Mechanism and effect of alkoxy silanes on the restoration of decayed wood used in historic buildings

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ABSTRACT

It was necessary to protect and restore the decayed wood in the ancient buildings, in which reinforcing and restoring the damaged components with in situ and non-destructed strategy was preferred, following the heritage protection principles. For this purposes, series experiments were designed and performed to investigate the reaction mechanism and effects of ethyl orthosilicate and methyl triethoxysilane toward the decayed wood components sampled from Shuiyu Village, a historical tribe in Beijing. The results demonstrated that the organosilicon layer was fabricated onto the wood cell walls by condensation reaction, in which the Si–O–Si bonds were formed between alkoxy groups, and the Si–O–C bonds were generated between cellulose fibers and organosilicon. The organosilicon layer contributed to further improve physico-mechanical performances like hydrophobicity, mechanical properties, thermal stability of treated wooden components, except a slight color deviation. In all, the matrix of the ethyl orthosilicate and methyl triethoxysilane is a reliable agent for retaining the decayed part of wood components, which accomplished the satisfied performance improvements.

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1. Introduction

Wood is widely used as the main structural material of buildings in China with a long history, which results in a large number of splendid and valuable architectural heritages [1]. Wood, a kind of natural biodegradable substance, will be decayed to shrinkage, crack by some organisms under suitable temperature and humidity conditions [2]. Therefore, maintenance and reinforcement for the decayed wood components of traditional wood structure are indispensable. The present conservation technologies for deterioration of wood components could be divided into two categories. One is traditional technology, which is to reseal the decayed part of components and add new wood billets on the scathed area [3,4]. The other one is to replace the decayed parts with sawdust and carbon fiber, and bond with epoxy resin [5–7]. But these are controversial, especially when the components (even they are decayed), are abundant in historical value, art value and skill value, and removing them detracts from the building’s historical identity and integrity [8,9]. Furthermore, the unstable new additions would also affect stability of building structures [10]. Therefore, it is especially important to explore new ways that could retain the original wood components [11].

The wooden structures exposed to moisture are predominantly eroded by fungi [12]. In particular white-rot fungi and brown-rot fungi are responsible for substantial damages of wood, which leads to decomposition of polysaccharides within lignin and cellulose texture of the cell walls from the lumen face, through the secondary wall, until the highly lignified middle lamella [13,14]. In this process, the cell walls, deprived of the cellulose and lignin, irreversibly collapse, resulting in chemical and physical changes of their components [15], and the wood will shrinks and cracks once unprotected. As a result, the method of consolidating decayed part of wood through the wood cell textures reinforced, which retain the original wood components, could be the best way to solve the problems of wood deterioration, compared with traditional technology. The method is mainly applied with infiltration consist of penetrating a special liquid material into wood and widely utilized
in conservation of decayed wood for their excellent performances of improving physical and mechanical properties [16,17].

However, for the decayed wood components of ancient buildings, some concerns should be taken into account when applying the method: hydrophobicity, mechanical properties, thermal stability and esthetics. The reasons are listed as the following: (1) The degradation of wood is mainly caused by excessive moisture which could affect wood dimensional stability and result in shrinkage or swelling. Therefore, the improvement of hydrophobicity seems to be the optimum solution to prevent wood from being decayed [13]. (2) As the underlying problem of moisture in decayed wood has been solved, it is essential to restore mechanical strength, so that the wood components could continue to perform their ability in decorative and structural functions. (3) In addition to restore mechanical properties of decayed parts, the method applied should also protect the wood from fire threat, which is crucial to the durability of components and the long-term retention. (4) According to the authenticity principles of heritage protection, a purpose of the conservation is, first and foremost, to preserve the original appearances of precious wood heritages [13]. Thus, the esthetics of treated wood should also be concerned.

However, up to now, the attentions in related research were much paid to dimensional stabilization, mechanical performance or overall appearances of treated wood [11,18–20], but less to the comprehensive study on the reaction mechanism and the concerns above that are especially important for conserving decayed components of ancient buildings. Herein, this research met the requirements in the field of restoration with a systematic investigation on the consolidation effects of decayed wood of ancient buildings in terms of hydrophobicity, mechanical properties, thermal stability and esthetics, based on the idea of restoring the decayed wood components and consolidating decayed parts in situ. As well, the reaction mechanism of restoration was clarified via means of various characterizations like scanning electron microscope, Fourier transform infrared spectroscopy and so on.

Generally, some organosilicon compounds were adopted to consolidate the decayed wood parts due to their mechanical properties, thermal stability, along with the ability to stabilize wood morphology by intensifying the cell walls [18,21]. In addition, they usually consist of several organic functional groups and readily hydrolysable alkoxy groups [22,23]. Their polymerizations are composed of hydrolysis and condensation reactions called the sol–gel process [24,25]. In the process, hydrolyzed alkoxy groups can combine with other hydrolyzed alkoxy groups by establishing stable Si–O–Si bonds, producing a three-dimensional network structure of gel, after alkoxysilanes molecules are activated by moisture [26]. As the water molecules evaporate, the gel will shrivel to different types of silicon flakes with both organic and inorganic properties like excellent scirrhosity, climate resistance, hydrophobicity and thermal stability [27]. In this work, the mixture of two typical alkoxysilanes like the ethyl orthosilicate (TEOS) and methyl triethoxysilane (MTES) was selected as consolidation agent to investigate their ability and mechanism to restore decayed wood of ancient buildings.

2. Materials and methods

2.1. Wood samples

The objects in the research were small cuboid samples cut out from the decayed parts of wood rafters that were made of poplar wood and extracted from some ancient buildings located in the Shuiyu Village. The village, situated in Fangshan District, Beijing, is a famous historical tribe with the history more than one hundred years, which was selected into the list of first batch traditional villages of Beijing in March 2018. The decayed wood samples, with the measured density of only 37% of sound wood and water content of 8.1%, were divided into two treatment groups. The samples applied for mechanism characterizations, thermal stability and esthetic tests with dimension of 5 × 20 × 20 mm (radial (R) × tangential (T) × longitudinal (L) direction) were labeled as group A. The samples used for hydrophobicity and mechanical tests with dimension of 15 × 15 × 15 mm (radial (R) × tangential (T) × longitudinal (L) direction) were labeled as group B (Fig. 1).

2.2. Chemical and reagents

Both TEOS and MTES were utilized in this experiment. In addition to the general properties of silicon materials, TEOS and MTES display the good performance on their own distinct characteristics. TEOS was selected due to its cross-linking and reinforce properties that results from hydrolysable ethoxy groups. MTES was selected for its potential hydrophobic characteristic (methyl groups) and tackify properties that originate from its active groups and chemical activities (Fig. S1). As well, the performance of the combination of two agents is very outstanding, which could meet the critical requirements for heritage restoration [28,29]. Furthermore, the two agent are most likely to be applied in practice in a relatively short time, for their controllable reaction procedure and massive relevant research data [30–32].

2.3. Characterizations

The weights of pre- and post-treated samples were measured to calculate weight percent gain (WPG) of wood samples, following Eq. (1):

\[ \text{WPG} = \frac{W_t - W_0}{W_0} \times 100 \]

Some characterizations techniques were utilized to investigate the reaction mechanism of restoration for decayed wooden components. The microcosmic appearances of wood samples were imaged with the scanning electron microscope (Hitachi TM3030 Plus) coupled with the EDS detector of Model 550i (to obtain the X-ray maps for silicon) at magnification of 1200–1800 times. Changes in chemical compositions of wood samples were probed by Fourier transform infrared spectroscopy (Nicolet 6700 Spectrometer) in the wave-number range of 4000–400 cm⁻¹. Changes in bond energy of wood samples were detected with X-ray photoelectron spectroscopy (Thermo ESCALAB 250Xi).

2.4. Property tests

Some instruments were used to assess the changes of physico-mechanical properties of treated wood. Hydrophobicity of wood samples was measured according to contact angle of water droplet on the surfaces of wood by the optical contact angle & interface tension meter (KINO SL200KS). Mechanical properties of wood samples were evaluated under pressure of 8 N by performance tester (DZS-III) of hard and brittle material. Thermal stability of wood samples was measured with thermal gravimetric analyzer (Bo Yuan DTU-3) in the range of 25–650 °C at a heating rate of 10 °C/min with air streaming. Esthetic variations of wood samples were calculated by precise color reader (FRU WR-18) with ΔE value.

2.5. Restoration experiment

Ethyl orthosilicate, methyl triethoxysilane, alcohol and water were mixed in a molar ratio (0.66:1:4.49:1.66), with pH being adjusted to 2.0 by 0.1 mol/L HCl solution. After the mixed solution was stirred for 10 min at 20 °C, pH was adjusted to 7.0 with...
in (OH)₂ Si (HO)₃SiOH

0.5 mol/L NaOH solution to form consolidation solution. Then, the samples were immersed in the consolidation solution for 20 min. After that, the treated wood samples were taken out of the solution and air-dried at room temperature for two days, the following characterizations and physico-mechanical tests were made individually. For comparison, equal amounts of untreated wood samples (decayed without impregnating the treatment solution) and sound wood samples (non-decayed without impregnating the treatment solution) were characterized and tested.

3. Results and discussion

3.1. Explorations of reaction mechanism

The sol–gel chemistry of TEOS and MTES have been well investigated [33]. After being activated by water molecules, TEOS and MTES will experience a series of hydrolysis and condensation reactions as Equations (1.1), (1.2), (2.1)-(2.3) [34,35].

1) Hydrolysis reaction

\[
\begin{align*}
\text{Si(OEt)}_4 + 4\text{H}_2\text{O} & \rightarrow \text{Si(OH)}_4 + 4\text{EtOH} \\
\text{Si(OEt)}_3 + 3\text{H}_2\text{O} & \rightarrow \text{Si(OH)}_3 + 3\text{EtOH} \\
\text{CH}_3 & \quad \text{CH}_3
\end{align*}
\]

(1.1)

(1.2)

2) Condensation reaction

\[
\begin{align*}
\text{(HO)}_2\text{SiOH} + \text{HOSi(OH)}_3 & \rightarrow \text{(HO)}_2\text{Si–O–Si(OH)}_3 + \text{H}_2\text{O} \\
\text{(HO)}_2\text{Si(OH)}_3 & \rightarrow \text{Si–O–Si(OH)}_2 + \text{H}_2\text{O} \\
\text{CH}_3 & \quad \text{CH}_3 \quad \text{CH}_3 \quad \text{CH}_3
\end{align*}
\]

(2.1)

(2.2)

(2.3)

In fact, hydrolysis and condensation reactions were carried out in a complicated manner. The condensation will start once the first ethoxy groups are hydrolyzed and the water condensation will be accompanied by alcoholic condensation [36,37]. For convenience in discussion, only ideal hydrolysis (all the ethoxy groups are hydrolyzed) and water condensation are considered in the experiment.

Furthermore, the hydrolysis rate of siloxane have been proved to be faster than condensation rate in acidic condition, while the rates are opposite under basic condition [38]. The hydrolysis of MTES was faster than that of TEOS in acidic condition, however, the condensation of MTES becomes slower under basic conditions [39]. Therefore, the sol–gel progress in this experiment could be explained as follows: TEOS and MTES would be hydrolyzed to form Si(OH)₄ and CH₃Si(OH)₃ separately with acid catalyst after they were mixed with water. When the pH value of solution was adjusted to 7.0, Si(OH)₄ groups would firstly condensate into small oligomers which were contributed by water condensations in the form of Si–O–Si bonds [29]. CH₃Si(OH)₃ was later added on the surfaces of oligomers via the polycondensation between hydroxyl groups, considering that it condensates more slowly than that of Si(OH)₄ [39]. Lots of oligomers containing exposed CH₃ groups were then produced, as illustrated in Fig. 2. After treatment solution of oligomers was infiltrated into decayed wood, oligomers would further cross link with other oligomers via Si–O–Si bonds, and with hydroxyl groups of wood cellulose via Si–O–C bonds and hydrogen bonds [40,41]. As a result, three-dimensional network structure of organosilicon gel were formed in wood structure. The gel layer fabricated onto wood cell walls would finally be established after the evaporation of water and alcohol [42] (Fig. 2).

WPG was generally applied to assess changes in weight of samples [43,44]. As listed in Table 1, it was observed that the WPG raises with the average value exceeding 83%, suggesting that a large number of products were generated in the wood structure.

Scanning electron microscopy was applied to explore the morphology and structure changes of treated wood [45]. As shown in Fig. 3(a)–(c), the cell walls of untreated wood attacked by fungi are thin and flabby in the terms of pit area, cross section and longitudinal section. However, great changes have taken place in the structures after the wood were treated. Fig. 3(d)–(f) shows that some flaky gels could be observed in the cell gaps and some are visible on wood cell surfaces, as was assumed in reaction mechanism. The attached gel layer made the cell walls of treated wood thicker than those of the untreated wood and potentially more stable and tough, which would improve the physico-mechanical properties of wood components in the ancient buildings. The EDS elemental...
Fig. 2. The proposed possible mechanisms of organosilicon gel layer formed in wood.

Fig. 3. SEM images of wood samples: (a)–(c) pit area, longitudinal section and cross section of untreated wood samples; (d)–(f) pit area, longitudinal section and cross section of treated wood samples.

Table 2
Elemental analysis of the scanned area.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Intensity(c/s)</th>
<th>Atomic %</th>
<th>Atomic ratio</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>7.25</td>
<td>40.155</td>
<td>0.9996</td>
<td>28.750</td>
</tr>
<tr>
<td>O</td>
<td>17.39</td>
<td>40.172</td>
<td>1.0000</td>
<td>38.314</td>
</tr>
<tr>
<td>Si</td>
<td>55.90</td>
<td>19.672</td>
<td>0.4897</td>
<td>32.935</td>
</tr>
<tr>
<td>Total</td>
<td>100.000</td>
<td>100.000</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

detection revealed that the element of silicon originating from TEOS and MTES is observed on the surface of flaky gels (Fig. 4(a) and (b)), which further confirms that the flaky gels are mainly composed of silicon material. Elemental analysis of the scanned area by EDS mapping (Table 2) shows that the intensity and atomic of silicon are 55.90% and 19.672% respectively, proving the high content of silicon element. Beside, silica was evenly formed in cell wall of wood according to the EDS elemental detection, which was affirmed by the detected element silicon in the cross section (Fig. 4(c) and (d)).

FTIR was used to testify changes in chemical compositions of wood [46,47]. Fig. 5 shows FT-IR spectra of sound, treated and untreated wood samples. In FTIR spectrum of untreated wood, the band at 3423 cm\(^{-1}\) was assigned to the O–H of cellulose and lignin [48,49]. The spectrum of treated wood shows a band at 1100 cm\(^{-1}\) corresponding to the asymmetric stretching vibration of Si–O–Si bridges [50,51], which overlaps with C–O and C–O–C stretching bands in the spectra of sound and untreated wood [38,52]. Moreover, in spectrum of treated wood, bands occurring at about 792 and 449 cm\(^{-1}\) were ascribable to the symmetric stretching and bending vibrations of Si–O–Si bonds unidentified in spectra of sound and untreated wood [53,54]. This confirms the formation of silicon network in the treated wood. Besides, the spectrum of treated wood shows weak intensity bands at 1275 and 2978 cm\(^{-1}\) which were
assigned to absorption vibrations of Si–C bonds and –CH3 units in Si–CH3 [52,55]. The –CH3 units could be valid for the improvement of materials’ hydrophobicity because of its non-polar trait [56,57]. Compared with the hybrid wood (wood powder mechanically being mixed with gel), Si–O–Si band in the spectrum of treated wood slightly shifts to 1062 cm⁻¹. As shown in Figs. S2 and S5, the blue shifts of Si–O–Si bond peak from 1050 cm⁻¹ in sound wood and untreated wood to ca. 1062 cm⁻¹ (Treated 1–Treated 5) indicated that organosilicon layer was likely linked with surfaces of cell walls by the intermolecular hydrogen bonding interactions [58,59]. It could be concluded the combination of organosilicon layer with the surfaces of cell walls. Furthermore, treated wood spectrum show a characteristic band at 956 cm⁻¹, which can be assigned to the stretching vibrations of Si–OH [60–62].

3.2. Physico-mechanical properties test

The changes in microstructures and chemical compositions of wood cells would normally exert impact on properties of wood, thus the hydrophobicity, mechanical properties, thermal stability and esthetics were evaluated.

Hydrophobicity of solid surfaces is influenced by both the surface morphology and chemical composition. Generally, the rougher surface structure or the lower surface energy contributed to the better hydrophobicity of surface [65–67]. In addition, the hydrophobicity is measured by contact angle of water droplet on solid surfaces [68,69]. The solid surfaces is classified as hydrophobic when the contact angle is greater than 90°, and hydrophilic when the contact angle is less than 90° [49,70]. Fig. 7 shows the contact
angle trends of treated, untreated and sound wood samples with time. Initially, the contact angles of three types samples are greater than 105°, displaying high hydrophobicity of the surfaces. However, the contact angles of untreated and sound wood samples gradually decrease with time prolonging and reach almost 60° at 70 s, which indicates the transformation of wood surfaces from hydrophobic to hydrophilic. A reasonable explain is that the hydrophobicity of untreated and sound wood was provided by the geometric roughnesses of wood surfaces which were stemmed from manufacturing process of samples, as shown in Fig. 8. Such hydrophobicity is unsta-

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of methyl groups fabricated onto its surfaces (Fig. 8), providing the wood stable hydrophobicity that was also affirmed by the water adsorption test determination. As illustrated in Fig. 53, the treated wood exhibited a very low rate of weight percentage gain during 8 d of immersion in distilled water, achieving almost constant value after 160 h, which is consistent with sound wood. In contrast, weight percentage gain of untreated wood is 2 times higher than that of treated wood because of its loose cellular structure.

The mechanical properties of treated, untreated and sound wood samples was investigated by indentation tests and the results are shown in Table 4. The parameters reveal that indentation hardness, recovery resistance and elastic modulus of untreated wood are separately about 10%, 1.26% and 3.56% of sound wood, indicating great losses of strength because of the degradation. The indentation hardness, recovery resistance and elastic modulus of the treated wood were 15, 27 and 20 times higher than those of untreated ones, which can maintain or even enhance the functions of the wood components for the ancient buildings. The significant increase in the mechanical properties of treated wood could be due to the excellent mechanical performances of organosilicon layer fabricated onto the wood cell walls. As Table 3 shows, the indentation hardness, recovery resistance, elastic modulus and toughness of organosilicon particles are 0.42, 11.99, 1.39 and 15.77 respectively, and the corresponding mechanical performances are higher than that of sound wood.

It should be noticed that the energy dissipation is an opposite variable to toughness in indentation test, and when energy dissipation gets higher, the toughness gets worse. Therefore, it can be concluded from Table 3 that treated wood demonstrates great decrease in toughness with an increase of 30% in energy dissipation rate, though reinforced by organosilicon particles of high toughness.

The thermal stability of treated, untreated and sound wood samples was tested via the thermogravimetric analyses (TGA) and differential thermal analyses (DTA), as depicted in Fig. 9. On the TGA curves of untreated and sound wood, there is a rapid decrease in their weight by flaming between 250 and 500 °C. By contrast, the treated wood displays a much lower rate of weight decrease for the existence of organosilicon. From 500 to 650 °C, more residuals of treated wood are remain than that of untreated and sound wood whose residuals gradually decrease to ca. 10% and 2% of their original weight. In addition, rates of weight decreases of untreated and sound wood are roughly similar, but the differences are generated by the evaporation of crystal water and the flaming of intact organic components in sound wood at 25–100 and 350–400 °C. On the DT curves of untreated and sound wood, the striking exothermic peaks for flaming are observed separately at 250–325, 360–440 and 450–550 °C. However, on the DT curve of treated wood, these striking peaks are much broadened and weakened, shifting its peaks to the higher temperature.

The results suggest that the fire-resistance property of treated wood was enhanced. The enhancements in fire-resistance can be attributed to the organosilicon layer observed in Fig. 3, for organosilicon layer formed in wood cell structures would melted and then covered the surfaces of wood cells with the temperature increasing. This organosilicon layer could stop oxidation and heat transfer from proceeding into the inner wood cell walls, and thus achieved a significant enhancement in fire-resistance properties [43,71].

![Figure 9](image1.png)

**Fig. 9.** The thermogravimetric (TG) and differential thermal (DT) analyses on treated, untreated and sound wood samples.

<table>
<thead>
<tr>
<th>Samples groups</th>
<th>Indentation hardness (GPa)</th>
<th>Recovery resistance (GPa)</th>
<th>Elastic modulus (GPa)</th>
<th>Energy dissipation rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sound wood</td>
<td>0.0034</td>
<td>3.8265</td>
<td>0.0773</td>
<td>77.46</td>
</tr>
<tr>
<td>Untreated wood</td>
<td>0.00035</td>
<td>0.0483</td>
<td>0.00275</td>
<td>48.24</td>
</tr>
<tr>
<td>Treated wood</td>
<td>0.00575</td>
<td>1.35755</td>
<td>0.0592</td>
<td>57.30</td>
</tr>
<tr>
<td>Organosilicon particles</td>
<td>0.42</td>
<td>11.99</td>
<td>1.39</td>
<td>15.77</td>
</tr>
</tbody>
</table>

![Figure 10](image2.png)

**Fig. 10.** Overall appearances of untreated and treated wood samples.

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The aims of organosilicon treatment are not only to improve the hydrophobicity, mechanical properties and thermal stability of decayed wood components, but also to preserve the original appearance that is critical in the terms of authenticity principles of heritage protection. Because few changes in dimensional of wood have occurred in this experiment, color changes were exclusively considered in esthetic measurement. Compared with untreated wood samples, several treated wood samples of the same batch were used to exhibit color changes via variable values of $\Delta E$ in Table 4. The values of $\Delta E$ ranging from 4.11 to 6.81 suggests that the color changes of treated wood are slight different from that of the untreated wood, and acceptable from the perspective of heritage protection. It can be observed that the treated wood retains original natural coloration of wood with minor color deviations from the untreated wood (Fig. 10). The color changes could be attributed to the transparency and refraction of organosilicon layer fabricated onto the wood cell walls.

4. Conclusions

In this study, the effects of organosilicon compounds of MTES and TEOS on the hydrophobicity, mechanical properties, thermal stability and esthetics of decayed wood components in ancient buildings were investigated with the reaction mechanism explored. The experimental results provided a great deal of valuable information for applying the organosilicon compounds in decayed wood component conservation. As expected, the organosilicon treatment produced tremendous changes to morphological structures of wood cells, which led to significant improvements of performances in different properties of decayed wood and met the requirement of retaining the original wood components with value and information reserved.

The exploration of mechanism by SEM-EDS, FTIR and XPS demonstrated that a large numbers of organosilicon flakes were generated in the cell gaps, cell surfaces as well as inside the cell walls, which led to a protective organosilicon layer. The organosilicon layer was conditioned by the hydrolyzed alkoy groups through forming siloxy bonds of Si–O–Si, and fabricated on the surfaces of wood cells by hydrogen bonds and stable bonds of Si–O–C.

The formation of organosilicon layer further improved the physico-mechanical properties of wood. Firstly, a stable hydrophobic surfaces on wood cell walls was contributed from organosilicon layer which could sealed the pores of wood surfaces and provided a large number of hydrophobic methyl groups. Secondly, the wood cell structures were greatly reinforced by the organosilicon layer for its excellent mechanical properties. Thirdly, fire-resistant was also enhanced by the organosilicon layer which could prevent oxidation and heat transfer from proceeding into the inner cell structures. Besides, the treated wood retained its natural coloration, despite minor color deviations from the untreated wood.

It should be underlined that performances of decayed wood were improved obviously, differences in physico-mechanical properties significantly existed between treated and sound wood. The differences could make the treat-decayed parts of wood discordant with other sound parts when the external temperature and humidity change. Therefore, the organosilicon treatment could be suitable for wooden component, only after the differences in properties are rigorously investigated. Further, it was visualized in the mechanical tests that the organosilicon layer was not available for toughness improvement and needs to be ameliorated by the introduction of toughening materials in the future studies.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.culher.2019.11.012.

References
