Comparative micro-structure and sorption isotherms of rice straw and wheat straw

Abstract

Straw bale construction can be utilised to deliver energy efficient and low carbon buildings, however there are concerns over the durability of straw. The water sorption property is a critical factor in the degradation behaviour of straw and other lignocellulosic materials. Typically, either wheat or rice straw is utilised for bale construction, however, this variation is not considered. This paper presents the sorption property of wheat and rice straw and the micro-structure that affect the sorption property. Although the surface and cross-sectional structure of the two straw species are different at micro scale, the sorption isotherms were not significantly different. This study on the water sorption property of rice and wheat straw allows for the assessment of durability within a wall construction. This impact of this research will be the growth in low carbon energy efficient straw bale construction with confidence over its long term durability characteristics.

Keywords: Straw bale construction, durability, Adsorption isotherm modeling, Hygrothermal properties, sustainability.
1. Introduction

Straw bale buildings combine features of low environmental impact (1), affordability (2), good thermal insulation properties (3) and provide a good hygrothermal environment for living (4). The use of straw bale in building construction began in Nebraska in 1890s (5) as a form of loadbearing construction, however modern methods can utilise straw bale in a prefabricated panels (6). Currently, the straw used in straw bale buildings is mostly wheat straw and rice straw (7). Since the first introduction of this type of building, durability of straw within walls has been a major concern (5, 8). Moisture within straw bale walls is widely considered as the major factor which influences durability of straw bale construction (8). The moisture content of straw bales can either be checked directly by using a moisture probe (9) or be predicted by using an equation to convert relative humidity (RH) in isothermal condition within straw bale walls to moisture content of straw (10, 11). Because the moisture probe method needs direct contact between the probe and the straw bales, installation of the probe into straw bale walls can involve the removal and replacement of render and straw (11). As a result, the method is a highly invasive monitoring method for straw bale buildings. A second approach to measurement is the indirect technique by converting measured relative humidity inside straw bale walls to moisture content of straw. Lamond and Graham (12) report a direct connection between the moisture content of straw and the surrounding relative humidity of straw at the same temperature. This relationship allows for the assessment of moisture content of the straw bale inside the wall through the measurement of relative humidity (12).

The aim of this paper is to establish the variation in isothermal moisture adsorption of two different types of straw used within construction. This paper compares two species, rice straw and wheat straw, in terms of sorption isotherms and physical structure at a microscopic level. Differences in moisture sorption behaviour have
the potential to impact on the comparative durability of straw bale buildings, and
it is therefore critical for designers and architects to understand the impact that
the choice of straw species has on the long term viability of this construction
system.

2. Background

Straw is a plant fibre with similar micro cellular structure to wood cells (13). As
with wood fibres, straw is also a porous material (13). The porous nature traps
moisture (14) from the surrounding air regulating fluctuation in RH (14) and straw
is commonly referred to as a hygroscopic building material (15). The moisture
content of straw tends to equilibrate with the RH of the surrounding environment
in the immediate vicinity (16). As with other hygroscopic materials, the water
vapour sorption of straw has five phases (Figure 1): Single layer of adsorbed
molecules, multiple layers of adsorbed molecules, interconnected layers, free
water in capillaries and supersaturated (17).

Figure 1. Phases of moisture content of hygroscopic materials. (reproduced from
(17))
Plant fibre and wood have two unique features in high RH situations which are not observed with inorganic hygroscopic materials; the fibre saturation point and capillary condensation (14, 16). Both cell walls and cavities between cells can adsorb moisture vapour in high humidity conditions (14). To describe the stage at which cell walls become saturated, the term fibre saturation point is used (16). The fibre saturation point is the moisture content at which the cavities between cells contain no water but the cell walls are fully saturated (16). It is difficult to estimate the fibre saturation point of plant fibre and wood in practice (13). The normal assumption is that the fibre saturation point of wood occurs at a moisture content of 27-32% (14, 16). As RH increases, water vapour can condense on the walls of pores in plant fibres (16). This capillary condensation contributes a significant amount to the moisture content of plant fibre when RH is over 70% (13). However, because of the dense structure of cellulose chains between wood cells, the capillary condensation is likely to occur after the moisture content exceeds the fibre saturation point (16). The capillary condensation is also affected by the pore structure and size of plant fibre and wood cell (13).

The sorption isotherm describes the direct relationship between moisture content of hygroscopic materials and the RH in surrounding environment (18). The isotherm is commonly achieved by using either a desiccator method or a climatic chamber method in laboratory (19), however Hedlin (10) used a jacketed air bath to demonstrate the isotherms for five different grain straw species. The desiccator method uses saturated salt solutions to create a known RH in a sealed container at 23°C and the climatic chamber uses an external source of water vapour to adjust the RH level in a sealed chamber (19). Based on the experimental results, Hedlin (10) produced an equation to predict moisture content of straw:
\[ \varphi = \frac{1 - K_c (1 - \frac{C_s}{C})}{1 + \left[ \frac{C_s - 1}{n} \right]^3/i} \]

Where:

\( \varphi = \text{relative humidity} \)

\( C = \text{moisture content at relative humidity } \varphi \)

\( C_s = \text{fibre saturation moisture content (400\%)} \)

\( n = C_s / C_{50\% RH} \)

\( K_c = 0.0227 \)

\( i = 1.6 \)

The equation was calibrated by straw species including oat straw, barley straw, flax straw and two types of wheat straw at 14 different RH levels at 70 °F (21.1 °C) (10). The equation cannot convert RH in the surrounding environment to moisture content without air temperature, meaning its usefulness in predicting moisture content of straw bales in walls is limited. However, the effects of temperature have little effect on water absorption of straw in the day to day life of straw bale buildings. Strømdahl (13) used a climatic chamber to investigate the water absorption properties of four plant fibres. The sorption isotherm of wheat straw showed no significant differences at 20°C, 40°C and 60°C. Compared with the isotherms at 20°C and 40°C, the moisture content of wheat straw is slightly lower at 60°C. As the air temperature is not likely to reach 60°C, this isotherm is not relevant for predicting moisture content of straw within straw bale walls. Experimental results show no significant differences of moisture content within the straw at temperatures of 5°C, 15°C, 25°C and 35°C (20). Based on the experiment, Lawrence et al. modified the Hedlin equation by ignoring effects of temperature difference (11). The modified equation has similar results in predicting moisture content of wheat straw (Figure 2). The simplified equation is shown as:
\[ C = \frac{C_s}{1 + n \left( \frac{K_m}{\varphi} - 1 \right)^{i/3}} \]

Where:

- \( C = \) moisture content at relative humidity \( \varphi \)
- \( C_s = \) fibre saturation moisture content (400%)
- \( \varphi = \) relative humidity
- \( n = 44 \)
- \( K_m = 1 - K_c = 0.977 \)
- \( i = 1.6 \)

Figure 2. Comparison of Lawrence expression and Hedlin equation (10).

Carfrae (15) reviewed published sorption isotherms of wheat straw and rice straw. The sorption isotherm of Sain and Broadbent (21) is the only published isotherm of rice straw. Compared to research on wheat straw, the research by Sain and Broadbent presents an adsorption isotherm for rice straw with notably lower moisture content in each RH situation. Rice straw showed less water adsorption...
than wheat straw at all relative humidity levels. The research of Sain and Broadbent (21) used vacuum desiccators containing sulfuric acid to achieve the designed RH levels, not in accordance with current standards, and as a result the different water adsorption properties of rice straw and wheat straw remain uncertain. Apart from this research, there is limited published sorption isotherm data for rice straw which follows current standards. It is therefore uncertain whether the reported differences are a function of different straw species or different experimental methods.

Concluding from the literature, the adsorption isotherm of wheat straw is well understood. However, the adsorption property of rice straw is not well understood at the moment. This paper studies the microstructure and adsorption property of rice straw and compares the adsorption isotherm with wheat straw. The adsorption isotherms of the two straw species are further researched by considering macrostructural differences between the two straw species.

3. Materials and Methods

3.1. Materials

The straw species considered are wheat straw and rice straw. The rice straw is sourced from northeast China and the wheat straw is from England. The rice straw and wheat straw were studied both in the form of straw bundles and short lengths of straw (chips). The bundles were around 20g in mass, 40mm in diameter and 160mm in length. The density of the bundles was set at around 110kg/m³ to reflect the density of straw bales which are used in construction. For the DVS tests samples consisted of two chips in each test. One chip consisted of only the sheath, and the other chip included a higher density node in order to be fully representative of the material. The straw chips weighed 3-5g and were 40-60mm in length.
3.2. Methods

There are two parts to this study. The first part is the characterization of microstructure of rice straw and comparison of the microstructure with wheat straw. The second part is the sorption isotherm study.

3.3. Characterization

The outer surface and cross-section of the two straw species, were imaged with a JEOL SEM6480LV Scanning Electron Microscope (SEM) at an accelerating voltage of 10kV. Following initial drying the specimens were gold coated in a sputter coater for five minutes.

3.4. Sorption Isotherm

The study followed BS EN ISO 12571:2013, using both the climatic chamber method and the desiccator method. A dynamic vapour sorption (DVS) machine was used to produce a continuous isotherm for the climatic chamber method. This allowed for comparison of both methods. Straw chips were used in the DVS method and small bundles were used in the desiccator method. Both forms of straw were placed dried at 105°C until no further mass change occurred, at which point they were weighed to establish a zero moisture content mass.

3.4.1. DVS method

The DVS machine used for this study was the DVS Intrinsic, manufactured by Surface Measurement Systems Ltd. The advantage of the machine was that it was capable of producing a highly sensitive and rapid sorption isotherm and desorption isotherm. The specimens were examined at relative humidity levels...
between 0% - 95% at 5% relative humidity intervals at 23°C. The equipment used two different mechanisms to establish the time intervals at which to change relative humidity levels. Between 0% and 65% RH, changes were based on the change of specimens in mass over time. When \( \frac{dm}{dt} < 0.002 \text{g/min} \) the change to the set RH point was initiated. Between 70% RH to 95% RH, because the rate of adsorption of both species of straw becomes significantly slower, changes were made at 1600 minute intervals in order to achieve a full isotherm within an acceptable time period. At maximum RH, this equated to a \( \frac{dm}{dt} \) of 0.036-0.038g/min.

In this study the specimens consisted of single pieces of rice straw and wheat straw. The microstructure of the straw cross section was different to the outer surface of the straw. As a result, it was hypothesised that the cross section of straw might have an impact on the water sorption property of straw. The research differentiated the adsorption isotherm of the two straw species by considering microstructural differences between the outer surface and cross-section of straw. Each specimen was subjected to three sorption/desorption cycles and then the ends were sealed with wax, and a further three sorption/desorption cycles were performed (Figure 3).

Figure 3. Rice straw chip with wax sealing in edge in the DVS Intrinsic2
3.4.2. Desiccator method

The desiccator method was used in this study for RH levels where fibre saturation was expected to occur. Two saturated salt solutions were used to produce sorption isotherms in a high relative humidity environment. The saturated salt solutions used in the research were ammonium sulfate solution and potassium sulfate solution to produce RH 81.13%±0.28% and RH 97.42%±0.47% respectively (19). Small straw bundles were used in the desiccator method. There were three rice straw bundles and three wheat straw bundles at each humidity levels as shown in Figure 4. The specimens were placed in the sealed containers with different saturated salt solutions. Due to accuracy ranges of relative humidity provided by saturated salt solutions are listed in the BS EN ISO 12571:2013, installation of temperature and humidity sensor was not required in the desiccators method in the standard (19). In consideration of monitoring the hygrothermal environment within the two setups of desiccator method, a HTC-1 temperature and humidity sensor was placed in the containers. The sensors have accuracy of ±0.3°C from -25°C to 85°C and ±1%RH@50% (±3% 0%-95%RH). The containers were maintained at a temperature of 23 °C. These HTC-1 sensors confirmed that the desired humidity of the environment was achieved from the respective salt solutions at the given temperature.
For each salt solution, six specimens (three rice straw and three wheat straw) were studied. The specimens were maintained at these conditions for a period of 6 weeks, at which point they were weighed, to the nearest 0.01g to establish their moisture content. Considering the average mass of the bundles, this represents a potential error of 0.1%. The specimens were then replaced in the container for a further 24 hours and re-weighed. This process was repeated until the change in mass was less than 0.1% between readings. At this time it was established that there was no further mass change and it could be assumed that equilibrium had been achieved. (19).

4. Results and analysis

4.1. Physical Characteristics

The cross-section of wheat straw and rice straw are presented in Figure 5 at a similar magnification. Compared with rice straw, the wheat straw contains larger cells. The cellular diameter of wheat straw continuously reduces as it progresses...
from the core towards the external surface (epidermis). However, the convoluted
structure of the rice straw contains cells with less variability in size. The cellular
size of rice straw has similar diameter to the smallest cells of wheat straw
(approximately 5-10µm). The wheat straw and rice straw both incorporate
vascular bundles containing phloem and xylem cells although the size of these
bundles in the wheat straw is significantly larger than the rice straw. The bundle
sizes are labeled in red ovals in Figure 6. The differences in tissue density and
cell sizes between wheat straw and rice straw may lead to different vapour
sorption properties for the two species of straw at high humidity levels.

Figure 5. Cross-section of wheat straw (left) and rice straw (right).

Differences between wheat straw and rice straw have also been identified on
external surfaces in the SEM images (Figure 6). Unlike the visibly smooth surface
of wheat straw, rice straw possesses little spikes. The size of each spike is
approximately 10-20 µm with the pointed ends oriented parallel to the straw stem
(Figure 7).
Figure 6. External surface of wheat straw (left) and rice straw (right)

Figure 7. Spiked features on the external surface of rice straw.

The spiked features do not cover the entire external surface of rice straw, and are therefore not expected to have any significant impact on the moisture sorption properties. The effect of the spikes on rice straw may enhance the mechanical properties of rice straw bales by providing interconnectivity and it needs further study. It should be noted that anecdotally, Californian straw bale builders report that rice straw bales are more rigid than wheat straw bales, which could be explained by this surface phenomenon (1).

4.2. Sorption isotherm

4.2.1. Effect of straw variety on sorption and desorption behaviour

The result of the sorption isotherm study shows no significant difference between
the water sorption properties of the two species in either the DVS method and the desiccator method. Despite significant differences in the cross-sections of the two straw species, the cellular structure has only a minor impact on the water sorption properties of each straw species (Figure 8). The moisture content of the two straw species shows less than 1% difference in the RH range from 0% to 90%. The peak moisture content of the two straw species at 95% RH is slightly more different (Table 1). The largest difference of moisture content between each sets is 4.8% in the DVS method. The results of the desiccator method show around 3% more moisture content for wheat straw than for rice straw at each similar relative humidity (Table 2). All data are presented to demonstrate the range of measurements between the three specimens. There is no statistical difference between the isothermal sorption properties of the different types of straw based on the t-test.
Figure 8. Sorption and desorption isotherm of untreated straw (left) and edge sealing straw (right) in the DVS method.

Table 1. Moisture content % of dry mass (MC) of wheat straw and rice straw by DVS at high RH levels

<table>
<thead>
<tr>
<th>RH Level</th>
<th>80%</th>
<th>95%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straw Species</td>
<td>Rice Straw</td>
<td>Wheat straw</td>
</tr>
<tr>
<td>Mean MC</td>
<td>15.73</td>
<td>15.94</td>
</tr>
<tr>
<td>Coefficient of Variation</td>
<td>2.0%</td>
<td>2.9%</td>
</tr>
</tbody>
</table>

Table 2. Moisture content % of dry mass (MC) of wheat straw and rice straw in two saturated salt solutions.

<table>
<thead>
<tr>
<th>RH Level</th>
<th>81.13% RH (ammonium sulphate)</th>
<th>97.42% RH (potassium sulphate)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straw Species</td>
<td>Rice Straw</td>
<td>Wheat straw</td>
</tr>
<tr>
<td>Mean MC</td>
<td>22.40</td>
<td>19.36</td>
</tr>
<tr>
<td>Coefficient of Variation</td>
<td>48%</td>
<td>21%</td>
</tr>
</tbody>
</table>

4.2.2. Effect of test method

Both DVS method and desiccator methods achieved similar moisture content result for the two species of straw. The experimental results of the DVS method and desiccator method are not statistically different based on the t-test. Because the
differences are small, they are likely to be associated with experimental error and
with significantly different sizes and quantities of specimen and natural variations
in the material.

The DVS method and desiccator method achieved variations of moisture content
of the two straw species at different RH levels. There was a much greater variation
of moisture content of the rice straw in the desiccator method than wheat straw
following the dessicator method. There are two possible reasons for this. Firstly,
differences in microstructure may affect the rate of adsorption of the two straw
species. The irregular shaped bundles of cells of rice straw may be responsible
for the larger variation of moisture adsorption of the rice straw specimens.
Secondly, only 3 specimens of wheat straw and rice straw were used in each
dessicator container. This limited number of specimens required by the standard
might be fewer that needed to account for the structural variations that occur in
natural materials.

Compared with the desiccator method, the DVS method produces data with much
smaller variations. The moisture content variations are less than 2\% in all relative
humidity conditions for each adsorption isotherm experiment. The DVS method
also features larger quantity of data in producing sorption isothermal model of the
specimen. There are total 20 data points are collected in the DVS method
comparing with 2 moisture content data of specimen in the desiccators method.
Considering the advantages of the DVS method, this research analyzes the data
collected from the DVS method rather than the dessicator method in the following
sections.

4.2.3. Effect of end capping on sorption isotherm

The open ended straw specimens showed larger variations of moisture content in
the monolayer sorption and multi-layer sorption than the sealed end specimens
Open ended rice straw showed larger variation of moisture content than open ended wheat straw during multi-layer sorption between 50%RH to 70%RH. However, the larger variation was not observed in the sealed end cycles. The sealed end cycles for rice straw and wheat straw showed significantly lower variation in monolayer sorption and multi-layer sorption. Below the fibre saturation point there was little difference seen between the two species, whilst above that point differences were more marked. The different cross sections of the straw species are likely to have an impact on water sorption properties of wheat straw and rice straw. During monolayer sorption and multi-layer sorption, access to the cut ends of the straw may be the reason for the variation in the water adsorption property of rice straw and wheat straw. Around the fiber saturation point, the cross section of the two straw species may have ability to minimize the variation in water sorption observed in the two straw species. Because the results were based on one sample of each straw species, further research will need to focus on a larger quantity of specimens.

Although the ultimate moisture content of both open ended and sealed straw were found to be similar, the kinetics of adsorption were different. This is significant because in real situations, air humidity levels fluctuate and moisture content of straw may not reach fully stability. As a result true moisture adsorption/desorption will not be expected to map onto a model of moisture based on a stabilized sorption isotherm, and differences between sealed and open ended straw will be more marked.
Figure 9. Moisture content variation of adsorption process of each specimen versus RH in the DVS

Using the DVS method, RH increments are changed according to a set time period, and this does not allow full stability to be achieved (Figure 10). At 95% RH, the sealed end straw specimens showed a slower rate of mass change than the unsealed straw specimens at the beginning of the exposure. Both rice straw and wheat straw show slower response to RH change in the DVS method with sealed ends. However, because of the limited duration of time set, the specimens did not reach full saturation at the 95% RH level. At the final stages of exposure, \( \frac{dm}{dt} \) of the specimens was between 0.0036-0.0038 g/min which is greater than the set
point for incremental change used by the DVS at humidity levels below 70% (0.002g/min).

Figure 10. Mass change of rice straw (left) and wheat straw (right) in the 95% RH in the DVS method.

In the 5%RH to 65% RH range, this study used a dm/dt rate of below 0.002g/min as the set point for the achievement of equilibrium and to initiate a change in RH levels. The time taken to achieve equilibrium was similar for both rice straw and wheat straw at each set point. There were, however differences in the kinetics when comparing open ended straw with sealed end straw. On average it was found that over a full cycle of 0% to 65%, the open ended specimens achieved equilibrium 300 minutes (75%) more rapidly than the sealed ended specimens (Figure 11). This is assumed to be because access of humidity to the pores is only through one face of the stem wall for the sealed end specimens, whereas for the open ended specimens the pores can be accessed from both sides of the stem wall.
Figure 11. Dm/dt of Rice straw without end-capping (left) and Rice straw with end-capping in 0-65%RH

The results of the DVS method show that the open ended straw reach equilibrium quicker than the closed end straw in all RH levels. The effect of open ended straw will likely depend on the relative ratio of exposed ends to the predominant surface area of the stem wall, and therefore the aspect ratio of the straw. The aspect ratio of the specimens in this investigation are not of typical in straw bale constructions and therefore the findings still need to be validated through further research on moisture movement of full scale straw bale walls. However, this finding may contribute to selection of the stacking method of straw bales in different climatic conditions, and modifying the makeup of a straw bale specifically for construction purposes.

4.2.4. Suitability of Sorption Models

Reviewing the experimental results of adsorption isotherms in the DVS method, the sorption isotherm of rice straw and wheat in this research closely map the equation of Lawrence et al (11).
At all RH levels in the DVS method, moisture contents of rice straw and wheat straw show 1%-2% less moisture content than that given by the equation of Lawrence et al (11). Lawrence et al based their equation on full equilibrium at each RH set point, but the data in the present study are based on accepting an equilibrium when $dm/dt < 0.002g/min$ up to 70% and on a set time above that RH level. This introduces a systematic error into the system. Since the primary value of sorption isotherms is to model performance, it is proposed that the use of a more rapid kinetic, such as the one used in this study, is more realistic than to use data achieved from complete equilibrium. A modification to the Lawrence equation to account for this change in the kinetic can be achieved by simply changing the constant 'n' in equation (1) from 44 to 54. The Lawrence equation fitted the moisture content of specimens of desorption cycles of the DVS method. The modified equation would be used to predict the moisture content of wheat straw and rice straw in the sorption cycle and the Lawrence equation can be used to predict the moisture content of wheat straw and rice straw in the desorption cycles. Applying a modified equation results in a isotherm which is much closer to the data produced by this study, and which more closely approximates to non steady state conditions likely to be encountered in straw bale buildings. (Figure 13).
Figure 13. Proposed equation with results of DVS method and equation of Lawrence et al. (11)

5. Conclusion

Moisture content of straw is central to the evaluation of durability of straw bale constructions. Existing sorption isotherms of straw provide a non-intrusive method to predict straw moisture content. This paper uses both DVS method and desiccator method to produce isotherms for rice straw and wheat straw. This study compares the cross-section of rice straw and wheat straw and establishes the impact of the differences on their moisture sorption properties. This study has shown that there are notable micro-structural differences between wheat straw and rice straw. Experimental results suggest that the physical
differences between wheat straw and rice straw have a negligible impact on the
equilibrium moisture content of the two straw species. Irrespective of straw
species, water sorption characteristics of the two straw species is similar. Open
dended straw can equilibrate up to 25% more rapidly with the environment than
closed ended straw, and could vary with differing aspect ratios of straw. The
implication of this is that straw bales which have been trimmed to size (resulting
in single strands of straw across the width of the bale) will equilibrate more rapidly
than straw bales which consist of straw which has been folded at the edge of the
bale. Bales made with cut ended straw may therefore have greater moisture
buffering capacity than bales made with folded straw. In the long term, it would be
expected that trimmed bales would therefore have less durability. A modified
isotherm equation is proposed on basis of the experimental results of the DVS
method. The modified equation more closely models water sorption of straw on
basis of real situation. The Lawrence equation would fit water desorption of straw
on the situation in this research.

Understanding of the water sorption isotherm of rice straw can ensure a high
degree of confidence in the hygroscopic condition in straw-bale wall constructions
made from rice straw. Further research is required to fully understand the impact
of the difference between closed and open ended straw on durability and sorption
kinetics.

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