Oil seed production is another important sector contributing to economy. Mustard, soybean, groundnut, etc. are few major sources of vegetable oil. Studies showed that biofuel can be produced using soap stock from oil refineries (Dhanke and Wagh, 2020). The cake that is obtained after pressing for expelling oil is loaded with antioxidants, vitamins, proteins, fibres and minerals. They can be used as food supplements as well as animal feed. For maximum utilization of bio-materials, agricultural industries must focus on advanced developments (Landucci et al., 2013). Increased demand has forced conflict among bio-resources and food production. The utilization of lignocellulosic biomass from trees, crop residues, and grasses with genetically modified crops shows positive pathway for meeting demand and requires more advanced approaches. The utilization of genetically engineered crops and adequate evacuation of crop residues may face problems such as increased soil erosion, depleted soil quality and pollution due to wider application of chemical pesticides and fertilizers. With advancements of genetic engineering approaches, techniques for minimizing the limitations must be developed.

4. Characterization of agro-waste biomass

Heterogeneity is a significant feature of agro-waste biomass. The possibility and ability to recover products from biomass depends on the biomass properties. Thermochemical and biochemical conversion are primarily available methods for product recovery from biomass (Khoo et al., 2013; Fasahati et al., 2022). The selection of conversion approaches is based on the biomass features and hence characterization of biomass is important for better understanding of physico-chemical properties of biomass that helps in determining the biomass suitable for type of conversion. These properties are essential for effective utilization of biomass during biochemical conversion processes. The characteristics of biomass are completely dependent on the organic constituents such as hemicellulose, cellulose and lignin that vary from biomass to biomass, depending on the origin, source, climatic conditions, etc. (Singh et al., 2017). Based on the application of biomass,

characterization may be performed by means of proximate or ultimate analysis, utilizing many different tools, or by both. Proximate analysis determines the biomass physical properties that affect the combustible properties of biomass. Ultimate analysis gives both qualitative and quantitative determination of chemical properties of biomass by means of elemental composition and constitutes some parameters like heating rate of biomass (Shen et al., 2010; Nimmanterdwong et al., 2021). Table 2 shows elemental components and composition of agricultural biomasses and their application in bioenergy generation.

4.1. Techniques for biomass characterization

Characterization of biomass requires utilization of variety of analytical approaches and provides information on composition as well as on the optimization of pre-treatment process. Fig. 2 shows different characterization studies of agro-waste biomasses for bioenergy production. However, the choice of analytical approach used for characterization is determined by the intended application of biomass, so that the results are accurate and specifically needed for the desired application.

4.1.1. Organic compounds characterization

This is a sensitive approach for quantitative and qualitative method of determining the organic materials present in the biomass. Chemical bonds are being found by generating transmittance percentage peaks represented in spectra. This technique is particularly used for finding modifications in structure of biomass (J. Huang et al., 2020). Fourier transform infrared (FTIR) can also be used for finding difference among the functional groups. This method is widely utilized for biomass characterization for various applications including thermochemical and biochemical conversion. FTIR can be utilized for examining the removal of components such as lignin and hemicellulose from pre-treated biomass and can be used for analysing the reactive components present in the biomass and understand how these components influence thermochemical conversion of biomass. FTIR works on the principle that organic compounds absorb light within the infrared region in the range 400 and 4000 cm^{-1} .

4.1.2. Structural characterization

Information regarding the structural components of the biomass can be found using x-Ray diffraction (XRD) instruments. Crystalline size and structure, chemical composition, thickness and orientation are the structural information that can be obtained using XRD analyses (Kataki et al., 2017). Various materials including nanomaterials can be analysed with the help of x-ray diffraction analyser. This technique is specifically utilized for analysing the efficacy of hydrolysis process for chemically pre-treated biomass that is utilized for producing many sugar compounds such as oligosaccharides.

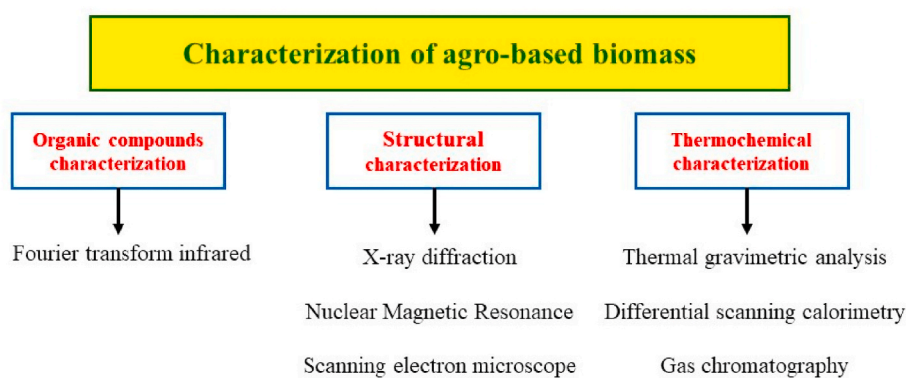
Nuclear Magnetic Resonance (NMR) is an appropriate technique for examining biomass materials that are utilized for production of biofuels or any value-added products. Detailed information on structural analysis of biomass, specifically lignocellulosic biomass materials, can be obtained by exploitation of NMR technique. Cellulose crystallinity can also be found using this technique. While XRD is based on the scattering of X-ray that can be classified into two components, i.e. crystalline and amorphous structures, NMR is based on the orientation that gives complete information on structure and chemistry of the biomass material in its solid state. Production of value-added products using lignocellulosic biomass require huge content of cellulose and hemicellulose and NMR can be used for facilitating biomass conversion effectively.

Scanning electron microscope (SEM) produces pictorial representation of an object through scanning the sample surface by focusing electron beam that connects with the sample for producing signals, in order to obtain information regarding to surface morphology and composition. Prior to analysis, using SEM, biomass materials require pre-treatment for minimizing the size of the biomass particles, for

Table 2

Elemental compounds and composition of agricultural biomass and its application in bioenergy generation.

S.No	Agro biomass	Elemental Compounds				Composition (wt %)				Bioenergy	References
		C	H	N	O	Ash	Moisture	Volatile matter	Fixed carbon		
1.	Palm Kernel Shell	43.20	6.84	0.92	38.03	10.67	3.03	69.66	16.64	Bio-oil	Sohni et al. (2018)
2.	Peanut	59.27	8.18	3.30	22.39	6.86	–	93.95	0.81	Biomethane	Ugwu and Enweremadu (2020)
3.	Wood pellet	51.0	6.0	0.4	42.5	0.3	8.0	76.5	15.2	Biofuel	Kim et al. (2021)
4.	Rice husk	44.2	6.2	–	52.4	14.2	7.0	59.5	20.8	Bioenergy	Naqvi et al. (2020)
5.	Groundnut shell	51.1	5.9	1.1	41.9	4.4	6.7	61.7	27.2	Bio-oil	Bhatnagar et al. (2020)
6.	Coffee husk	46.41	6.33	2.66	44.51	3.55	9.06	77.09	19.36	Bio-oil	Setter et al. (2020)
7.	Neem Press Seed Cake	44.71	4.22	3.7	42.08	4.95	12.5	75.05	7.5	Bio-oil	Dhanavath et al. (2019)
8.	Lignin	61.3	6.23	0.70	31.4	0.3	3.4	74.0	26.1	Biofuel	Farrokh et al. (2018)
9.	Corn straw	50.80	6.24	1.21	41.54	9.40	8.7	75.1	15.5	Bio-oil	Delgado et al. (2013)
10.	Wheat straw	46.3	5.7	2.0	–	8.0	8.9	16.0	56.0	Agglomerates	Funke et al. (2018)
11.	Rice husk	38.23	5.46	0.32	39.44	16.53	–	70.60	12.87	Bio-oil	Zhang and Xiong (2016)
12.	Corn stalk	72.28	3.14	1.09	22.47	16.73	4.36	23.79	55.12	Biochar	Wang et al. (2014)
13.	Pine sawdust	70.68	3.60	2.40	23.11	12.33	–	23.95	63.72	Biosorption	Peng et al. (2012)
14.	Sugarcane Bagasse	32.5	5.0	0.36	61.5	–	–	49.61	42.79	Bioelectricity	Kanwal et al. (2019)
15.	Spent coffee grounds	52.99	7.29	2.50	37.22	0.74	3.16	86.0	10.1	Biofuel	Zhang et al. (2018)

**Fig. 2.** Different characterization studies of agro waste biomass for bioenergy production.

facilitating any type of micro or nanoscopic examinations. The physical and chemical morphological structure of the biomass can be analysed using SEM. The acquired data can be utilized for deciding about the type of pre-treatment process for biomass, in order to obtain maximum susceptibility to thermochemical or biochemical conversion processes. The main drawback is the existing limitations with complete analysis of samples with moisture, since most of these analytical tools go perfect with dry samples only. Hence, moisture content in samples must be completely removed before examination, in order to prevent any interference of structural alterations that may influence the SEM images of the given sample ([Munawar et al., 2021](#)).

4.1.3. Thermochemical characterization

Thermal gravimetric analysis (TGA) can be used for determining the combustible behaviour of the biomass material. This approach is used for determining the thermal properties and parameters related to thermochemical conversion processes. TGA helps in analysing the proximal data. The desired samples are heated to particular temperature at specific heating rate in presence of inactive nitrogen or argon so as to monitor and identify the sample at increasing temperature. The mass of the sample varies as temperature gets varied and is plotted against time or temperature ([Ahmad et al., 2021](#)). TGA works in high temperature that resembles thermal energy production unit. Studies relating to determining the thermal kinetics of biomass decomposition are quite helpful in determining the qualitative data that helps to understand the operating conditions and factors involved in thermochemical conversion processes. TGA is performed at various temperatures and at different heating rates and the peak that is obtained shows the changes in the mass against time/temperature.

Differential scanning calorimetry (DSC) is an analytical tool that is

utilized to uptake the heat energy in the sample under controlled temperature increase (or decrease). The heat flow occurs due to phase transitions among the crucibles and is monitored by DSC ([Musellim et al., 2018](#)). The process includes heating of two crucibles: one is as reference without sample present in it and the other is the one in which the sample to be measured is placed. The heat flow or energy is determined as function of temperature that helps in finding the thermal activity of the biomass material to be analysed. DSC is more valuable for examining the biomass designed as raw material for thermochemical conversion and the information from DSC helps in enhancing the efficiency of activation energy on the biomass conversion rate.

Gas chromatography GC/MS tool is used for analyzing non-volatile compounds and utilizes high temperature for heating the sample to decompose it into smaller components. Those molecules are separated by GC and are finally identified using mass spectroscopy. This analytical tool is specifically utilized for examining biomass materials that are designed as raw material in hydrothermal liquefaction or pyrolysis process for production of value-added products or biofuels ([Shahbeig and Nosrati, 2020](#)). Pyrolysing biomass is a complex process that involves chemical reactions when biomass material is subjected to controlled temperatures. As different biomass varies in structure and composition, the degradation of compounds occurs at varied temperatures and heating rate.

5. Pre-treatment methodologies

Pre-treatment is an important process for thermochemical and biochemical conversion of agricultural biomass and includes structural modifications focusing on the recalcitrant nature of biomass ([Mozhiarasi, 2022](#); [Zhao et al., 2022](#)). Pre-treatment is necessary for enhancing

characteristics and nature of biomass for increasing the efficient energy utilization of biomass. There are various methods of pre-treatment processes. Fig. 3 demonstrates various methods of pre-treatment processes for the conversion of agro-waste biomass. There are various pre-treatment processes involving heat for degrading of lignocellulosic biomass and release of sugar contents. The major demerits of pre-treatment processes include cost and optimizing process so as to obtain minimum degradation of other necessary elements (Ma et al., 2018; Yoruklu et al., 2020). These drawbacks have to be solved by analysing the researchers' efforts and identifying novel approaches that will further accomplish conventional (physical, biological, chemical, mechanical and thermo-chemical) and non-conventional (ionic liquid/electrical) pre-treatment, as required. The choice of type of pre-treatment process must focus and depend on the type, nature and origin of biomass and its wide applicability in biorefinery and bioconversion processes.

5.1. Conventional treatment

5.1.1. Physical pre-treatment

The main aim of physical pre-treatment process is to minimize the size of the biomass particle through mechanical methods for increasing the morphological characteristics such as surface area and pore size. When Lignocellulosic materials are subjected to physical pre-treatment processes, it decreases the crystallinity of cellulose and degree of polymerization (Sidana and Yadav, 2022). However, these two properties are important before thermochemical and biochemical conversion of biomass. There is no detailed information regarding the mode of action of physical pre-treatment method, specifically with how the chemical composition of biomass is altered and affect the morphology. Depending on the application of biomass, the physical pretreatment method is chosen. For biochemical conversion of Lignocellulosic biomass, milling process is employed for reducing the biomass size that enhances the digestion of Lignocellulosic biomass by involving enzymes (Amin et al., 2017). Similarly for thermochemical conversion, milling process is required for torrefaction, pelletization before thermochemical conversion. In addition to milling, chipping is also another method used before thermochemical conversion. Since thermochemical conversion are very size specific, selection of a suitable physical pre-treatment process is vital for obtaining higher efficacy. These methods involve heat or high temperatures for making alterations that result in biomass with improved characteristics. The primary drawback of mechanical pre-treatment is the possible loss of lignocellulosic materials in lignocellulosic biomass sources, which makes the cellulosic content

inaccessible. The merit of this is that any kind of material can be used and the method doesn't affect the chemical and original nature of the raw source so the properties of raw material remain unchanged (Khan et al., 2022).

5.1.2. Chemical pretreatment

Chemical pre-treatment process constitutes the utilization of organic and inorganic compounds for disrupting the morphology of biomass by interacting with polymeric bonds present in organic compounds. Agro-waste, biomass specifically lignocellulosic materials, cannot be broken down using chemical pre-treatment and can be considered as recalcitrant (Sun et al., 2020). This may be due to complexity in structure, cellulose crystallinity, lignification and biomass heterogeneity. During chemical pre-treatment process, structural complexity of lignocellulosic biomass is destroyed, resulting in minimizing the crystalline nature of cellulose, depolymerization, cellulose degradation and lignin breakdown (Galbe and Wallberg, 2019). Chemical pre-treatment is performed for isolating specific biopolymeric composition of the biomass material. Chemicals such as acids, alkalis, ionic liquids and organic solvents are utilized for chemical pre-treatment processes and this is a highly practiced method because of the easily existing, high treatment capacity, and the low-price production. Even low concentrations of the diluted acids are enough for high effective treatment maximum, and 100% efficiency is achieved in this method (Saravanan et al., 2021).

5.1.3. Biological pre-treatment

Biological method of pre-treatment involves utilization of enzymes that are produced by microbes particularly fungi for degrading, depolymerizing and cleaving biomass components such as cellulose, hemicellulose and lignin. This kind of pre-treatment possess has various advantages, compared to other two aforementioned methods, since it does not produce any toxic by-products, increased productivity of required products, minimal energy consumption, substrate specificity and reaction process (Karami et al., 2022). However, slow process, requirement of huge space and maintaining optimum conditions for microbial growth are few limitations that limits biological pre-treatment processes. Normally, residence time is nearly 14 days for this process and, as microbes consumes organic content in biomass, biological pre-treatment process needs to be considered from other aspects, like techno-economic issues (Ahmad et al., 2022; Chan et al., 2022). Hence, this method is not commonly utilized by researchers and is less attractive, compared to other approaches. Commonly utilized microorganisms for pre-treatment process include white-rot fungi, actinomycetes, brown and soft-rot fungi, and sometimes bacterial community. These fungi are known for their efficacy in removing lignin and hemicellulose as well as minimal effect on cellulose. Though many pre-treatment methods have been examined, many methods are in stage of development and require comparison and checking availability, compared to other pre-treatment methodologies. Most of the pre-treatment techniques involve downstream and upstream processing cost related to operation, initial investment, recycling and waste treatment methods. This method uses no chemical substances for treatment but makes use of the enzymes released from microbes for lignin removal, and its efficiency varies from 78 to 99% and the efficiency sometimes relays on some parameters (Deivayanai et al., 2022).

5.1.4. Thermal technique

Treatment option for biomass within a selective temperature range from 50 to 300 °C successfully hikes the anaerobic digestive rates. It also delineates the microbial count from waste components. Hence, such pre-treatment ranks as most utilized technique so far, indeed being chosen as a massive pre-treatment strategy for agricultural outcomes, sewage sludge as well as municipal organic waste fractions. Microwave heating, hydrothermal and steam explosion are usual treatment techniques involved for incrementing complete biodegradation of agricultural biomasses (Cesaro and Belgiorno, 2014). Though such thermal method

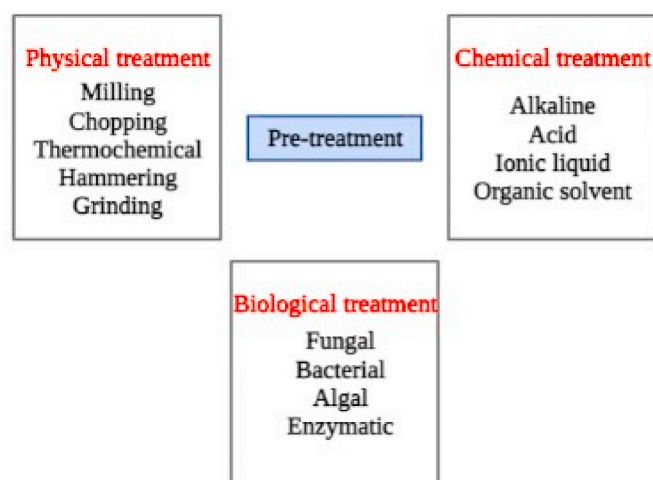


Fig. 3. Various methods of pre-treatment process for the conversion of agro waste biomass.

possess more merits of easy digestion of organic biomasses, higher ranges or lengthier thermal processes can unevenly trigger component reactions. This could lead to complex recalcitrant formation or inhibit substrate components that thwart bioenergy generation. Hence, temperature as well time adopted for such technique must be carefully chosen depending on the characteristic constituents of biomass (Elliot and Mahmood, 2012). Apart from hydrothermal mechanism, steam dependent or explosion treatment is well chosen to attain definitive depolymerization of agricultural biomass. At higher heating levels, steam aids as an acid media and assists water hydrolysis. In such treatment, the agricultural biomass is well exposed to hot fluid of steam along with higher pressure rates for certain minutes and then followed by lowering of both parameters, resulting in biomass explosion beneath the component (Hendriks & Zeeman et al., 2009). Steam available at higher heating ranges dissolves lignin/hemi-cellulosic constituent followed by secondary explosion mechanism for further structural breakage of residual agricultural biomass producing finer materials.

5.1.5. Mechanical pre-treatment

This method involves several comminutive actions of milling, grinding, chipping etc., The main objective of such methodology is to break the solid components completely, simultaneously liberating biomass fragments possessing smaller sizes and porous permeability beneath its overall structural arrangement (Paudel et al., 2017). By such method, the enlargement in surface coverage is more acceptable to biomass constituents and enhances complete digestion much effectively (Esposito et al., 2011). Several demonstrations have deployed that component size is indirectly proportional to biomass utilization intensity. Deduction in component size range of agricultural crop residues increments the exposure of biomass substrate for further hydrolysis procedure (Elliot and Mahmood, 2012). More interestingly, the shearing/cutting action of biomass fibre components is the most predominant mechanism to render positive impact on the digestive performance by incrementing surface coverage.

5.2. Non-conventional pre-treatment

5.2.1. Ionic solution treatment

Ionic solutions technique employs ionic solvents containing lesser melting rates along with non-volatile features that dominate with lignocellulosic components for hydrogen linkages. Such treatments are designated to be much efficient than earlier chemical approach, in specific for lignocellulosic wastages including wheat, corn and wood (Gao et al., 2013). Biogas generation through rice straw, water hyacinth, spruce needles and other leaves was substantially hiked aftermath pre-treatment step with 1-N-butyl-imidazolium chloride. Owing to higher cost of such technique utilizing above solvent alone as a single component, it is reformed by merging with ammonia (Nguyen et al., 2010). Such improvisation enhanced more than 80% cellulose recovery along with 95% rice straw conversion inclusive of reduced processing duration. Mostly this technique has been utilized for ethanol processing and thereby its efficacy towards biomass has been not yet discovered. Anyhow, it forecasts to possess maximum potential to be recommended for residues with lignocellulosic components such as agricultural feed-stocks owing to its delignification capability (Hartmann et al., 2000).

5.2.2. Electrical technique

Pulsed electrical methodology has been involved for wastewater, bio-solids along with biomasses adopting high magnitude electrical fields. This leads to an electrical decomposition and enhanced surface distribution and also permeating capacity beneath the biomass (Singh et al., 2016). Biogas generation procured from vegetable and fruit waste of agricultural fields was selectively incremented after such electrical technique compared to hydrolysis step. Porous nature of woodchips and switch-grass also get increased, anyhow lignin component is somewhat

persistent to such pre-treatment. Additionally, electron radiations can be aided to pre-treat biomasses of lignocellulose (Safavi and Unnthorsson, 2017). Such technique involves ionizing energy to vary chemical/biological linkages of the target component and also has shown to enhance enzymatic hydrolysis step of rice straw.

5.3. Impacts of pre-treatment procedures

The essential objective of such review is designed on the concept that pretreatment techniques for agricultural residues of biomass are much selective to feedstock variety and its salient chemical/physical composition. Vegetable/fruit wastes (starch rich) are maximum digestible whereas cereal components/animal manure consisting lignocellulosic constituents as dominative features are intolerant to aromatic hydrolysis owing to hindrance of lignin assembly along with its carbohydrate bonding, for e.g., the strong affinity betwixt cellulose/hemicellulose along with the inclusion of crystalline cellulosic components (Kumar and Murthy, 2011). It is tough to justify the feasible pretreatment technique for individual agricultural feedstock owing to diversified outcomes ranked so far. Nevertheless, certain pretreatment techniques have rated to acquire better working performance for selective agricultural feedstock components when compared to other techniques of pre-treatments (Paudel et al., 2017).

6. Challenges and future scope

Though researchers are focusing on agricultural biomass as alternate source to fossil fuels for producing bioenergy and biofuels, challenge remains in making the process economically feasible as energy crops are utilized for consumption. During the pre-treatment process, inhibitory compounds such as weak acids produced form phenolic compounds and other derivatives interferes with the process of fermentation and saccharification. These components limit the yield and productivity, influencing the expense of the whole process, that is major challenge. It is estimated that nearly 33% of the total cost accounts for pre-treatment process alone and present approaches available for lignocellulosic biomass needs high capital investment for maintaining stable performance when genetically engineered microorganisms are utilized for commercial operations and developing more suitable and efficient pre-treatment methods for lignocellulosic biomass. As surveys state that demand for energy has increased tremendously in recent years, there exists a great requirement for renewable energy that should not interfere with the food supply. Biofuels and bioenergy produced from lignocellulosic biomass offers more economic and feasible production of bio-energy from wastes.

More efforts and technological innovations are required for making the process cost-effective and easy to use. There is insufficient knowledge of the analytical tools employed for identification and characterisation techniques, so more manuals and educations are needed for usage (Chia et al., 2022). The major drawback is that there is a problem in complete dissolving of the lignin and its breaking down, therefore enzyme degradation method is being used. The pH is regulated at 7 and it increases the yield rate when the bacteria species are exploited. Nano-particles are used in enhancing the fermentation process and helps in boosting the biomass production. It is known that single analytical tool or technique could not provide all necessary information regarding physical and chemical nature of biomass with high resolution and sensitivity Ackom et al., 2013. This may resolve the challenges related to experimental analysis. Characterization approaches possess their own merits and demerits and selection of specific method must depend on the characteristics of biomass. Based on recommendations from previous review articles, pretreatment strategy for agricultural waste biomass, the effectiveness is reliant on feedstock properties and in specific lignocellulosic contents. Heavier quantities of lignocellulosic waste acquires a severe pretreatment methodology such as thermal, alkali and thermochemical processes. Anyhow, such techniques can create

negative effects on such biomass wastes if not suitably implemented. Hence, nonconventional methodology could be an alternative choice which overcomes the drawbacks of conventional treatment processes owing to its positive background features.

7. Conclusion

Agro and forestry are the major sources of environmental waste and they can be used as sources for bioenergy production. Such resources have been utilized in developing countries after conduction pretreatment methods for the best effective availability of biomass. Many detailed explanations of pre-treatment and various characterization processes with lignin removal efficiency have been detailed. The study reveals that biomass characteristics can be enhanced using genetic engineering approaches. In addition, pre-treatment techniques like the enzymatic method are very high-yielding and good method for future bioenergy production. In this work, the drawbacks of the methods used for bioenergy production from agro waste were also indicated and awareness was provided to overcome the obstacle. Overall, a comprehensive study was reported on feedstock, characterization and pretreatment methodologies which are exploited in bioenergy production from agro waste.

Author contributions statement

J.Aravind Kumar - Original draft; S.Sathish - Conceptualization; D. Prabu - methodology, A.Annam Renita - funding, A.Saravanan-resources, V.C.Deivayanai-draft revision, M.Anish - draft revision, J. Jayaprabakar - concept revision, mirserik Baigenzhenov - Methodology, Ahmad Hosseini-Bandegharaei- co-correspondence and complete draft revision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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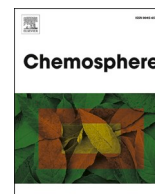
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Update

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