Wheat straw biorefinery for agricultural waste valorisation

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This study justifies strategies for a new concept of agricultural waste management prior to biorefinery, based on comprehensive materials science investigations. Efficient pretreatments for the extraction, separation and fractionation of agricultural waste in conjunction with understanding of the details of its microstructure and properties can be essential for high-efficiency biorefinery. The information in this study will serve as a valuable and fundamental basis for researchers and industries in the sector of straw biomass biorefinery. In order to achieve the maximum efficiency possible in agricultural waste valorisation, it is crucial to understand that not all parts of the straw are equally valuable and can be treated in the same biorefinery process. In this study, the wheat straw stem, which is composed of nodes and internodes, has been shown to have distinct properties and characteristics. Separation of these anatomical parts before the biorefinery process presents a unique area for future research investment, as it can lead to higher performance of the intended product. For example, the node has higher extractives and ash content, which prove to be a diminishing factor for biocomposite or bioenergy production.

1. Introduction

Life-cycle efficiency improvements can be made by recycling and remanufacturing of waste or manufacturing by-products. This practice is directly linked to the circular economy, which is becoming increasingly significant as a research area in the UK and worldwide. In 2017, humankind caused 32 billion Mt of carbon dioxide (CO₂) to be released to the atmosphere, which was in addition to naturally sourced emissions.¹ Despite the billions of dollars invested in research, the total quantity of global greenhouse gases (GHGs) emitted per year has continued its inexorable rise.

Valorisation can be defined as environmentally friendly, industrially feasible and the sustainable conversion of agricultural waste to energy and other useful materials.² The focus should be on the successful transition from laboratory-scale to pilot- and full-scale demonstrations of biorefinery of agricultural waste into multiple products and by-products such as biofuels, bioproducts, fertilisers, heat and/or electricity.^{3–7} Advances in technologies such as biotechnology, process chemistry and engineering are leading to the concept of biorefining.⁸ In a biorefinery, agricultural waste can be valorised in an integrated manner, thereby maximising its economic value and reducing the waste streams produced.⁹ Wheat straw biomass conversion processes are appealing to industries, and extension to future scenarios is easy for the public to envision. Fundamental research has historically been focused on wheat straw biomass conversion to fuels, chemicals and materials;^{10–15} however, a limited sum of these efforts has been successfully translated into commercial practice.

Successful utilisation of wheat straw requires comprehensive understanding of the following points: (a) structure, (b) chemistry, (c) morphology and (d) how these characteristics are changed by

a given pretreatment and processing. Scientific investigation giving an accurate database of the characteristics and composition of wheat straw agricultural waste is a basic requirement for any scheme in conversion and valorisation. Therefore, this paper will present the characteristics of wheat straw and discuss its anatomical components for a selective biorefinery strategy contributing to efficacy of the valorisation concept.

2. Wheat straw main constituents

Wheat straw is a polymeric composite with cell walls made up of cellulose (linear and crystalline), hemicellulose (branched non-cellulosic and non-crystalline heteropolysaccharides) and lignin (branched non-crystalline).¹⁶ Lignin is primarily a structural material that adds strength and rigidity to cell walls.^{17,18} Lignin acts as a matrix together with hemicelluloses for cellulose microfibrils, which are formed by ordered polymer chains that contain tightly packed, crystalline regions, as presented in Figure 1.

Cellulose is made from a long chain of glucose molecules that are linked to each other mainly by $\beta(1\rightarrow4)$ glycosidic bonds. Cellulose has a simple structure, which makes it biodegradable. Hemicellulose is a polysaccharide macromolecule from different sugars, and it is different from cellulose in that it is not chemically homogeneous and has a lower molecular weight. Hemicellulose has branches with short lateral chains containing several sugars, whereas cellulose has hydrolysable oligomers.¹⁶ Apart from the three main groups of organic mixtures, straw comprises several other organic compounds, such as extractives, proteins, wax that protects the epidermis of the straw, sugars, salts and insoluble ash, including silica. Wheat straw contains 35–40% cellulose, 20–35% hemicellulose and around 20% lignin.²⁰ The small lignin percentage in wheat straw makes it a good raw material for production of bioethanol.²¹

Figure 1. Cellulose strands surrounded by hemicellulose and lignin¹⁹

2.1 Anatomical and microstructural variations

On a mass basis, wheat straw has $57 \pm 10\%$ internodes, $10 \pm 2\%$ nodes, $18 \pm 3\%$ leaves, $9 \pm 4\%$ chaffs and $6 \pm 2\%$ rachis¹³ (see Figure 2). The composition of the chemical elements changes between and within the anatomical parts of wheat straw. The internodes of wheat straw have concentric rings leaving a lumen in the centre. The outermost ring contains a dense layer, which is cellulose-rich, called the epidermis. The epidermis has a concentration of silica on the surface. Underneath the epidermis, there is a loose layer, which contains parenchyma and vascular bundles.²²

For the experimental work, the leaves were separated from the stem, and then the stems were grouped and cleaned. The internodes were grouped, and the nodes were carefully cut and separated.

Figure 2. Wheat straw with nodes and internodes

When investigating the node's morphology along the longitudinal direction, interesting results were revealed. By taking cross-sectional images after grinding small layers with smooth abrasive paper moving upwards to the wheat grain, the three-dimensional (3D) image shown in Figure 3 was achieved. The morphological investigation began from the internode instantly before the node and then passed through the node core zone, continuing forward to where the brown elliptical rings become smaller and the beginning of the upper internode is exposed. The brownish elliptical rings become smaller and smaller until they fade. This is the start of the hollow upper internode.

Unlike the node, the longitudinal microstructure of the internode was found to be consistent. The outer surface of the straw internode contains wax and inorganic substances and then follows a region with fibre bundles (vascular bundles) integrated in a region of parenchyma and vessel elements. The epidermis is a complex tissue with bubble-shaped polygonal short and long cell types, as shown in Figure 4. The epidermis is thin but has dense and thick-walled cells with an outer wall coated with a waxy film of cutin cuticle.²²

2.2 Elemental and cell wall composition of wheat straw

There are distinct differences among the physico-chemical characteristics and cell wall components of the node and internode, making them appropriate or deficient for a specific biorefinery pathway. Energy-dispersive X-ray spectra were obtained using an Inca Energy 400 microanalysis system. The chemical elements detected were analysed using a database of standard samples. The ratios of all elements detected were automatically calculated from their normalised peak areas. For quantitative element analyses, the recorded energy-dispersive X-ray analysis (Edax) results were analysed by using the Oxford Inca version 4.02 software package. The bulk structure of the wheat straw consists of carbohydrates and lignin with a considerable amount of carbon (C) and oxygen (O) and a trace amount of silicon (Si) weight percentage (Table 1). The outer surface of the internode has a considerably higher silicon weight percentage than the inner surface – that is, 5.8% compared with 0.8% . More silicon (in the form of silica) is located mainly on the outer surface (epidermis) of wheat straw.

The assessment of cell wall composition of straw biomass is usually conducted on milled samples of the whole stem, without separating nodes and internodes. The cell wall composition of the internode may be rather different from that of the node. Table 2 shows the main chemical components of wheat straw investigated following NREL/TP-510-42620.²³ The wheat straw node yielded slightly higher extractives and ash content than the internode, which can be related to their microstructures – that is, the higher ash and extractive content of the node is attributed to its thicker epidermal tissue. The extractives are a heterogeneous group of substances including resin acids, sterol esters, waxes, triglycerides, fatty acids, sterols, fatty alcohols and a selection of phenolic compounds.²⁴ As shown in Table 2, the percentages of extractives

Figure 3. Node outer (a) and inner (b) surface longitudinal views and the images corresponding to the positions in the node shown by the arrows. Numbers 1–7 represent the start and finish analysis

Figure 4. Optical microscopy image of the internode cross-section

Table 1. Node and internode profile elemental composition based on Edax–scanning electron microscopy analysis

Profile surface	Sample	Percentage: %			Oxygen/ carbon
		Carbon	Oxygen	Silicon	
Inner	Internode	54.1 (2)	45.0 (1)	0.8 (4)	0.83
	Node	54.1 (9)	45.6 (7)	0.7 (6)	0.84
Outer	Internode	51.3 (2)	43.4 (5)	5.8 (2)	0.84
	Node	53.7 (3)	43.5 (8)	2.8 (2)	0.81

Values in parentheses are coefficients of variance (%)

in nodes are higher than in internodes, for both hot-water extraction and ethanol extraction. The results also showed that the node contains more ash, in both non-extracted samples (structural ash) and samples extracted through hot-water and ethanol extraction.

2.3 Characterisation of surface chemical distribution

Figure 5 shows the surface chemical distributions of wheat straw node and internode inner and outer surfaces. Table 3 summarises the characteristics of surface profiles of the node and internode. The intensities of the 2850 and 2920 cm^{-1} bands are much higher in the node (Figure 5), which is ascribed to the higher intensity of waxes on the surface. Moreover, by comparing the inner surface with the outer surface, it is observed that the broad and more intense band in the 3200–3600 cm^{-1} region reflects the hydrophilic tendency of the inner surface of both the node and internode. In wheat straw, some chemical bonds are present in the node but are absent in the internode and vice versa – namely, 2955, 720 and 790 cm^{-1} in the node and 985 cm^{-1} in the internode.

3. Strategies for valorisation of wheat straw

Without an appropriate strategy for disposal of agricultural waste, many aspects of the environment may be negatively affected.³¹ The most abundantly available, cheap and renewable raw material for bioethanol production is lignocellulosic biomass due to its high cellulosic content. Wheat straw is a potential source of feedstock for biofuel production that does not compete with food.³²

Lignocellulosic materials from agriculture waste biomass are the largest sources of hexose (C-6) and pentose (C-5) sugars with a potential for the production of biofuels, chemicals and other by-

Table 2. Extractives and ash content of wheat straw (% dry straw)

Sample	Hot-water extraction		Ethanol extraction		Non-extracted samples
	Extractives: %	Ash content: %	Extractives: %	Ash content: %	Ash content: %
Internode	4.2 (4)	0.9 (8)	3.8 (9)	1.5 (2)	3.2 (2)
Node	4.6 (5)	1.3 (7)	4.0 (7)	1.9 (3)	5.3 (4)

Values in parentheses are coefficients of variance (%)

Figure 5. Attenuated total reflection–Fourier transform infrared spectra of wheat straw internode and node outer and inner surfaces

Table 3. Band assignments and observed differences between the node and the internode

Wave number: cm^{-1}	Band assignment	Observations	Reference
720	Methylene CH_2 in-plane deformation rocking	Detectable only in the node outer surface	Jiang <i>et al.</i> ²⁵
790	Si–C stretching vibration		
985	Si–O stretching vibration	Detectable only in the outer surface of the internode	
1160	C–O–C antisymmetric bridge in hemicellulose and cellulose	Sharper in the internode than in the node	
1435	C=O methoxyl group in lignin	Sharper in the internode inner surface than in the outer surface	
1510	C=C lignin aromatic ring stretch	Sharper in the internode of treated straw	Xiao <i>et al.</i> ²⁶
1739	Carboxyl groups	High intensities in internode and node outer surfaces	Alemdar and Sain ²⁷
2850, 2920	Symmetric and asymmetric stretching of CH_2 in the aliphatic fraction of waxes	Sharper in the node than in the internode and in the outer surface than in the inner surface	Merk <i>et al.</i> ²⁸
2955	Asymmetric stretching of CH_3 in fatty acids	Detectable only in the node of untreated straw	Dodson ²⁹
3200–3600	OH stretching vibration of hydroxyl groups	Higher intensity for the inner surface compared with that for the outer surface, in both the node and internode	Stelte <i>et al.</i> ³⁰

products. For bioethanol/biofuel production, the three main steps include pretreatment, enzymatic hydrolysis and fermentation²¹ (see Figure 6).

Physical, chemical, physico-chemical and biological pretreatments have been utilised for adding value to wheat straw as agricultural waste. The pretreatment must be carefully designed and its

parameters must be chosen in a way that it improves enzymatic hydrolysis, avoids carbohydrate degradation, prevents the formation of inhibitors for the following hydrolysis and fermentation processes, produces high yields of monosaccharides, generates highly hydrolysable cellulose for efficient conversion to chemicals and is economically feasible – for example, low energy and low cost.^{33–35}

Figure 6. Agricultural waste valorisation: lignin recovery and bioenergy production

In an investigation on valorisation of wheat straw, Kaparaju *et al.*⁹ studied the production of bioethanol from cellulose, biohydrogen from hemicelluloses and biogas from the waste of bioethanol and biohydrogen processes. This is in line with an effective low-cost biorefinery concept. Some of the wheat straw was utilised without any treatment, and some was pretreated using a hydrothermal process. The pretreated wheat straw yielded a liquid fraction hydrolysate that contained hemicelluloses and a solid cellulose fraction. The investigation by Kaparaju *et al.*⁹ discovered that the production of biogas from wheat straw or production of different biofuels from wheat straw is an energy-efficient technology in comparison with the generation of mono-fuel, including bioethanol, from hexose sugar fermentation. They also concluded that an integrated waste biorefinery is more feasible than using individual biorefinery technology for the generation of single fuel.

Biological pretreatment weakens the heterogeneous straw biomass with lignin-biodegrading microorganisms that can degrade aromatic compounds. A biological process removes a substantial amount of lignin, which increases the enzymatic hydrolysis efficiency.³⁶ The important benefits of biological pretreatments are small energy input, no chemical obligation and environmentally friendly working style. On the other hand, one of the drawbacks is the slow pretreatment rate. The introduction of some kind of catalyst that can accelerate the process and improve efficiency is necessary.³⁷ A combination of biological pretreatment with mild physical, chemical or mechanical pretreatments is also one way of improving the slow rate of biological pretreatment, therefore improving industrial feasibility.³⁸ Yu *et al.*³⁹ showed that pretreatment of corn stalks with *Irpex lacteus* can modify the lignin structure and facilitate lignin biodegradation and xylan elimination under a mild alkaline environment (1.5% sodium hydroxide (NaOH), 30–75°C for 15–120 min).

Hydrothermal pretreatment (200°C for 10 min) was the initial stage in the procedure of turning wheat straw into second-

generation bioethanol,⁴⁰ where the enzymes were added to the fibre mass (mostly of cellulose and lignin), for bioconversion of cellulose to lower carbohydrates, enabling the fermentation of ethanol in the following stage. Pretreatment of straw for the production of bioethanol is estimated to account for 33% of the summed cost of bioethanol production.⁴¹ Developing an economically suitable processing is therefore the key for bioconversion of straw biomass into bioethanol. The ideal pretreatment in terms of technical aspects would be to (a) expose the cell wall constituents for enzymatic attack, (b) increase the porosity and surface area of the substrate, (c) diminish cellulose crystallinity and (d) disrupt the heterogeneous structure of lignocellulosic biomass.³⁸

3.1 Wheat straw as a bio-based building product

Buildings under construction and in use generate a disproportionate amount of non-recyclable waste along with around 40% of GHG emissions. Therefore, the construction industry has seen increasing demands for natural novel eco-innovative products – for example, compressed straw boards. While in the distant past, most buildings were made of bio-sourced, ultimately biodegradable materials, wood, straw, reed, rammed earth or adobe, today, there is a tendency to regard these as primitive: either insufficiently durable, too weak to withstand the forces of nature or loads in use over time. This is not necessarily an accurate perception. In the case of straw, its life in a building is indefinite provided that it is kept dry.⁴² It is the emergent understanding of the potential durability of straw and its self-evident modest structural capability that has prompted research that aims to reinstate it as a viable building material for modern building construction. As the growth of straw converts airborne carbon dioxide into carbon by photosynthesis, its incorporation into the fabric of a long-lived building after suitable processing would fit in with two pressing policy objectives referred to earlier: first, that of achieving a ‘closed-loop’ cycle of

growth, material use and ultimate safe return to nature and, second, ensuring the effective capture and long-term storage of atmospheric carbon dioxide.

This study developed and demonstrated pilot results, where an environmentally friendly pretreatment was employed, which led to an improved interface between resins and the microporous surface of straw. The results showed that the chemical functionalities of various surface profiles (i.e. when cut longitudinally in half, inner and outer) altered the bonding performance – that is, the extractive and aliphatic fraction of waxes and the silica concentrated on the outer surface – and inhibited the bonding quality, which translates into an inefficient stress transfer under load. The pretreatment ($P < 0.05$), however, could significantly (a) modify the surface of straw with the partial removal of extractives, waxes and silica, which made it more hydrophilic and more compatible with water based resins, and (b) cause the microcellular structure of straw to expand and hence induce mechanical entanglement on the micro level on resin solidification. Therefore, these pilot results have given the author the motivation to upscale the pretreatment. So far, the manufacture of biocomposites, whether for use in vehicles or in construction, has been held back due to their non-reliable load-bearing capability. Research on biocomposites from straw by-products has been focused on utilising them in small particle and/or short fibre form,⁴³ as fillers in plastic composites, while, as proposed herein, the mechanical properties of the composites could be potentially enhanced by using longer straw strands. The highly processed products from straw, entailing the extraction of cellulose for papermaking, require high-energy inputs and pose significant negative environmental impacts and a cost penalty arising from the need to treat large quantities of complex effluents. The other product stream is straw as bales with almost no processing. However, straw bales underutilise the inherent properties of straw, where structural performance tends to be affected by compaction and in humid climates decay is caused by internal condensation.

This research has revealed that the micro-architecture of straw nodes is very different from that of the internode.⁴⁴ The node is much less structurally competent, and distributions of nodes and soluble starch from nodes have been found to deteriorate the performance of the straw strand/stem when these are reconfigured into a biocomposite material. This study has found out this is due to the node's morphology and surface chemical functional groups.⁴⁵ Moreover, the values of tensile strengths of wheat straw internodes are in the range 66–89 MPa, whereas the node showed a tensile strength in the range 12–20 MPa,⁴⁴ further proving the fact that the node could act as a defect in biocomposites.

4. Challenges and perspectives of agricultural waste valorisation

There is a lack of effective utilisation of wheat straw for further biorefinery and bioconversion to value-added products. The complex chemical structure of straw biomass has various

mechanisms for resisting attacks on its structural sugars from microorganisms. These include (a) the epidermal tissue, particularly the cuticle and waxes; (b) the arrangement and density of the vascular bundles; (c) the relative amount of sclerenchymatous (thick wall) tissue; and (d) the heterogeneity of cell wall constituents.⁴⁶ To overcome biomass recalcitrance and thus produce cost-competitive bioproducts from straw biomass, the new findings on the fundamental properties of straw material need to be integrated into conversion processes.

The main challenge is to demonstrate the feasibility of one novel technology chain aimed at valorising several types of agricultural wastes by converting them into an array of valuable bio-based products (e.g. bioplastics, biocomposites and bioenergy) while also minimising any residual or consequent waste requiring disposal. There are needs within different fronts, including but not limited to (a) technical development and product innovation, (b) increasing the bio-based product market demand and (c) gathering sufficient and accurate information about specific variations in straw biomass properties, which is not straightforward.

The change from a linear economy towards a circular economy is essential, and, therefore, the selective and optimised biorefinery of agricultural waste can be the vision to promote a shift from the consumption of fossil reserves to renewable resources, leading to mitigation of GHG emissions and their impact on climate change. Collaborative projects between farmers, research institutes, industrial beneficiaries and policymakers are vital for the success of valorisation of agricultural wastes. The strategies for these types of activities should aim to reduce the carbon dioxide footprint of bio-based products, promote the creation of new job and market opportunities and lead towards an expanded bio-economy, greatly needed for future environmental and economic sustainability.

5. Conclusions

Utilisation of agricultural waste as raw materials positively affects environmental and socio-economic aspects by not only generating additional income for the farmers, but also generating cost-effective high-performing bioproducts. This research contributed to the comprehensive understanding of wheat straw biomass (*Triticum aestivum* L.) by (a) examining and revealing the morphology of the node and internode with a 3D model of the node and its core;²² (b) analysing the different physico-chemical properties of the node and internode and their surface profile functionalisation;^{44,47,48} (c) developing an environmentally friendly pretreatment for surface modification and optimisation of interfacial bonding;⁴⁹ and (d) studying the interfacial bonding and developing a physical model of failure mechanisms in the straw composite.⁴⁹ The complicated heterogeneous characteristics of straw make their comprehensive analysis essential prior to the bioconversion process. It is worth emphasising that the straw biomass's chemical composition can vary with species, location, storage time, harvest, stage of maturity, environmental conditions and anatomical parts – that is, the node and internode.

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