

1 Wheat straw bio-refinery for agricultural waste valorisation

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7 **Abstract**

8 This study justifies strategies for new concept of agricultural waste management prior to bio-
9 refinery, based on comprehensive material science investigations. Efficient pre-treatments on the
10 extraction, separation and fractionation of agricultural waste in conjunction with understanding
11 the details of microstructure and properties can be essential for high efficiency bio-refinery. The
12 information in this study shall serve as valuable and fundamental basis for researchers and
13 industries in the sector of straw biomass bio-refinery. In order to achieve the maximum efficiency
14 possible in agricultural waste valorisation, it is crucial to understand that not all parts of the straw
15 are equally valuable and can be treated in a same bio-refinery process. In our studies, wheat
16 straw stem that is composed of nodes and internodes has shown to have distinct properties and
17 characteristics. Separation of these anatomical parts before bio-refinery process presents a
18 unique area for future research investment as it can lead to higher performance of the intended
19 product. For example, node has higher extractives and ash content that proved to be a diminishing
20 factor for bio-composites or bio-energy production.

21 **Key words:** 1) Material characterisation; 2) Sustainable materials; 3) Biomass conversion.

30 **1. Introduction**

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

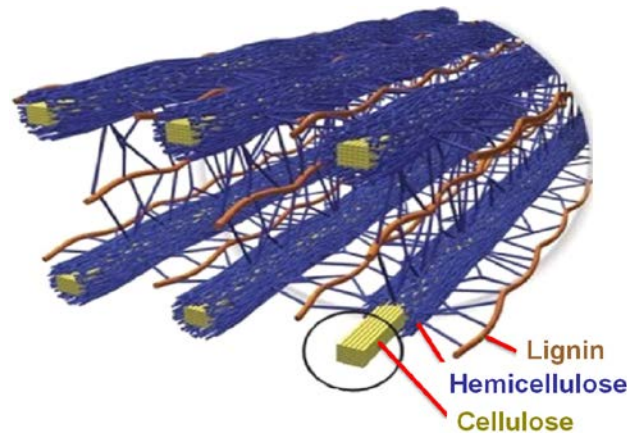
38 Valorisation can be defined as the environmentally friendly, industrially feasible and sustainable
39 conversion of agricultural waste to energy and other useful materials (2). The focus should be on
40 the successful transition of laboratory scale to pilot and full scale demonstrations of bio-refinery
41 of agricultural waste into multiple products and by-products such as biofuels, bio-products,
42 fertilisers, heat and/or electricity (3–7). Advances in technologies such as biotechnology, process
43 chemistry, and engineering are leading to the concept of bio-refining (8). In a bio-refinery,
44 agricultural waste can be valorised in an integrated manner and thereby it can maximise the
45 economic value and reduce the waste streams produced (9). Wheat straw biomass conversion
46 processes are appealing to the industries and extension to future scenarios is easy for the public
47 to envision. Fundamental research has historically been focused on wheat straw biomass
48 conversion to fuels, chemicals and materials (10–15), however, limited sum of these efforts have
49 been successfully translated into commercial practice.

50 A successful utilisation of wheat straw requires comprehensive understanding of the following
51 points: 1) structure; 2) chemistry; 3) morphology, and 4) how these characteristics are changed
52 by a given pre-treatment and processing. Scientific investigation giving accurate database on the
53 characteristics and composition of wheat straw agricultural waste is a basic requirement for any
54 scheme in conversion and valorisation. Therefore, this paper will present the characteristics of
55 wheat straw and discuss anatomical component for a selective bio-refinery strategy contributing
56 to efficacy of the valorisation concept.

57 **2. Wheat straw main constituents**

58 Wheat straw is a polymeric composite with cell walls made up of cellulose (linear and crystalline),
59 hemicellulose (branched non-cellulosic and non-crystalline hetero-polysaccharides) and lignin

60 (branched non-crystalline) (16). Lignin is primarily a structural material to add strength and rigidity
61 to cell walls (17,18). Lignin acts as a matrix together with hemicelluloses for the cellulose
62 microfibrils which are formed by ordered polymer chains that contain tightly packed, crystalline
63 regions, represented in [Figure 1](#).



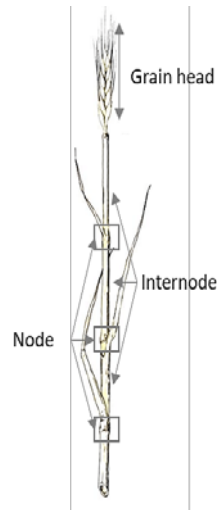
64 **Figure 1 – Cellulose strands surrounded by hemicellulose and lignin** (19)
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66 Cellulose is made from a long chain of glucose molecules that are linked to each other mainly by
67 β (1 \rightarrow 4) glycosidic bonds. Cellulose has a simple structure, which makes it biodegradable.
68 Hemicellulose is a polysaccharide macromolecule from different sugars and it is different to
69 cellulose in that it is not chemically homogeneous and has lower molecular weight. Hemicellulose
70 has branches with short lateral chains containing several sugars, whereas cellulose has
71 hydrolysable oligomers (16). Apart from the three main groups of organic mixtures, straw
72 comprises several other organic compounds such as extractives, proteins, wax that protects the
73 epidermis of the straw, sugars, salts and insoluble ash including silica. Wheat straw contains 35
74 – 40% cellulose, 20 – 35% hemicellulose, and around 20% lignin (20). The small lignin percentage
75 in wheat straw makes it a good raw materials for production of bioethanol (21).

76 **2.1 Anatomical and microstructure variations**

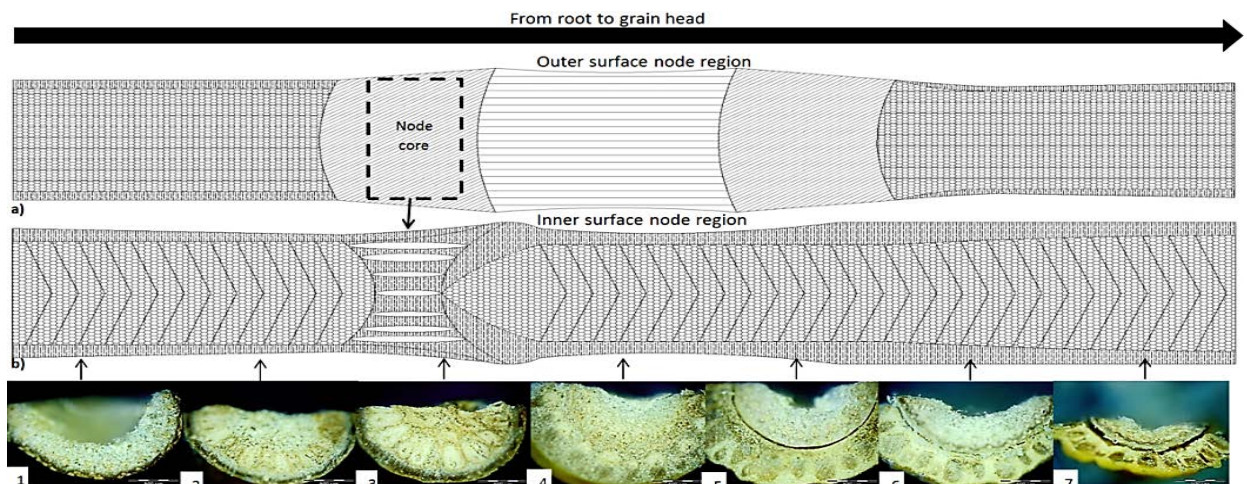
77 On a mass basis, wheat straw has $57 \pm 10\%$ of internodes, $10 \pm 2\%$ of nodes, $18 \pm 3\%$ of leaves,
78 $9 \pm 4\%$ chaffs and $6 \pm 2\%$ rachis (13) (see [Figure 2](#)). The composition of the chemical elements
79 changes between and within anatomical parts of wheat straw. Wheat straw's internodes have
80 concentric rings leaving a lumen in the centre. The outermost ring contains a dense layer, which
81 is cellulose-rich, called the epidermis. Epidermis has a concentration of silica on the surface.

82 Underneath the epidermis, there is a loose layer, which contains parenchyma and vascular
83 bundles (22).



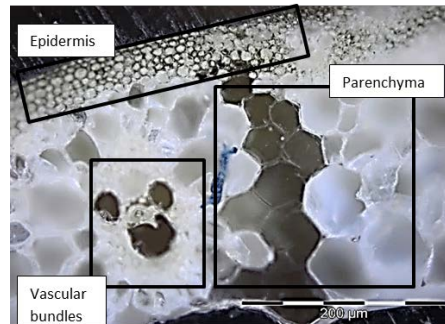
84
85 **Figure 2 – Wheat straw with nodes (N) and internodes (IN)**

86 For the experimental work, the leaves were separated from the stem, and then the stems were
87 grouped and cleaned. The internodes were grouped and nodes were carefully cut and separated.
88 When investigating the node's morphology along the longitudinal direction, interesting results
89 were revealed. By taking cross-section images after grinding small layers with smooth abrasive
90 paper moving upwards to wheat grain, the 3D image as illustrated in Figure 3 was achieved. The
91 morphological investigation began from the internode instantly before the node and then pass in
92 the node core zone, which continues forward to where the brown elliptical rings get smaller and
93 the beginning of the upper internode exposes. The brownish elliptical rings get smaller and
94 smaller until they are fade. This is the start of the hollow upper internode.



95
96 **Figure 3 – Node outer (a) and inner (b) surface longitudinal view and the corresponding**
97 **images to the position in the node shown by the arrows**

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



104 **Figure 4 – Optical microscopy image of internode cross-section**
105

106 **2.2 Elemental and cell wall composition of wheat straw**

107 There are distinct differences amongst the physicochemical characteristics and cell wall
108 components of node and internode, making them appropriate or deficient for a specific bio-refinery
109 pathway. The energy dispersive X-ray spectra were attained using an INCA Energy 400
110 microanalysis system. The chemical elements detected were analysed using the database of
111 standard samples. The elemental ratio of all elements detected was automatically calculated from
112 their normalised peak areas. For quantitative element analyses, the recorded EDAX results were
113 analysed by using Oxford INCA Version 4.02. The bulk structure of the wheat straw consisted of
114 carbohydrates and lignin with a considerable amount of carbon (C) and oxygen (O), and a trace
115 amount of silicon (Si) weight percentage ([Table 1](#)). The outer surface of internode has
116 considerably higher Si weight percentage than the inner surface, i.e. 5.8% compared to 0.8%.
117 More silicon (in the form of silica) is located mainly on the outer surface (epidermis) of wheat
118 straw.

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123 **Table 1 Node and internode profile elemental composition based on EDAX-SEM analysis**

Profile	Sample	Percentage %			O/C
		C	O	Si	
Surface	Inner Internode	54.1	45	0.8	0.83
		(2)	(1)	(4)	
	Node	54.1	45.6	0.7	0.84
		(9)	(7)	(6)	
Outer	Internode	51.3	43.4	5.8	0.84
		(2)	(5)	(2)	
	Node	53.7	43.5	2.8	0.81
		(3)	(8)	(2)	

124 * Values in () are Coefficient of Variance %

125 The assessment of cell wall composition in straw biomass is usually conducted on milled samples
 126 of the whole stem, without separating node and internode. The cell wall composition of the
 127 internode may be rather different from that of the node. [Table 2](#) shows the main chemical
 128 components of wheat straw investigated following the NREL/TP-510-42620. Wheat straw node
 129 yielded slightly higher extractives and ash content than internode, which can be related to their
 130 microstructure, i.e. higher ash and extractives content in the node are explained by thicker
 131 epidermis tissue. The extractives are a heterogeneous group of substances including resin acids,
 132 sterol esters, waxes, triglycerides, fatty acids, sterols, fatty alcohols and a selection of phenolic
 133 compounds (23). As shown in [Table 2](#), the extractives in nodes are higher than in internodes, for
 134 both hot-water extraction and ethanol extraction. The results also showed that the node contains
 135 more ash, in both non-extracted samples (structural ash) and extracted samples through hot
 136 water and ethanol.

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143 **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	
	(%)	(%)	(%)	(%)	
Internode	4.2	0.9	3.8	1.5	3.2
	(4)	(8)	(9)	(2)	(2)
Node	4.6	1.3	4.0	1.9	5.3
	(5)	(7)	(7)	(3)	(4)

144 * Values in () are coefficient of variance %

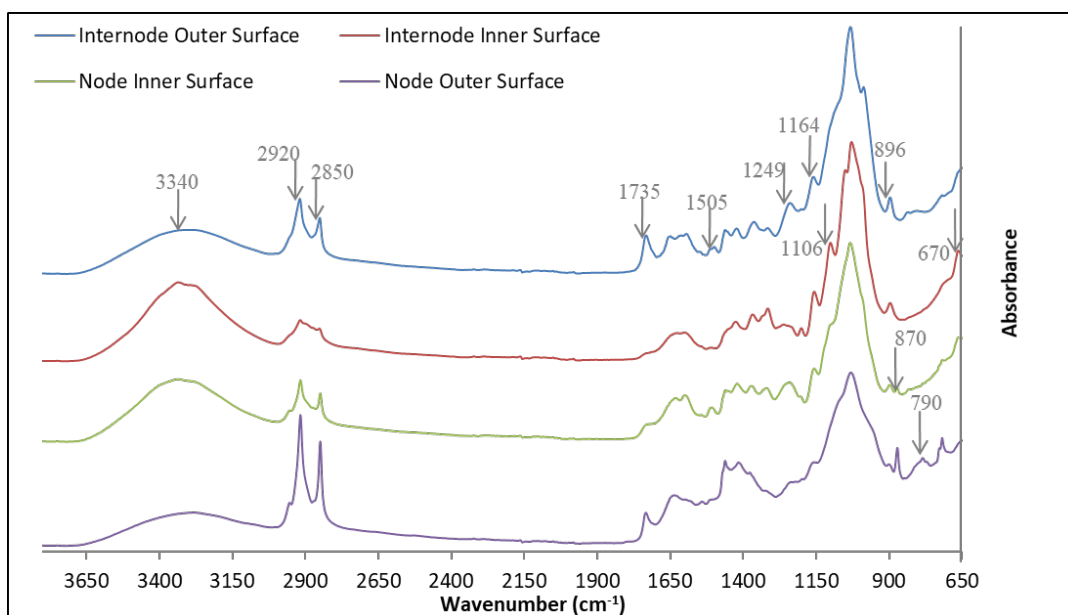
145 **2.3 Characterization of surface chemical distribution**

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

154 **Table 3 Band assignments and observed differences between node and internode**

Wavenumber (cm^{-1})	Bands assignment	Observations	Ref.
720	Methylene CH_2 in-plane deformation rocking	Only detectable in node outer surface	(24)
790	Si-C stretching vibration		
985	Si-O stretching vibration	Only detectable in outer surface of internode	
1160	C-O-C antisymmetric bridge in hemicellulose and cellulose	Sharper in internode than node	

1435	C=O methoxyl group in lignin	Sharper in internode inner surface than outer surface	
1510	C=C lignin aromatic ring stretch	Sharper in internode of treated straws	(25)
1739	Carboxyl groups	High intensities in internode and node outer surfaces.	(26)
2850 & 2920	Symmetric & asymmetric stretching of CH ₂ in aliphatic fraction of waxes	Sharper in node than internode and outer than inner surface	(27)
2955	Asymmetric stretching of CH ₃ in fatty acids	Only detectable in node untreated	(28)
3200-3600	OH stretching vibration of hydroxyl groups	Higher intensity for the inner surface compared to outer surface, both in node and internode	(29)



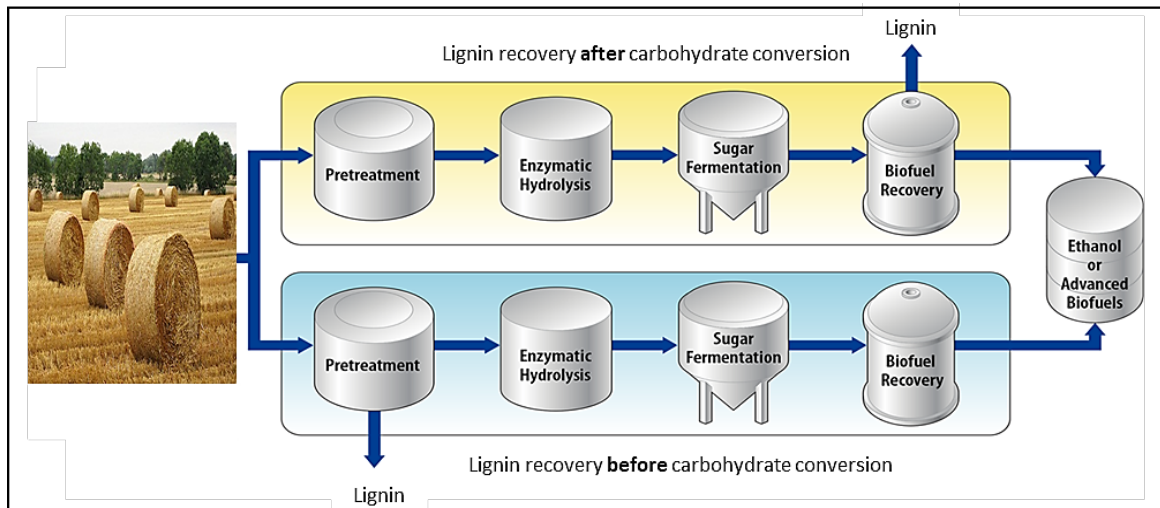
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156 **Figure 5 – ATR-FTIR spectra of wheat straw internode and node outer and inner surface**

157 **3. Strategies for valorisation of wheat straw**

158 Without an appropriate strategy for disposal of agricultural waste, many aspects of the
 159 environment may be negatively affected (30). The most abundantly available, cheap and
 160 renewable raw materials for bioethanol production is lignocellulosic biomass due to its high

161 cellulosic content. Wheat straw is a potential source of feedstock for biofuel production that does
162 not compete with food (31).

163 Lignocellulosic materials from agriculture waste biomass are the largest sources of hexose (C-6)
164 and pentose (C-5) sugars with a potential for the production of biofuels, chemicals and other by-
165 products. For bioethanol/biofuel production, the three main steps include pre-treatment,
166 enzymatic hydrolysis and fermentation (21) (see Fig 6).



167
168 **Figure 6 – Agricultural waste valorisation: lignin recovery and bio-energy production**

169 Physical, chemical, physicochemical and biological pre-treatments have been utilised for adding
170 value to wheat straw as agricultural waste. The pre-treatments must be carefully designed and its
171 parameters must be chosen in a way that improves the enzymatic hydrolysis, avoids
172 carbohydrates degradation, prevents formation of inhibitors for the following hydrolysis and
173 fermentation processes, produces high yields of monosaccharides, generates highly hydrolysable
174 cellulose for efficient conversion to chemicals and be economically feasible, e.g. low energy and
175 low cost (32–34).

176 In an investigation on valorisation of wheat straw, Kaparaju et al. (9) studied the production of
177 bioethanol from cellulose, bio-hydrogen from hemicelluloses and biogas from the waste of
178 bioethanol and bio-hydrogen processes. This is in line with an effective low-cost bio-refinery
179 concept. Some of wheat straw was utilised without any treatment, and some was pre-treated
180 using hydrothermal process. The pre-treated wheat straw yielded liquid fraction hydrolysate that
181 contained hemicelluloses and a solid cellulose fraction. Kaparaju et al. (9) investigation
182 discovered that the production of biogas from wheat straw or production of different biofuels from

183 wheat straw was an energy efficient technology in comparison to mono-fuel generation including
184 bioethanol from hexose sugars fermentation. They also concluded that the integrated waste bio-
185 refinery is more feasible than using individual bio-refinery technology for the generation of single
186 fuel.

187 Biological pre-treatment weakens the heterogeneous straw biomass with lignin biodegrading
188 microorganisms that can degrade aromatic compounds. A biological process removes substantial
189 amount of lignin, which increases the enzymatic hydrolysis efficiency (35). The important benefits
190 of biological pre-treatments are small energy input, no chemical obligation, environmentally
191 friendly working style. On the other hand, the drawbacks are the slow pre-treatment rate. The
192 introduction of some kind of catalyst is necessary which can accelerate the process and improve
193 the efficiency (36). Combination of biological pre-treatment with mild physical, chemical or
194 mechanical pre-treatments is also one way of improving the slow rate of biological pre-treatment,
195 therefore improving the industrial feasibility (37). Yu et al. (38) showed that pre-treatment of corn
196 stalks with *Irpex lacteus* can modify the lignin structure and facilitate lignin biodegradation and
197 xylan elimination under mild alkaline environment (1.5% NaOH, 30–75°C for 15–120 min).

198 Hydrothermal pre-treatment (200°C for 10 minutes) was the initial stage in the procedure of
199 turning wheat straw into second generation bioethanol (39), where the enzymes were added to
200 the fibre mass (mostly of cellulose and lignin), for bioconversion of cellulose to lower
201 carbohydrates, enabling the fermentation of ethanol in the following stage. Pre-treatment of straw
202 for the production of bioethanol is estimated to account for 33% of the summed cost of bioethanol
203 production (40). Developing an economically suitable processing is therefore the key for
204 bioconversion of straw biomass into bioethanol. The ideal pre-treatment in terms of technical
205 aspects would be to i) expose the cell wall constituents for enzymatic attack, ii) increase the
206 porosity and surface area of the substrate, iii) diminish the cellulose crystallinity and disrupt the
207 heterogeneous structure of lignocellulosic biomass (37).

208 **3.1 Wheat straw as bio-based building product**

209 Buildings under construction and in use generate a disproportionate amount of non-recyclable
210 waste along with around 40% of greenhouse gas emissions. Therefore, the construction industry
211 has seen increasing demands for natural novel eco-innovative products e.g. compressed straw
212 boards. While in the distant past, most buildings were made of bio-sourced, ultimately

213 biodegradable materials: wood, straw, reed, rammed earth or adobe; today, there is a tendency
214 to regard these as primitive: either insufficiently durable, too weak to withstand forces of nature
215 or loads in use over time. This is not necessarily an accurate perception. In the case of straw, its
216 life in a building is indefinite; provided that it is kept dry (41). It is the emergent understanding of
217 the potential durability of straw and its self-evident modest structural capability that has prompted
218 research that aims to reinstate it as a viable building material for modern building construction.
219 As the growth of straw, converts airborne CO₂ into carbon by photosynthesis, its incorporation
220 into the fabric of a long-lived building after suitable processing would fit in with two pressing policy
221 objectives referred to above. Firstly, that of achieving a "closed-loop" cycle of growth, material
222 use and ultimate safe return to nature and, secondly, ensuring the effective capture and long-
223 term storage of atmospheric CO₂.

224 Our developed and demonstrated pilot results, where an environmentally friendly pre-treatment
225 was employed, which led to an improved interface between resins and the micro porous surface
226 of straw. The results showed that chemical functionalities of various surface profiles (i.e. when
227 cut longitudinally in half, inner and outer) altered the bonding performance, i.e. extractive, aliphatic
228 fraction of waxes, and silica concentrated on the outer surface, inhibited the bonding quality which
229 translates into an inefficient stress transfer under load. The pre-treatment ($P < 0.05$) however,
230 could significantly: (i) modify the surface of straw with the partial removal of extractives, waxes,
231 and silica which made it more hydrophilic and more compatible with water based resins, (ii) cause
232 the microcellular structure of straw to expand and hence induce the mechanical entanglement on
233 a micro level upon resin solidification. Therefore, these pilot results have given us the motivation
234 to upscale the pre-treatment. So far, manufacture of bio-composites, whether for use in vehicles
235 or in construction, has been held back due to their non-reliable load-bearing capability. Research
236 on bio-composites from straw by-products has been focused on utilising them in small particle
237 and/or short fibre form (42), as fillers in plastic composites, while, as proposed herein, the
238 mechanical properties of the composites could potentially increase by using longer straw strands.
239 The highly processed products from straw, entailing the extraction of cellulose for papermaking
240 require high-energy inputs and pose significant negative environmental impacts and a cost
241 penalty arising from the need to treat large quantities of complex effluents. The other product
242 stream is straw as bales with almost no processing, however, straw bales underutilise the inherent

243 properties of straw, where structural performance tends to be affected by compaction and in
244 humid climates decay is caused by internal condensation.

245 Our research has revealed that the micro-architecture of straw nodes is very different from that of
246 the internode (43). It is much less structurally competent and distributions of node and soluble
247 starch from nodes have been found to deteriorate the performance of the straw strand/stem when
248 these are reconfigured into a bio-composite material. We found out this is due to node's
249 morphology and surface chemical functional groups (Ghaffar, Fan and McVicar, 2017). Moreover,
250 the values of tensile strengths of wheat straw internodes are in the range of 66-89MPa, whereas
251 the node showed a tensile strength in the range of 12-20MPa (43), further proving the fact that
252 node would act as a defect in bio-composites.

253 **4. Challenges and perspectives of agricultural waste valorisation**

254 There is a lack of effective utilization of wheat straw for further bio-refinery and bioconversion to
255 value added products. The complex chemical structure of straw biomass has various mechanisms
256 for resisting attacks on their structural sugars from microorganisms, these include: 1) the
257 epidermal tissue, particularly the cuticle and waxes, 2) the arrangement and density of the
258 vascular bundles, 3) the relative amount of sclerenchymatous (thick wall) tissue, 5) the
259 heterogeneity of cell wall constituent (45). To overcome the biomass recalcitrance and thus,
260 producing cost-competitive bio-products from straw biomass, the new findings of the fundamental
261 properties of straw material sciences need to be integrated into the conversion processes.

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

269 The change from a linear economy towards a circular economy is essential and therefore the
270 selective and optimised bio-refinery of agricultural waste can be the vision to promote a shift from
271 the consumption of fossil reserves to renewable resources, leading to mitigation of greenhouse
272 gases emissions and their impact on climate change. Collaborative projects between farmers,

273 research institutes, industrial beneficiaries and policy makers are vital for the success in
274 valorisation of agricultural wastes. The strategies for these types of activities should aim to reduce
275 the carbon footprint of bio-based products, promote the creation of new job and market
276 opportunities, and lead towards expanded bio-economy, greatly needed for future environmental
277 and economic sustainability.

278 **5. Conclusions**

279 Utilisation of agricultural waste as raw materials positively affects environmental and socio-
280 economic aspects by not only generating additional income to the farmers, but also generating
281 cost-effective high performing bio-products. Our research contributed to comprehensive
282 understanding of wheat straw biomass (*Triticum aestivum* L.) by: 1) examining and revealing the
283 morphology of node and internode with a 3D model of node and its core (22). 2) analysing different
284 physicochemical properties of node and internode and their surface profile functionalisation
285 (43,46,47). 3) developing an environmentally friendly pre-treatment for surface modification and
286 optimisation of interfacial bonding (48). 4) studying the interfacial bonding and developing a
287 physical model of failure mechanisms in straw composite (48). The complicated heterogeneous
288 characteristics of straw, makes their comprehensive analysis essential prior to bioconversion
289 process. It is worth emphasising that straw biomass's chemical composition can vary with
290 species, location, storage time, harvest, stage of maturity, environmental conditions and
291 anatomical parts, i.e. node and internode.

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