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ORIGINAL ARTICLE

The effects of chemical components and particle size on the mechanical properties of binderless boards made from oak (*Quercus* spp.) logs degraded by shiitake fungi *(Lentinula edodes*)

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Abstract
Binderless boards are composite boards that rely on self-bonding mechanisms for inter-fibre bonding. *Quercus acutissima* and *Quercus serrata* logs degraded by *Lentinula edodes* (shiitake fungi) were used in this study to investigate whether physical and chemical changes induced by shiitake fungi can enhance board mechanical properties. Binderless boards were manufactured with 0.8 g/cm^3 target density, 220 °C pressing temperature, 5 MPa pressure, and pressing duration of 10 min. Boards made from logs degraded for ≥ 26 months were stronger than control boards and met modulus of rupture (MOR) and internal bonding (IB) requirements for fibreboards. Chemical composition and particle size distribution of the wood powder used to make the boards were determined to elucidate the drivers of board mechanical properties. The proportion of small particles (< 150 μm) showed a strong positive correlation with MOR for both species and hot water extractives showed a strong positive correlation with IB for *Q. acutissima* boards. Introduction of shiitake fungi pre-treatment to the production process may enhance the mechanical strength of binderless boards.

Keywords Binderless board · Internal bonding · Shiitake · Degradation

Introduction

Wood-based composites are produced by combining wood strands, particles, fibres, veneers, or thin boards with adhesives or by other fixation methods. Medium density fibreboards (MDFs) have replaced traditional lumber in segments of the furniture and interior industry due to their relatively low cost and use of by-products from the lumber production process [1]. However, urea formaldehyde, a synthetic binder used for the production of MDFs and particleboards, may be carcinogenic and accounts for a large portion of manufacturing costs [2]. Binderless boards, a type of MDF that does not use synthetic binders, could potentially be a commercially viable and environmentally-friendly product in the MDF sector.

Research on binderless boards has coincided with the drive for agro-industrial waste recycling and the development of ecomaterials. As such, the feasibility of using various agro-industrial waste to manufacture binderless boards has been investigated. Lignocellulosic agro-industrial by-products, such as inter alia, kenaf core [3], oil palm biomass [4], moso bamboo [5], and rice straw [6] have been used to manufacture binderless boards. Although the exact determinants of the self-bonding mechanism are yet to be elucidated, the thermosetting behavior of lignin at elevated temperatures [7], polymerization of hot water extractives [8], particle size [9], and lignin–furfural resin formation [10] have been cited as possible causes of self-bonding between composite fibres.

Quercus spp. (oak) logs degraded by *Lentinula edodes* (shiitake fungi) were selected as the materials for this study because wood degraded by shiitake fungi has the merits of being a waste material from the mushroom cultivation process and a product of natural lignocellulosic enzymatic systems. The effect of enzyme pre-treatment on wood fibres for the production of binderless boards has been investigated [11], but the focus was on laccase, an oxidase enzyme that is specific to phenolic molecules in wood substrates. In

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addition to ligninolytic enzymes, shiitake fungi also contain cellulose and hemicellulose degrading enzymes that reduce polysaccharides to low molecular weight monomer and oligomer sugars for uptake [12]. There are many shiitake fungi induced chemical and physical changes in spent logs, two of which may potentially be favorable for binderless boards: (1) increase in hot water extractives, which can promote polymerization [8] and cross linkage [10] between various chemical components during the hot press process and (2) structurally weakened cells that may breakdown into smaller particles during the milling process, which can lead to a better particle packing system [9] with less voids and enhance stress transfer [13]. Furthermore, there is a large volume of waste logs produced by the mushroom cultivation industry [14]. Despite sawdust having a greater share of wood purchased for shiitake mushroom cultivation, spent logs were chosen as the materials for this study for better clarity over species origin and consistency in growing conditions, an important consideration for chemical component and particle size analysis.

The key research goal is to determine the mechanical properties of binderless board that have been made from wood degraded by shiitake fungi for varying durations during the mushroom cultivation process and how shiitake fungi induced changes in wood affects mechanical properties and internal bonding mechanisms of binderless boards.

Materials and methods

Materials

The samples were provided by MUSH-Shimura Noen in Shizuoka. The farm uses *Quercus acutissima* and *Quercus serrata* branches that are less than 11 years and have a diameter of c. 150 mm as log substrates for shiitake mushroom cultivation. The logs are typically felled in February and inoculated with shiitake spawn in March. Fruiting is stimulated in September by soaking the logs in cold water, after which mushroom harvesting starts. The logs are kept in greenhouses throughout winter. Mushroom yield varies throughout the cultivation period, but generally increases up to 2 years and then decreases; mushroom yields of this farm generally increased by c. 1.5 kg per log around 2 years and then decreased. The yield recovers by the third year when the bark softens sufficiently for shiitake fungi degradation. The logs are discarded after the fourth year of cultivation due to significant decreases in yield.

Undegraded logs and logs degraded by shiitake fungi for 2, 14, 26, 38, and 50 months were used as materials for this study. They were sterilized in an autoclave at 121 °C for 1 h and dried in an oven at 105 °C until the moisture content (MC) reached 0%. The logs, including inoculation points and spawn, were debarked, converted to wood powder (WP) using a Wiley Mill (WT-150; Miki Seisakusho, Japan) with a 1 mm screen mesh, and stored as a mix of homogenized WP. The hardness and density of the logs varied with degradation duration, a proxy for the degree of degradation; consequently, the time required to powderize the wood differed. Approximately 500 g of each sample was powderized to produce WP; WP that did not pass through the 1 mm screen mesh (less than 5% by weight) was excluded as waste WP. WP for the manufacture of boards and chemical analyses were sampled from the same mixture.

Chemical composition

Alcohol benzene extractives, hot water extractives, holocellulose, α cellulose (cellulose), and hemicellulose content was determined in accordance with the Wood Science Experiment Manual [15]. Lignin [16] and ash content [6] was determined in accordance with previously reported methods.

Alcohol benzene extractives were extracted by the soxhlet extraction method for 6 h. Hot water extractives were removed by heating the sample in a water bath at 100 °C for 3 h. Holocellulose was isolated by bleaching WP without extractives (defatted wood) in a water bath at 75 °C for 3 h; sodium chlorite and acetic acid were used as the bleaching agent and catalyst. Cellulose was isolated by mixing 17.5% NaOH aqueous solution with the sample in a water bath. Lignin content was determined by adding acid insoluble lignin isolated by 72% sulphuric acid (Klason Lignin) and acid soluble lignin (ASL) content. Klason Lignin was isolated by adding 72% sulphuric acid to defatted wood. The sample was put in a water bath for 3 h and stirred frequently. The concentration of the sample was reduced to 3% and treated in an autoclave at 121 °C for 30 min. ASL content was determined spectrophotometrically by observing its absorptivity at 205 nm. Ash was isolated by heating (oxidation) the sample in an electric furnace at 700 °C for 3 h.

Particle size distribution

WP was separated into different particle size classes using an orbital sieve shaker (Electromag Sifter Pot Mill, Itoh, Japan) with sieve apertures of 500, 355, 250, and 150 μm. Approximately 10 g of WP was processed through the machine for 30 min. The particle size distribution of WP samples was determined by weight.

Board manufacture

Binderless boards were manufactured according to a previously reported method [3]. The amount of wood particles used to manufacture each board was determined according to the target dimensions of the board (300 × 300 × 5 mm), target

density (0.8 g/cm^3), and MC of WP. The average MC of WP was 7% and the minimum and maximum MC of WP was 4 and 8%, respectively; the MC of WP ranged from 6 to 8% when the sample with MC of 4% was excluded. The relationship between MC of WP and the mechanical properties of the boards was examined, but there was limited correlation.

Binderless boards were made by dispersing WP to form a homogenous single-layer mat within a 300 × 300 mm forming box. The mat was pressed at 220 °C and 5 MPa for 10 min in a hot press machine (Model 1569; Komatsu, Japan). The relationship between actual board density and the mechanical properties of the boards was examined, but there was limited correlation.

Evaluation of board mechanical and physical properties

The modulus of rupture (MOR) and internal bonding (IB) tests were conducted with an universal testing machine (Model 4204; Instron; Canton, Massachusetts, USA) and in accordance with the guidelines in JIS Fiberboard Standard 5905-2014 [17]. Five specimens were tested and used to compute the average sample MOR and IB values and sample standard deviation.

Linear models were used to conduct correlation analysis on the mechanical properties of the boards, chemical components, and proportion of WP in various size classes. Additional statistical analyses were conducted to examine the effects of degradation duration and tree species on board mechanical properties. Analysis of variance (ANOVA) and all-pairwise comparisons tests (Tukey HSD) were conducted to investigate the variance within and between groups and to determine statistically homogenous groups.

Results and discussion

Mechanical properties of the boards

Figure 1 shows the results of the MOR test. The MOR values of both *Q. acutissima* and *Q. serrata* control boards (*Q. acutissima*: 3.0 N/mm^2 and *Q. serrata*: 3.2 N/mm^2) were much lower than the MOR of boards made from logs degraded for ≥ 14 months. A gradual increase in MOR with degradation duration was observed. The highest MOR values were obtained from 38-month samples (*Q. acutissima*: 8.5 N/mm^2 and *Q. serrata*: 8.4 N/mm^2). MOR of boards made from logs degraded for 50 months (*Q. acutissima*: 6.0 N/mm^2 and *Q. serrata*: 6.4 N/mm^2) was lower than 26-month and 38-month samples. The ANOVA test indicated that degradation duration had the greatest influence on MOR ($p < 0.0001$), while tree species did not have a statistically significant effect on MOR ($p = 0.8006$). The Tukey

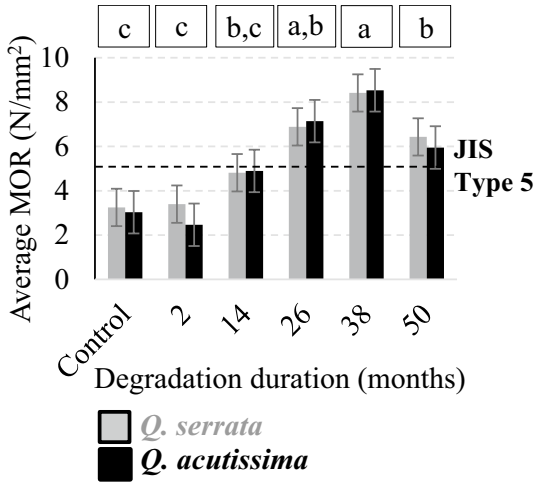

Fig. 1 Degradation duration of raw materials and MOR of boards made from *Q. serrata* and *Q. acutissima* WP. Bars indicate standard error and means marked with different letters (a, b, c) are significantly different at $p < 0.05$ (Tukey HSD). Line marked as JIS Type 5 represents JIS standard threshold [17]

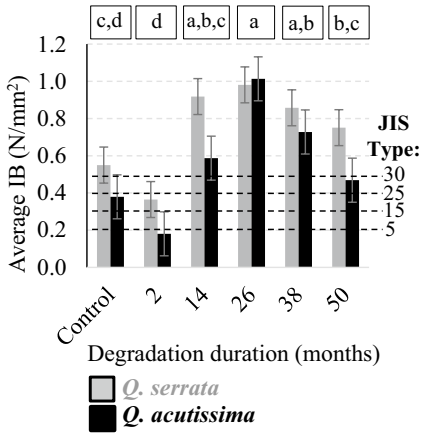

Fig. 2 Degradation duration of raw materials and IB of boards made from *Q. serrata* and *Q. acutissima* WP. Bars indicate standard error and means marked with different letters (a, b, c, d) are significantly different at $p < 0.05$ (Tukey HSD). Lines marked as JIS Type 5–30 represent JIS standard thresholds [17]

HSD test indicated that boards made from logs degraded for ≥ 26 months were statistically distinct from the control boards.

Figure 2 shows the results of the IB test. The lowest and highest values were obtained from boards made from logs degraded for 2 months (*Q. acutissima*: 0.18 N/mm^2 and *Q. serrata*: 0.36 N/mm^2) and 26 months (*Q. acutissima*: 1.01 N/mm^2 and *Q. serrata*: 0.98 N/mm^2), respectively. The ANOVA test indicated that degradation duration and tree species had a statistically significant effect on IB (p for degradation duration and tree species were < 0.0001 and 0.0031, respectively). The Tukey HSD test indicated

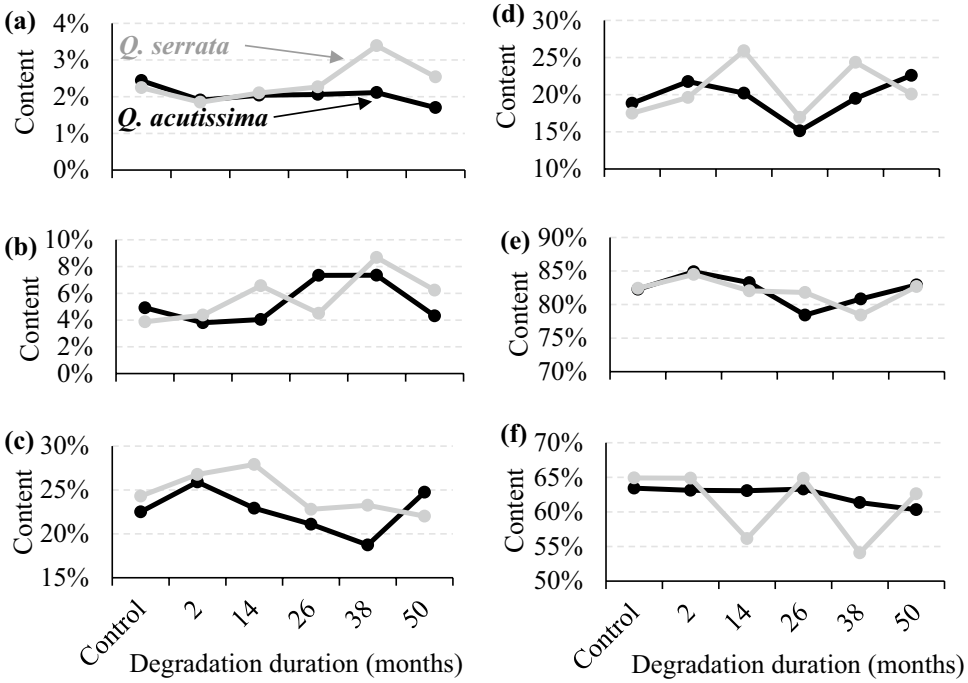

Fig. 3 Chemical content profiles of individual chemical components of *Q. serrata* and *Q. acutissima* WP (the *y*-axis (content) denotes proportion of each chemical component in WP); **a** alcohol benzene extractives; **b** hot water extractives; **c** lignin; **d** hemicellulose; **e** holocellulose; and **f** α cellulose

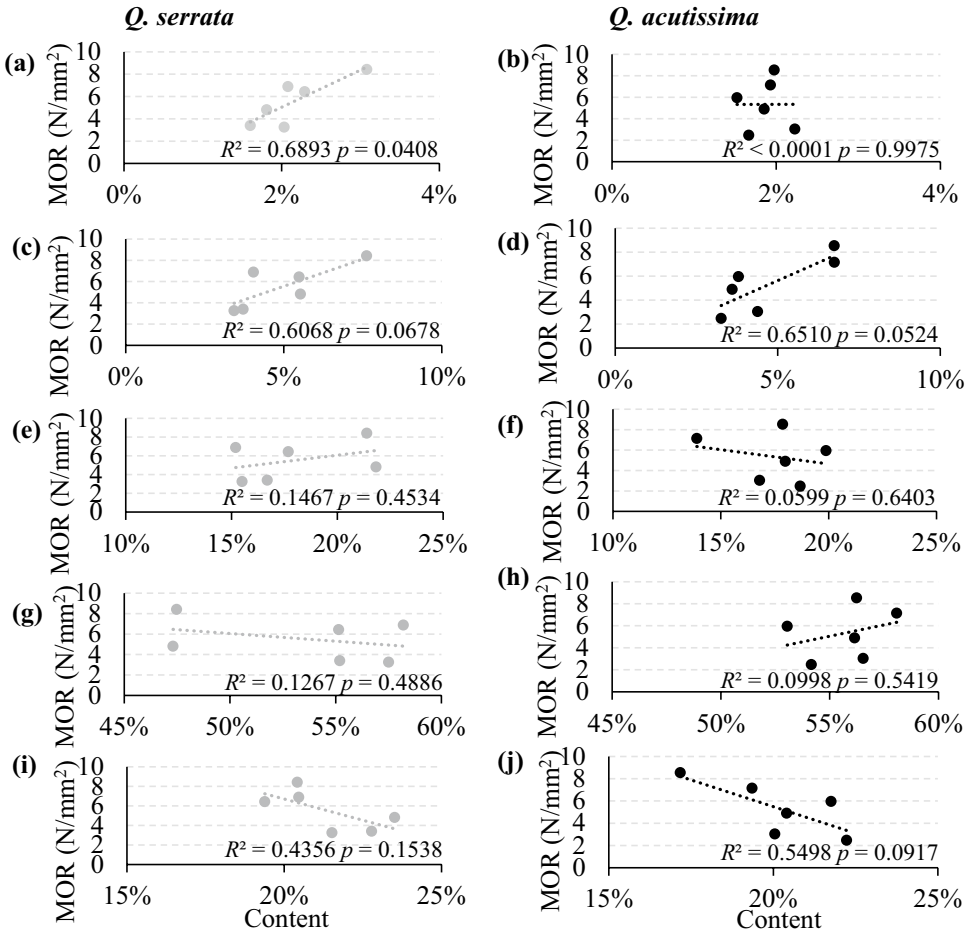

Fig. 4 Relationship between MOR of boards and individual chemical components of WP, on a species by species basis [the *x*-axis (content) denotes proportion of each chemical component in WP]; **a, b** alcohol benzene extractives; **c, d** hot water extractives; **e, f** hemicellulose; **g, h** α cellulose; and **i, j** lignin

that boards made from logs degraded for 26–38 months were statistically distinct from the control boards.

Both species exhibited similar trends, where boards made from logs degraded for longer periods had higher MOR and IB values than the control. It is important to note that the logs do not represent a temporal study of changes to the same logs over 50 months, because they were inoculated in different years. The observed decrease in the 50-month samples may be due to a lower degree of degradation for that particular batch (due to unique climate, greenhouse, or handling conditions when they were inoculated, in 2012), rather than a reverse in degradation trends. The ANOVA test indicated that degradation duration had a statistically significant effect on MOR and IB. The Tukey HSD test indicated that boards made from logs degraded for longer durations (≥ 26 months for MOR and 26–38 months for IB) were statistically distinct from control boards. These results suggest that the shiitake fungi degradation process induces changes in the oak logs that enhance binderless board mechanical properties.

In addition, JIS sets minimum MOR and IB requirements for fibreboards [17]. Boards made from logs degraded for ≥ 26 months exceeded the JIS Type 5 MOR threshold of 5.0 N/mm^2, but the control boards and boards made from logs degraded for ≤ 14 months did not meet the requirements. All boards, with the exception of *Q. acutissima* boards made from 2-month WP, met the JIS Type 5 IB threshold of 0.2 N/mm^2. Boards made from logs degraded for 14–38 months even met the IB threshold for JIS Type 25 boards of 0.5 N/mm^2.

Possible factors affecting board mechanical properties

Chemical components

The chemical composition of *Q. serrata* and *Q. acutissima* WP was investigated (Fig. 3). The chemical composition of WP from logs degraded for differing durations did not show

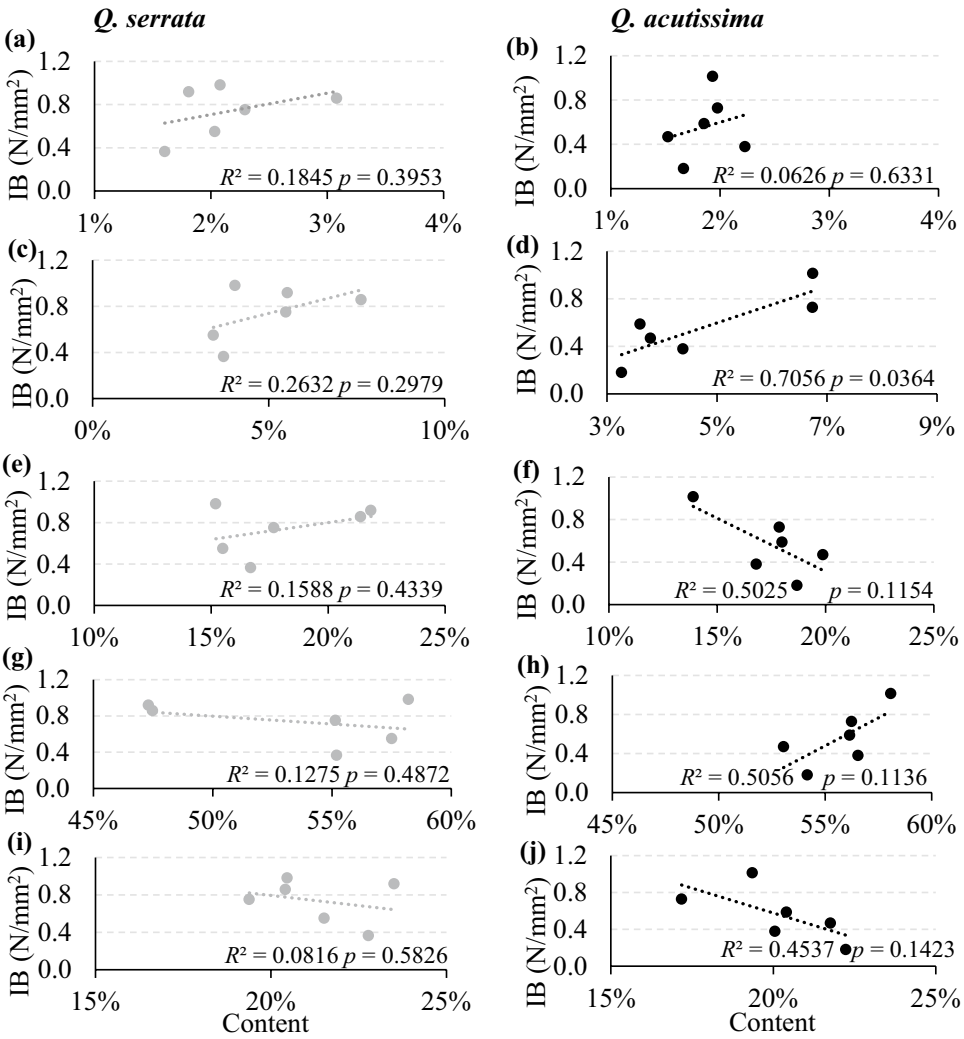

Fig. 5 Relationship between IB of boards and individual chemical components of WP, on a species by species basis (the *x*-axis (content) denotes proportion of each chemical component in WP); **a, b** alcohol benzene extractives; **c, d** hot water extractives; **e, f** hemicellulose; **g, h** α cellulose; and **i, j** lignin

any consistent trends. Figure 3 suggests that the chemical components of shiitake degraded wood is not a time-dependent linear process. This is supported by previous studies, where different degradation rates and changes in the degradation rates were observed for each chemical component throughout the degradation process [12]. Consequently, the effect of time was excluded from the correlation analysis (Fig. 4).

The effect of chemical composition on MOR was investigated (Fig. 4). The correlation analysis indicated that hot water extractives content had a moderate positive correlation with MOR for both species (*Q. serrata:* $R^2 = 0.6068$, $p = 0.0678$; *Q. acutissima:* $R^2 = 0.651$, $p = 0.0524$) and alcohol benzene extractives also had a moderate positive correlation with MOR, but for *Q. serrata* only ($R^2 = 0.6893$, $p = 0.0408$).

Natural variability in hot water extractives content, ranging from 3.3 to 8.0%, has been reported for undegraded Japanese oak wood [18]. It has also been reported that shiitake degraded *Quercus mongolica* logs had higher hot water extractives content than undegraded logs [19]. The chemical composition of *Q. acutissima* and *Q. serrata* control samples in the present study are similar (Fig. 3), but differ greatly for degraded samples. Since shiitake fungi degrades wood polysaccharides into sugars for uptake [20], changes in hot water extractives content likely occur throughout the degradation process; it may either increase due to inefficient uptake of monomer and oligomer sugars from the breakdown of hemicellulose and cellulose, or decrease because the monomer and oligomer sugars are easily absorbed by the fungi. Nevertheless, the hot water extractive content of logs degraded for 2–50 months is likely at least partially driven by the shiitake degradation process. Furthermore, MOR is a measure of bending strength, which is positively impacted by the bonding of surface particles and negatively impacted by surface imperfections. Polymerization of monomer sugars, melting and filling in lumen voids, and crosslinking between the carbohydrates and lignin have been cited as possible ways hot water extractives promote internal bonding [8]. Previous studies that added glucose and sucrose to control samples found that the xylose/arabinose ratio decreased, indicating that the monomer sugars polymerized during the hot press process [8]. Mechanical interlocking and fusion of fibres were observed in the morphological analyses of the boards. Melted sugars, which subsequently filled in the cell lumen void areas or acted as an adhesive fibre coating, were cited as a possible cause; this was further supported by the increase in lignin and cellulose content [8]. The results demonstrated that the addition of 20% glucose or 20% sucrose improved the MOR and IB of binderless boards made from oil palm trunks by 1.5–2 times [8]. The maximum difference in hot water extractives content in this study was 4% and a strong positive correlation with MOR was observed, though the effect of hot water extractives was not isolated.

The results also showed a strong positive relationship between MOR and alcohol benzene extractives for *Q. serrata* samples (Fig. 4). Tannin, a substance classified as an alcohol benzene extractive (though at times also classified as a hot water extractive), is a key distinguishing feature between the chemical composition of *Q. serrata* and *Q. acutissima*. Tannin found in *Q. serrata* is predominantly comprised of hydrolyzable tannin, whereas *Q. acutissima* contains condensed tannin [21]. Hydrolyzable tannins are derivatives of gallic acid, which are esters linked to glucose. Incidentally, formic and acetic acid are released from lignocellulosic materials during the first stage of the hot press process [22]. Hydrolyzation of *Q. serrata* tannin during the hot press process may release glucose and enhance MOR through the abovementioned mechanisms [8, 10].

The effect of chemical components on IB was investigated (Fig. 5). IB showed a strong positive correlation with hot water extractives content ($R^2 = 0.7056$, $p = 0.0364$) and a moderate positive correlation with cellulose content ($R^2 = 0.5056$, $p = 0.1136$) for *Q. acutissima* samples. As mentioned previously, polymerization, melting and filling, thermal softening, and crosslinking are possible ways hot water extractives promotes internal bonding [8, 10]. However, the results indicate that IB has a strong positive relationship with hot water extractives for *Q. acutissima* samples

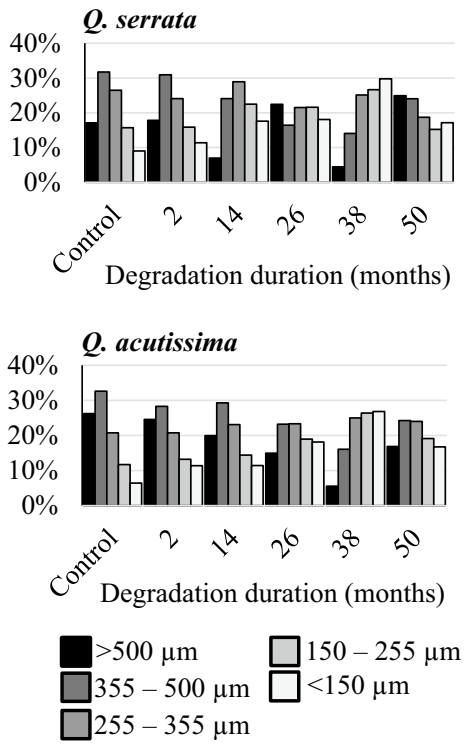

Fig. 6 Particle size distribution for *Q. serrata* and *Q. acutissima* WP

only (Fig. 5). It can be observed from Fig. 3 that hot water extractives content of the two species begins to diverge from 14 months.

Shiitake fungi have been reported to degrade the cell walls of fibres and rays differently [23]. Fibre cells are degraded from the cell lumen out. Rays are degraded in a similar fashion initially, but the presence of an amorphous layer (defined by the author as a layer comprised of a protective layer and an isotropic layer that contains hemicellulose, pectic substances, cellulose microfibrils, and lignin) that follows a thin layer of inner cell wall prevents shiitake fungi from further degrading the cell wall from this direction [23]. Shiitake fungi then begins to degrade the cells from the middle lamella [23], a lignin-rich layer that binds the cells together. *Q. acutissima* has a higher percentage of ray cells, 19.7% compared to 9.7% for *Quercus crispula*, a species that is anatomically very similar to *Q. serrata* [24]. The differences in the hot water extractives and lignin content profiles exhibited in Fig. 3 may be due to the abovementioned differences in shiitake fungi behavior and anatomical features of the tree species: shiitake fungi are better able to degrade *Q. serrata* polysaccharides because of a lower proportion of ray cells, resulting in a higher concentration of hot water extractives content in the logs at an earlier stage; conversely, due to a higher proportion of ray cells, shiitake fungi are required to degrade the lignin-rich middle lamella in *Q. acutissima* logs before it can access the polysaccharide-rich cell wall.

Particle size distribution

The particle size distribution of *Q. serrata* and *Q. acutissima* WP was investigated (Fig. 6). The results show that WP from logs degraded for longer durations had a higher proportion of < 250 µm WP than the control and 2-month WP. The proportion of 355–500 µm WP showed the reverse trend; it was higher for the control and 2-month WP, but markedly lower

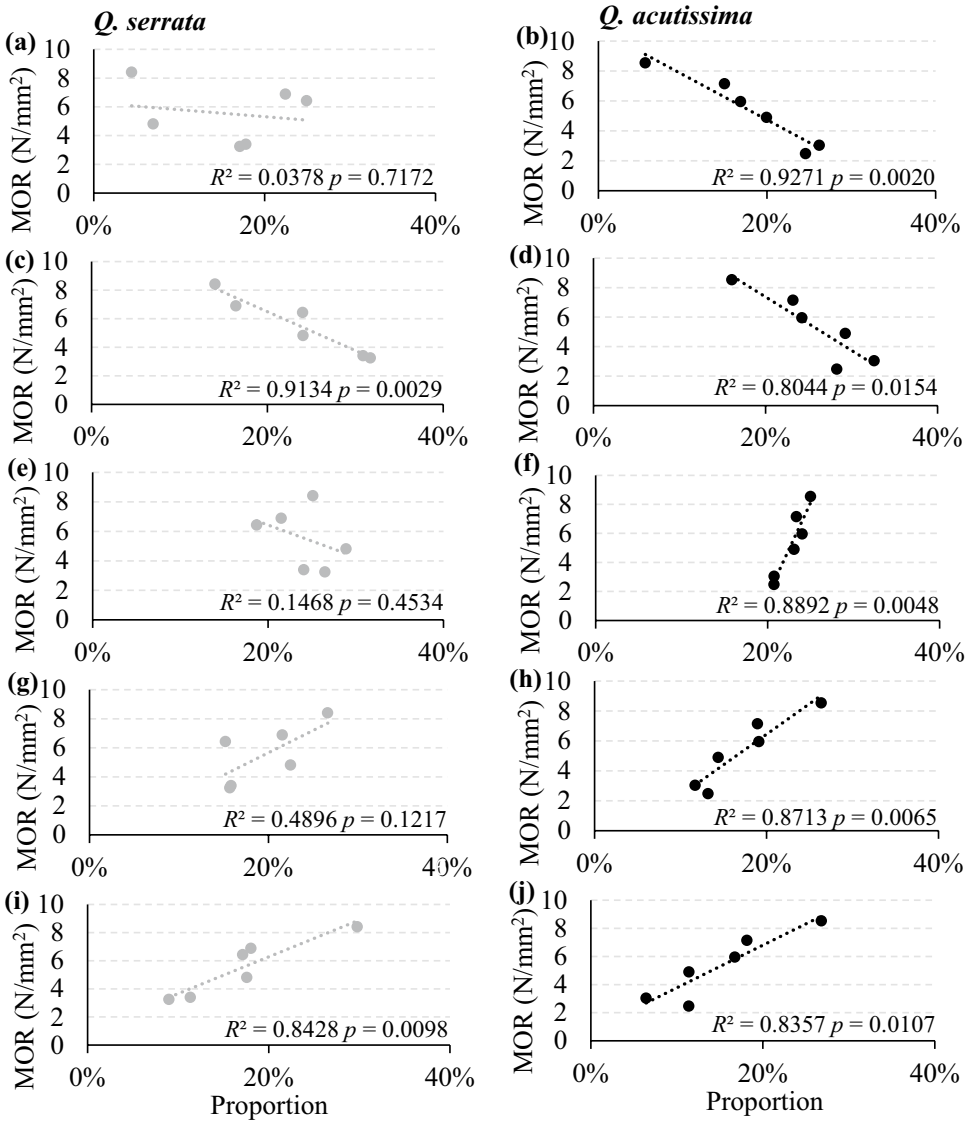

Fig. 7 Relationship between MOR of boards and the proportion of different sized particles of WP, on a species by species basis; **a, b** particles > 500 µm; **c, d** particles between 355 and 500 µm; **e, f** particles between 250 and 355 µm; **g, h** particles between 150 and 250 µm; and **i, j** particles < 150 µm

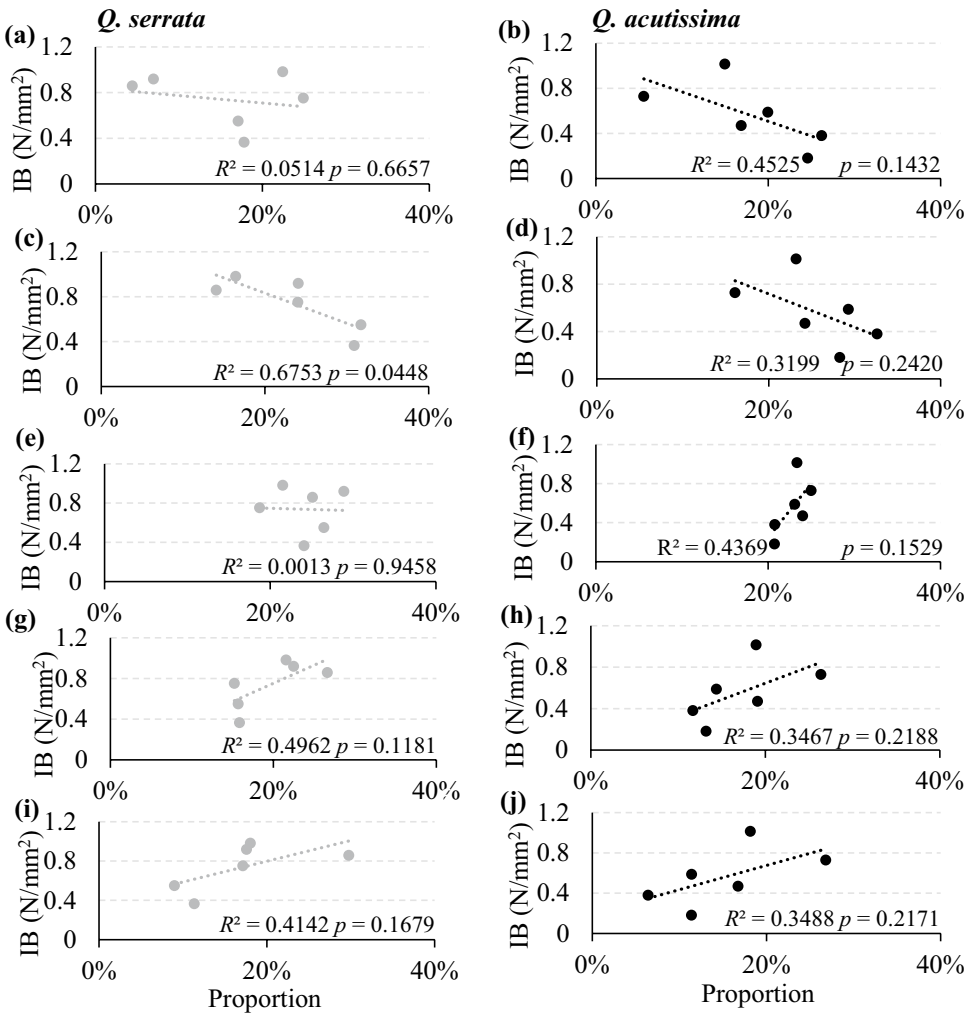

Fig. 8 Relationship between IB of boards and the proportion of different sized particles of WP, on a species by species basis; **a, b** particles > 500 µm; **c, d** particles between 355 and 500 µm; **e, f** particles between 250 and 355 µm; **g, h** particles between 150 and 250 µm; and **i, j** particles < 150 µm

for WP from logs inoculated for longer durations. Shiitake fungi do not degrade all parts of a cell wall uniformly [23] and the voids created by the fungi become structural weaknesses of the cell that may potentially result in the collapse of cell walls during the milling process. Since the proportion of attacked cells increases with the degree of degradation, cells of logs that have a higher degree of degradation may have a higher tendency to collapse during the powderization process, thereby resulting in a larger proportion of small particles (< 250 µm) in the WP mixture.

The effect of particle size distribution on MOR was investigated (Fig. 7). The proportion of small particles (< 150 µm) had a very strong positive correlation with MOR for both species (*Q. serrata:* $R^2 = 0.8428$, $p = 0.0098$; *Q. acutissima:* $R^2 = 0.8357$, $p = 0.0107$), whereas relatively large particles (355–500 µm) had a very strong negative correlation with MOR (*Q. serrata:* $R^2 = 0.9134$, $p = 0.0029$; *Q. acutissima:* $R^2 = 0.8044$, $p = 0.0154$).

The effect of particle size distribution on IB was investigated (Fig. 8). Overall, the particle size distribution of WP had a moderate effect on IB, with the proportion of 355–500 µm WP, in particular, having a strong negative correlation with IB for *Q. serrata* samples (R^2 of 0.6753 and p value of 0.0448).

The results of the particle size distribution analysis suggests that the physical characteristics of WP has a greater influence on MOR and IB than the chemical composition of the raw materials. A greater proportion of small particles may enhance particle packing [9] and decrease total surface void area [13], thereby enhancing stress transfer among the fibres and increasing MOR. Particle packing may also decrease the amount of voids in the matrix and increase the number of internal bonding sites, thereby improving IB [9].

The aim of the present study was to examine the effects of shiitake fungi degradation on the mechanical properties of binderless boards. Additional studies which isolate the influence of particle size distribution on board mechanical properties should be conducted by artificially changing the particle size distribution of undegraded *Quercus* spp. logs to similar ratios observed in the 26 or 38-month samples of

the present study. Further studies should elucidate the degree to which each chemical component and particle size class affects board mechanical properties.

Conclusion

Binderless boards made from *Q. acutissima* and *Q. serrata* logs degraded by shiitake fungi for 26, 38, and 50 months during the mushroom cultivation process had higher MOR and IB values than the control. Binderless boards made from wood degraded for 26 (*Q. acutissima*: 1.01 N/mm^2 and *Q. serrata*: 0.98 N/mm^2) and 38 (*Q. acutissima*: 8.5 N/mm^2 and *Q. serrata*: 8.4 N/mm^2) months exhibited the strongest IB and MOR, respectively. Insights into possible factors driving the MOR and IB results were also gained.

MOR was influenced by a variety of factors, including degradation duration, hot water extractives content, lignin content, IB, and particle size distribution. In particular, there was a very strong positive and negative correlation between MOR and the proportion of small (< 250 μm) and large (> 500 μm) WP, respectively. The greater number of contact points between fibres and a more efficient particle packing system are possible reasons smaller particles improve board bending strength.

Degradation duration and tree species had a statistically significant effect on IB. Hot water extractives content had a strong positive correlation with IB for *Q. acutissima* samples, but there was limited correlation between IB and the other chemical components and particle size distribution. A big IB difference between the control board and boards made from wood degraded for more than 2 months was observed.

Boards made from logs degraded for ≥ 26 months exceeded the JIS Type 5 MOR and IB thresholds [17] of 5.0 and 0.2 N/mm^2, respectively.

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