

# Long-term performance of bio-based miscanthus mortar

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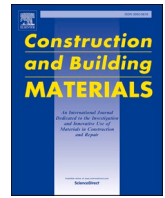
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## Long-term performance of bio-based miscanthus mortar

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### ABSTRACT

The long-term degradation behavior of embedded natural fibres is one of the key factors restricting the durability of bio-based mortars and concretes. In this study, to minimize the negative impact of miscanthus fibre on the service life of bio-based miscanthus mortar, the effects of different treatments (heat treatment, milling) on the long-term performance of bio-based miscanthus mortar are investigated. Results show that the heat-treated miscanthus fibres improve the compressive strength and flexural strength of miscanthus mortar by 82.7% and 26.9%, respectively, compared to the reference fibre, thanks to the reduced porosity and enhanced compatibility. The mechanical strength degradation of miscanthus mortar mainly occurs in the first month after being soaked in water. Moreover, the alkaline environment in miscanthus mortar causes the dissolution of some cellulosic components, resulting in the degradation of the miscanthus fibre. The removal of sugar from the miscanthus fibre is conducive to the strength development and durability of bio-based miscanthus mortar. It is concluded that heat-treated miscanthus fibres can be applied to improve the dimensional stability and degradation resistance of miscanthus mortar for better application of miscanthus in sustainable building materials.

### 1. Introduction

In recent years, renewable bio-based materials such as miscanthus, bamboo, peach shell, oil palm shell, etc. have been used as substitutes for replacing conventional raw materials such as gravel and sand, etc. in the construction sector [1–3]. Bio-based concrete generally possesses lightweight density [4,5], good thermal and acoustic insulation properties [6–8], as well as environmental friendliness [9–11]. Natural fibres are mainly composed of cellulose, hemicellulose and lignin, etc. [12], and the drawbacks of applying bio-based materials in cementitious materials include hygroscopicity, incompatibility and degradability, etc. [13]. Therefore, natural fibres generally need pretreatment prior to application in cementitious materials.

The low affinity between bio-based materials and cement paste is one of the main reasons for poor strength [14]. Moreover, the alkaline solution in the concrete pore will dissolve the cellulose of natural fibre, resulting in a strong degradation of the natural fibre [15], and eventually, reducing the service life of bio-based concrete. Previous studies have shown that the sugar released from natural fibre has a significantly negative effect on cement hydration and delays the setting and hardening of concrete matrix [16–18] because the extractives from natural

fibre affect the production of calcium-silicate-hydrate and portlandite.

Miscanthus (*x giganteus*) is a perennial biomass energy crop that is widely cultivated in Europe attributed to the highly efficient use of water, nitrogen and sunlight [19–21]. Compared to other natural fibres such as straw, hemp, etc., miscanthus is considered as a strong fibre, possesses enough firmness and excellent thermal insulating properties [22]. Thanks to the lightweight and porous properties, miscanthus is commonly used as a natural fibre for bio-based lightweight concrete [11,23]. For example, Dias and Waldmann [3] investigated the effects of miscanthus treated with silicate sealant and cement-based fluid on the performance of miscanthus concrete, and found the silicate sealant treated miscanthus improved the compressive strength. Chen et al. [10,22] reported the use of miscanthus fibre as an acoustic material to investigate the acoustic performance of miscanthus mortar, and obtained an ultra-lightweight miscanthus concrete with a density of 554 kg/m<sup>3</sup> and an acoustic absorption coefficient of 0.9. Wu et al. [24] studied heat-treated miscanthus fibre as a porous material to improve the phosphorus removal capacity of the mortar owing to the microporous nature, which contributed to the physical adsorption capacity. These studies confirm that miscanthus has great potential as an alternative material for sustainable building materials [25].

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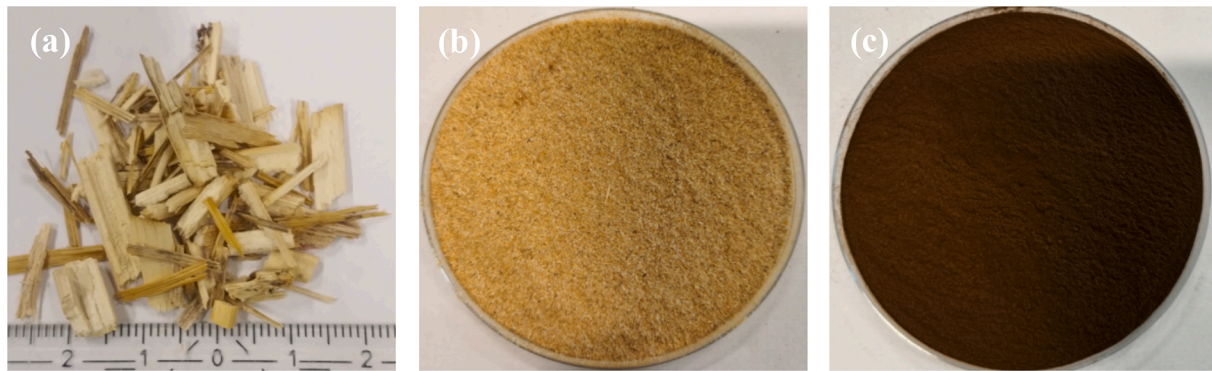


Fig. 1. Miscanthus used in this study (a) raw miscanthus, (b) miscanthus powder and (c) heat-treated miscanthus.

Although miscanthus fibre is covered in a closed environment after being applied to concrete, the alkaline environment has a dissolving effect on cellulosic components of miscanthus fibre, which will affect the mechanical strength, degradation resistance and dimensional stability of cementitious matrix containing miscanthus. Previous studies show that the sugar is released from the miscanthus under the alkali condition as the miscanthus is applied to concrete [1]. Besides, miscanthus fibre has a strong affinity with water, possesses high water absorption capacity, and the shrinkage and swelling of miscanthus have strong negative effects on the durability of bio-based miscanthus concrete. Bio-based miscanthus concrete generally has a higher porosity and water absorption [26], the mechanical strength, degradation characteristics and dimensional stability will be affected by the humidity of the ambient environment. Therefore, the strength degradation behavior, drying shrinkage and wet swelling characteristics of bio-based miscanthus mortar should be investigated systematically.

Organic matter is one of the main reasons causing the degradation and low strength of bio-based concrete [27]. Through the removal of cellulose, hemicellulose and lignin, etc., or to change the morphology of natural fibre and to improve the interface bonding capacity, various treatment methods have been applied including physical treatment (milling [24], rolling compression [28], etc.), chemical treatment (alkali, silane, etc. [1]) and heat treatment [27], etc. Among these methods, heat-treated bio-based materials, such as biochar, etc., can significantly enhance the mechanical strength of bio-based concrete thanks to the increased bonding between the mortar and the bio-based materials.

The thermal decomposition of lignocellulosic biomass generally results in a re-polymerisation reaction of biomass [29]. The decomposition of hemicellulose mainly occurs between 250 and 350 °C, represented by xylan; followed by cellulose decomposition, mainly between 325 and 400 °C, with a main pyrolysis product of levoglucosan; lignin is decomposed with a higher temperature range of 300–550 °C [30]. The water and oxygen content from the biomass can be fully or partially removed after heat treatment at a temperature of 200–300 °C [31], resulting in a reduction in the hydrophilicity and bio-degradation of the biomass, an improvement in the dimensional stability [29]. Previous studies have shown that heat-treated wood has increased dimensional stability, a decrease in mechanical strength, and a darkening of wood tissue [32]. Therefore, heat treatment is an effective method to improve dimensional stability and reduce the biological degeneration of bio-based materials [27,33]. Considering environmental benefits, the effects of physically treated miscanthus by the milling method on physical and mechanical properties, degradation behavior and dimensional stability of bio-based miscanthus mortar are also investigated for comparison.

This study focuses on investigating the physical and mechanical properties, long-term degradation behavior and dimensional stability of bio-based miscanthus mortar. The effects of the raw miscanthus (RM),

Table 1

Mix proportions of miscanthus mortar.

Sample	Cement (g)	Sand (g)	Water (g)	Miscanthus (Vol.%)
RM1	450	1350	225	1
HM1	450	1350	225	1
MP1	450	1350	225	1
MP1.5	450	1350	225	1.5
MP2	450	1350	225	2.0

heat-treated miscanthus (HM) and miscanthus powder (MP) on the degradation behavior of bio-based miscanthus mortar are investigated systematically. The degradation mechanism of miscanthus fibres in water and cement solution is evaluated by FTIR, XRD and SEM analyses. The physical and mechanical properties of bio-based miscanthus mortar, strength degradation after being submerged in water for 90 days are analyzed. The dimensional stability of bio-based miscanthus mortar including drying shrinkage and wet swelling are discussed. Based on the present results, the HM fibre shows outstanding performance in reducing the dimensional stability and degradation behavior, which can be applied to improve the service life of bio-based miscanthus mortar.

## 2. Materials and methods

### 2.1. Materials

Raw miscanthus (RM) and miscanthus powder (MP) supplied by Vibers (The Netherlands) are used in this study, as shown in Fig. 1. The RM is heat-treated using a vacuum furnace under nitrogen gas at 250 °C for 3 h, and then the heat-treated miscanthus (HM) is milled using a ball milling machine to obtain the HM powder (Fig. 1c). The purpose of heat treatment is to reduce the biomass of the RM and investigate the effects of the HM on the physical mechanics and degradation characteristics of miscanthus mortar. The specific densities of the RM, HM and MP are determined by using an AccuPyc II 1340 gas pycnometer, which are 1.64 g/cm<sup>3</sup>, 1.25 g/cm<sup>3</sup> and 1.57 g/cm<sup>3</sup>, respectively. CEM I 52.5 R Portland cement (supplied by ENCI, The Netherlands) and CEN-NORM standard sand are used as the binder and fine aggregates, respectively.

### 2.2. Mix proportions and sample preparations

The mix proportions of miscanthus mortar are shown in Table 1. The mixing ratio of cement, sand and water of the miscanthus mortar refers to the mix proportion and preparation process of the standard mortar [34]. Because the high content of miscanthus fibre has a negative effect on the mechanical strength [22], the RM and the HM with a dosage of the cement of 1.0 vol% is added to the mixture as the reference. The effects of the MP with different contents of the cement of 1.0 vol%, 1.5 vol% and 2.0 vol% on the physical and mechanical properties of bio-based miscanthus mortar are also investigated. The preparation of the



Fig. 2. Miscanthus mortar samples are submerged in water for (a) strength degradation test and (b) leaching test.

Table 2

Mix proportions of miscanthus fibre for degradation test.

Sample	Cement (g)	Miscanthus (g)	Water (g)	Miscanthus type
MP	–	10	100	Raw
CMP	10	10	100	Raw
HM	–	10	100	Heat-treated
CHM	10	10	100	Heat-treated

sample refers to the casting method of the standard mortar [34].

### 2.3. Testing methods

#### 2.3.1. Physical and mechanical properties

The sample at the age of 28 days is oven-dried at a temperature of  $110 \pm 5$  °C for not less than 24 h, and then the density, water absorption and porosity of the sample are determined, test procedures are described in ASTM C642-13 [35].  $40 \times 40 \times 160$  mm<sup>3</sup> samples are used for the compressive and flexural strengths at the age of 28 days according to EN 196-1 [34], with a loading rate of 2400 N/s and 50 N/s, respectively. The microstructures of the interfacial transition zone between the miscanthus fibre and the mortar are analyzed using a SEM analyzer (Phenom ProX).

#### 2.3.2. Degradation characteristics of miscanthus mortar

##### (1) Strength degradation of miscanthus mortar

Due to the high water absorption of bio-based materials and low compatibility with the mortar interface, the strength degradation of bio-based miscanthus mortar is investigated. The samples (RM1, HM1 and MP1) with a curing age of 90 days are submerged in water, and then removed from the water for compressive strength and flexural strength tests at the different time of 30, 60 and 90 days (Fig. 2a). The changes in mechanical strength of the bio-based miscanthus mortar before and after

the degradation test are analyzed.

The ion concentration leached from the miscanthus mortar during the degradation process is also investigated (Fig. 2b). The samples RM1, HM1 and MP1 and the deionized water are mixed with a liquid to solid ratio of 10 according to EN 12457-2 [36], and then leachates are periodically collected and filtered using a 0.22  $\mu$ m filter for leaching analysis. The potassium ( $K^+$ ), calcium ( $Ca^{2+}$ ) and sodium ( $Na^+$ ) ions leached from the sample are measured by ion chromatography (IC, Thermo Dionex Aquion) and other metal ions (Al, Fe, Mg, Cr, Sr, Zn, etc.) are measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES, SPECTROBLUE).

##### (2) Degradation behavior of miscanthus fibre

The mixture of cement and water is applied to simulate the cementitious environment where miscanthus fibre stays. The degradation behavior of miscanthus fibre in an aqueous solution and cement solution are investigated. The mixing ratio of miscanthus and water refers to the leaching test method (EN 12457-2 [36]), with a liquid to solid ratio of 10. The mix proportions of miscanthus and solution are presented in Table 2.

After mixing, the mixtures are shaken using a dynamic shaker with a constant speed of 250 rpm for 24 h and then the mixture is kept in the plastic bottle (Fig. 3). After being soaked for 20, 40, 60, 80 and 100 days, respectively, the samples are taken out from the plastic bottle and then oven-dried at 38 °C for no less than 48 h following the drying method [37]. The Fourier-Transform Infrared Spectroscopy (FTIR, PerkinElmer) is used to investigate the degradation characteristics of the miscanthus before and after being submerged in the solution by vibratory sequences analysis, with a vibration range from 4000 to 500  $cm^{-1}$  and a 4  $cm^{-1}$  resolution. The X-ray diffraction (XRD, Bruker AXS D4 endeavor) and SEM (Phenom ProX) are applied to analyze the crystal structure and micromorphology of miscanthus fibre during the degradation test. The change in the pH value of the solution with time is periodically measured using a pH meter.

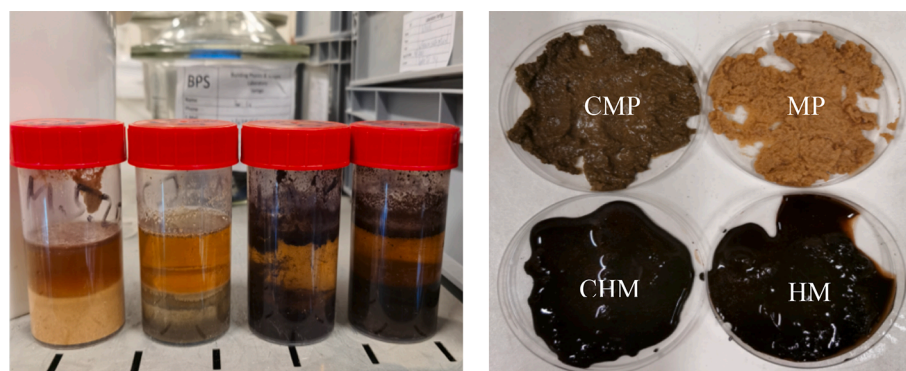


Fig. 3. Miscanthus fibre used for the degradation test.



**Table 3**  
Density, porosity and water absorption of miscanthus mortar.

Sample	Oven-dry density (kg/m <sup>3</sup> )	Changes in %	Porosity (%)	Changes in %	24-h water absorption (%)	Changes in %
RM1	1932 ± 19	–	16.7 ± 0.3	–	8.6 ± 0.2	–
HM1	2006 ± 26	+3.8	15.1 ± 0.4	–9.6	7.3 ± 0.2	–15.1
MP1	1893 ± 23	–2.0	17.5 ± 0.3	+4.8	9.1 ± 0.2	+5.8
MP1.5	1809 ± 18	–6.4	18.5 ± 0.2	+10.8	10.1 ± 0.1	+17.4
MP2	1688 ± 28	–12.6	19.1 ± 0.2	+14.4	10.8 ± 0.1	+25.6

### 2.3.3. Dimensional stability of miscanthus mortar

#### (1) Drying shrinkage

Miscanthus fibre is sensitive to the humidity in the environment, which will affect the dimensional stability of bio-based miscanthus concrete. The drying shrinkage and wet swelling characteristics of miscanthus mortar are investigated. The drying shrinkage test is performed with at least in each case 3 prisms of 40 × 40 × 160 mm<sup>3</sup> according to DIN 52,450 [38], using a dial gauge with a distinguishability of 0.001 mm. The samples used for the drying shrinkage test are stored in the laboratory with an ambient temperature of 20 ± 2 °C and relative humidity of 65 ± 3 %. As the drying shrinkage of concrete generally changes significantly after demolding, the drying shrinkage and mass loss are recorded every two days in the first month after demolding. One month later, the drying shrinkage and mass loss are measured once a week.

#### (2) Wet swelling

The dimensional stability of miscanthus mortar under a saturated environment is investigated. The prismatic samples of 40 × 40 × 160 mm<sup>3</sup> after demolding are stored in water with a temperature of 20 ± 2 °C and a water cover of at least 10 mm. Similar to the drying shrinkage test method, the axial wet swelling and changes in the mass of the samples are measured periodically. The average value of the three samples is recorded as the result. The wet swelling characteristics of miscanthus mortar in the water environment are evaluated.

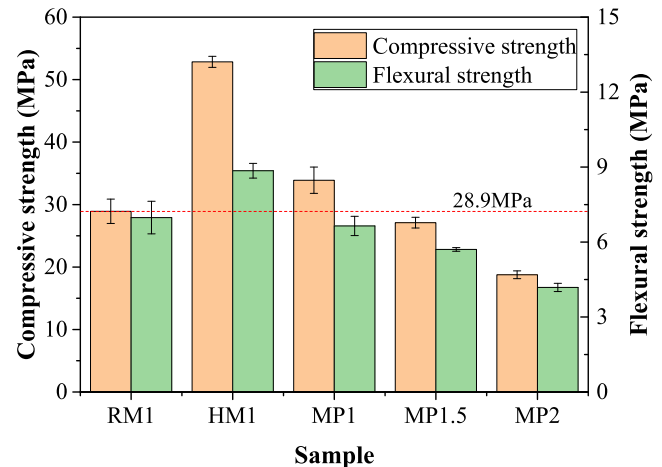
## 3. Results and discussion

### 3.1. Physical and mechanical properties of miscanthus mortar

#### 3.1.1. Density, porosity and water absorption

The density, porosity and water absorption of miscanthus mortar are shown in Table 3. The mortar HM1 slightly increases the oven-dry density of the miscanthus mortar and significantly reduces the porosity and water absorption, with a reduction of 9.6% and 15.1%, respectively, compared to the mortar RM1. This may be attributed to the good compatibility between the heat-treated materials and the mortar [27], which leads to the decrease of the microporous structure in the interfacial transition zone (Fig. 5). The low porosity and water absorption can reduce the passage for the ion exchange between the interior of the mortar and the external environment, which is helpful to improve the bio-degradation resistance of bio-based miscanthus mortar.

The results also show that the high content of miscanthus powder (MP1.5 and MP2) significantly reduces the oven-dry density of miscanthus mortar and increases the porosity and water absorption of miscanthus mortar. When the miscanthus powder content is 2% (MP2), the oven-dry density of miscanthus mortar reduces by 12.6%, compared to the mortar RM1. Therefore, the miscanthus powder is more effective than raw miscanthus when the miscanthus fibre is applied for the manufacture of lightweight, acoustic absorption or heat-insulating concrete.



**Fig. 4.** 28-day compressive strength and flexural strength of miscanthus mortar.

#### 3.1.2. Compressive strength and flexural strength

The mechanical strengths of miscanthus mortar at the curing age of 28 days are shown in Fig. 4. The compressive and flexural strengths of the mortar HM1 are significantly improved. The 28-day compressive and flexural strengths of the mortar HM1 are 52.8 MPa and 8.86 MPa, respectively, with an increase of 82.7% and 26.9%, respectively, compared to the mortar RM1. This may be attributed to the sugar removal of miscanthus after pyrolysis that increases the adhesion to the mortar, and the HM1 has a lower porosity. An inverse relationship between miscanthus sugar content and mechanical strength is observed [1]. The use of heat-treated bio-based materials to improve mechanical strength has been reported in previous studies, including apricot shell [5], oil palm shell [39] and biochar [40], etc. The removal of sugar from the bio-based materials by heat treatment and alkali treatment methods etc. is the key to enhance the mechanical strength of bio-based concrete.

The raw miscanthus has a large size and flat shape, the pores formed inside the mortar are larger than that of the miscanthus powder. The MP series show a significant increase in compressive strength, with an increase of 17.3%. However, the increase in flexural strength is not obvious due to the reduced fibre-bridging effect caused by the reduced size of miscanthus powder compared to untreated miscanthus fibre. The content of miscanthus has a significant effect on the mechanical strength. When the miscanthus powder content is 2%, the compressive and flexural strengths of the mortar MP2 are reduced by 34.9% and 39.8%, respectively, compared to the mortar RM1. The added miscanthus content should be considered with caution for applying bio-based miscanthus mortar in the structural elements.

#### 3.1.3. Microstructure analysis

The compatibility between bio-based materials and mortar is one of the main factors restricting the mechanical strength of bio-based concrete. As shown in Fig. 5, weak adhesion between miscanthus fibre and mortar is observed on the surface of the RM and MP. The HM shows good compatibility with mortar, which is also one of the main reasons for the better mechanical strength of the mortar HM1. Due to the shrinkage and

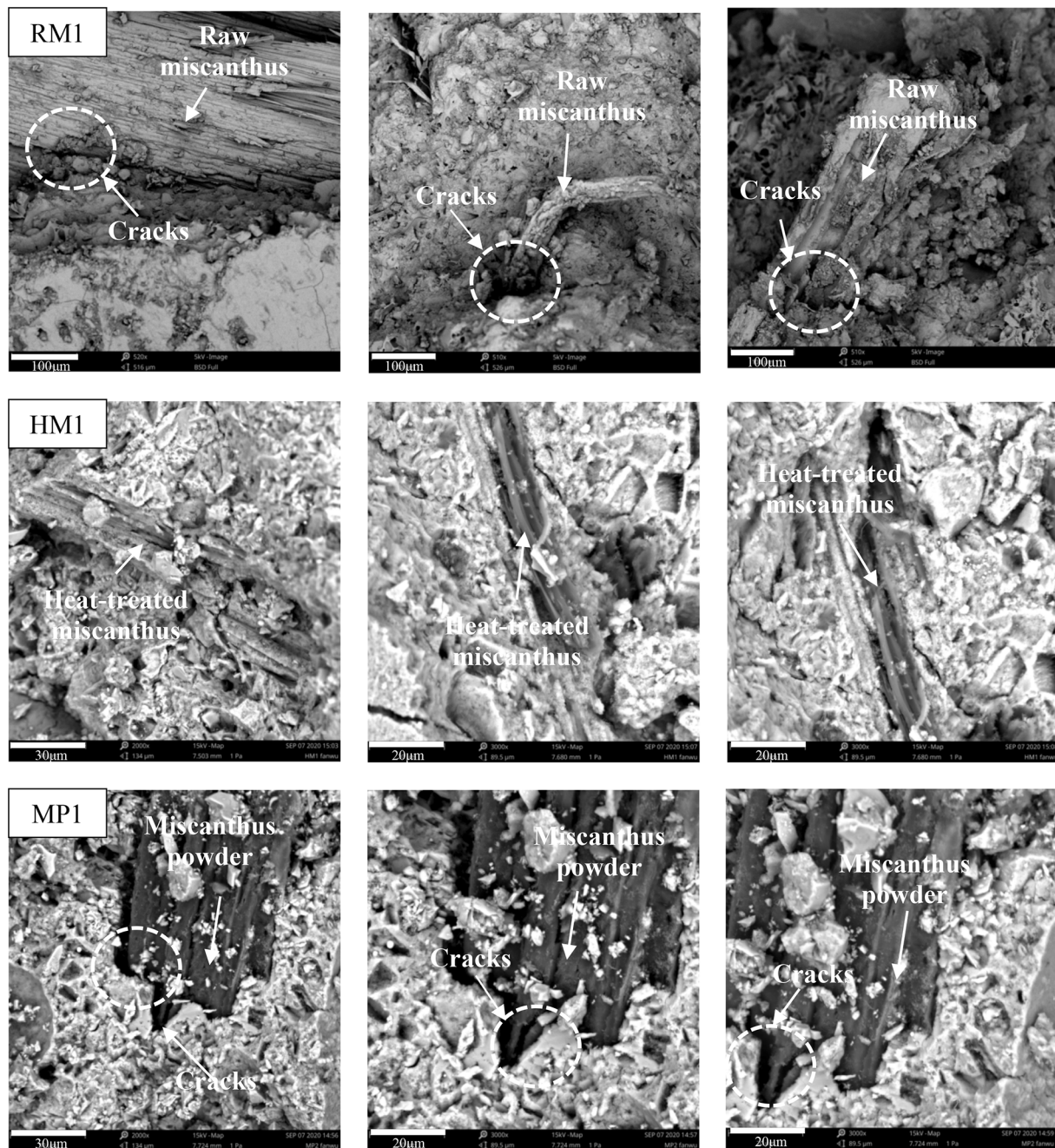


Fig. 5. Microscopic images of miscanthus mortar.

swelling characteristics of the miscanthus fibre, the changes in humidity of the environment will have a significant impact on the dimensional stability of bio-based miscanthus mortar. The dimensional stability of miscanthus mortar is discussed in Section 3.3.

### 3.2. Degradation characteristics

#### 3.2.1. Strength degradation of miscanthus mortar

The changes in mechanical strength of miscanthus mortar after immersion in water are shown in Fig. 6. The compressive strength and flexural strength of all miscanthus mortars decrease rapidly in the initial soaking stage of 30 days, and then the reduction in mechanical strengths becomes inobvious. The compressive strength of the mortars RM1, HM1 and MP1 decreases by 17.6%, 23.8%, and 16.9%, respectively, and the corresponding flexural strength decreases by 30.0%, 38.4%, and 17.3%,

respectively, after being submerged in water for 30 days. This may be because miscanthus mortar gradually absorbs water until saturation, the saturated miscanthus mortar has a negative effect on the mechanical strength. The wet swelling results confirm that the mass and wet swelling value of miscanthus mortar gradually increase within the first month (Section 3.3.2). The slight deformation (i.e. swelling) of the miscanthus mortar may be the main reason for the rapid decrease in mechanical strength in the first month.

The results also show that the mechanical strengths of the saturated miscanthus mortar at the 30-day and the 90-day have no obvious difference, indicating that the degradation of the miscanthus fibre has a limited influence on the strength degradation of bio-based miscanthus mortar, and the saturation of miscanthus mortar may be the main reason for the strength degradation. Moreover, when miscanthus fibre is soaked in an alkaline solution, a more fibrillated surface is produced [13],

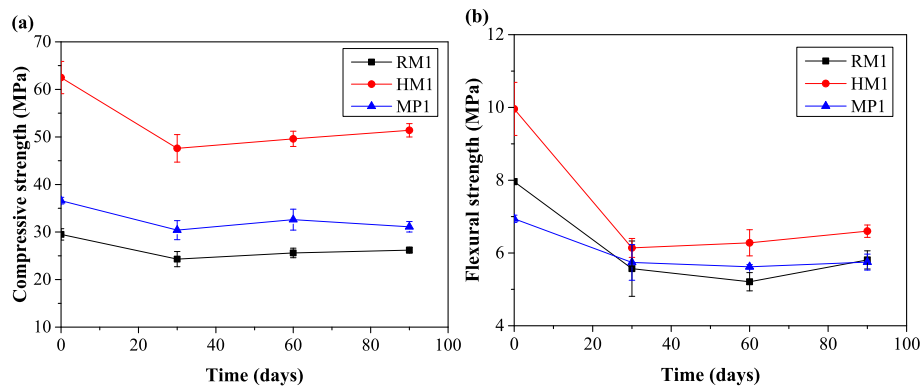


Fig. 6. Changes in mechanical strength of miscanthus mortar after being submerged in water.

Table 4

Metal ions leached from the miscanthus mortar.

Ions	Sample	Changes in the ion concentration with soaking time (mg/L)			
		30 days	60 days	70 days	90 days
Ca	RM1	30.9	278	353	403
	HM1	114	251	306	348
	MP1	125	297	384	438
K	RM1	81.8	104.2	106.7	111.6
	HM1	68.9	84.3	89.2	94.6
	MP1	73.4	97.4	98.9	104.5
Na	RM1	21.6	34.6	37.2	40.4
	HM1	21.4	28.1	31.5	34.5
	MP1	16.2	28.5	32.0	35.2
Al	RM1	0.556	0.856	0.957	1.092
	HM1	0.618	0.618	1.098	1.352
	MP1	0.177	0.158	0.495	0.681
B	RM1	0.007	0.007	0.008	0.007
	HM1	0.005	0.006	0.007	0.007
	MP1	0.004	0.005	0.006	0.006
Ba	RM1	0.248	0.683	0.815	0.961
	HM1	0.448	0.627	0.750	0.863
	MP1	0.393	0.744	0.902	1.039
Cr	RM1	0.005	0.010	0.012	0.015
	HM1	0.004	0.006	0.009	0.011
	MP1	0.003	0.009	0.014	0.017
Fe	RM1	–	–	–	–
	HM1	–	0.002	0.004	0.002
	MP1	–	–	–	–
Li	RM1	0.147	0.299	0.350	0.408
	HM1	0.172	0.240	0.283	0.330
	MP1	0.123	0.246	0.302	0.342
Mg	RM1	0.012	0.010	0.009	0.008
	HM1	0.009	0.010	0.008	0.007
	MP1	0.021	0.007	0.006	0.004
Sr	RM1	1.312	3.865	4.608	5.411
	HM1	2.621	3.464	4.119	4.663
	MP1	1.978	3.801	4.650	5.319
Zn	RM1	0.011	0.001	0.007	0.004
	HM1	0.005	0.005	0.005	0.006
	MP1	0.006	0.005	0.004	0.002

which is conducive to the strength stability of bio-based miscanthus mortar.

The ions leached from miscanthus mortar are presented in Table 4. The results show that calcium, potassium and sodium ions are the main ions leached from miscanthus mortar, much higher than the concentration of other ions. All ions released from the miscanthus mortar increase with the immersion time. Most of the ions leached from the mortar HM1 are less than that of the mortars RM1 and MP1 thanks to the low porosity of the mortar HM1. The high content of calcium ions leached from miscanthus mortar creates an alkaline environment, in which some sugars or other cellulosic components can dissolve [1,13]. The sugar removal from miscanthus fibre is beneficial to the cement hydration, strength development and durability of miscanthus mortar.

In addition, the airtight alkaline environment also delays the degradation of miscanthus fibre.

### 3.2.2. Degradation behavior of miscanthus fibre

#### (1) FTIR analysis

The FTIR results of miscanthus fibre after being submerged in solution are shown in Fig. 7. A broad band for the hydroxyl group (O–H) of cellulose and hemicellulose is observed in the range between 3600 and 3000  $\text{cm}^{-1}$ . The O–H bond of the hydrated products usually appears at 3434  $\text{cm}^{-1}$  [41]. When miscanthus is immersed in cement solution, more obvious peaks appear in the range of the O–H group for the CMP and CHM series. The increase in soaking time has no obvious effect on the O–H group.

The C–H or  $\text{CH}_2$  stretching of cellulose and hemicellulose are in the range between 2850 and 2920  $\text{cm}^{-1}$ . The CM and CHM series show an insignificant vibration peak at 2850–2920  $\text{cm}^{-1}$ . Boix et al. [1] reported that a small amount of hemicellulose can be extracted by water but not for cellulose, while the cellulose and xylose of miscanthus are significantly reduced after being soaked in a cement–water–sand mixture. This is because some cellulosic components are dissolved in the alkaline environment. As shown in Table 5, the leachates of the CMP and CHM series are highly alkaline with pH values of 11.4–12.5, while the MP and HM series are weakly acidic with pH values of 5.1–6.3. The highly alkaline environment has a dissolving effect on the miscanthus fibre components.

The MP shows an obvious band at 1730  $\text{cm}^{-1}$ , corresponding to carboxyl groups (C = O), while after being heat-treated (HM) or soaked in cement solution (CMP and CHM), the CM and the CHM show a disappearance of the C = O band. These results are consistent with the results of alkali-treated miscanthus, which is attributed to the removal of some hemicellulose and lignin in carboxyl groups (C = O) [13]. The C–O stretching band in the range of 1240 and 1250  $\text{cm}^{-1}$  shows a similar trend, a decreased intensity almost to disappearance. This may be because of the alkali hydrogen bonding dissociation of hemicellulose and cellulose, as well as the hydrolysis of the hemicellulosic ester bonds [42].

The C = C aromatic symmetrical stretching of lignin at 1515  $\text{cm}^{-1}$  does not show obvious changes for the MP and the HM because of the low pyrolysis temperature (250  $^{\circ}\text{C}$ ) applied in this study. The degradation temperature for cellulose, hemicellulose and lignin are 200–260  $^{\circ}\text{C}$ , 240–350  $^{\circ}\text{C}$  and 280–500  $^{\circ}\text{C}$ , respectively [43]. The low pyrolysis temperature does not reach the level for lignin decomposition. Only the CMP and the CHM show the disappearance of some C = C group, possibly because part of the lignin is decomposed in the highly alkaline solution.

Different from the miscanthus soaked in water (MP and HM), an obvious C–O stretching band is also observed at 713  $\text{cm}^{-1}$ , 872  $\text{cm}^{-1}$  and



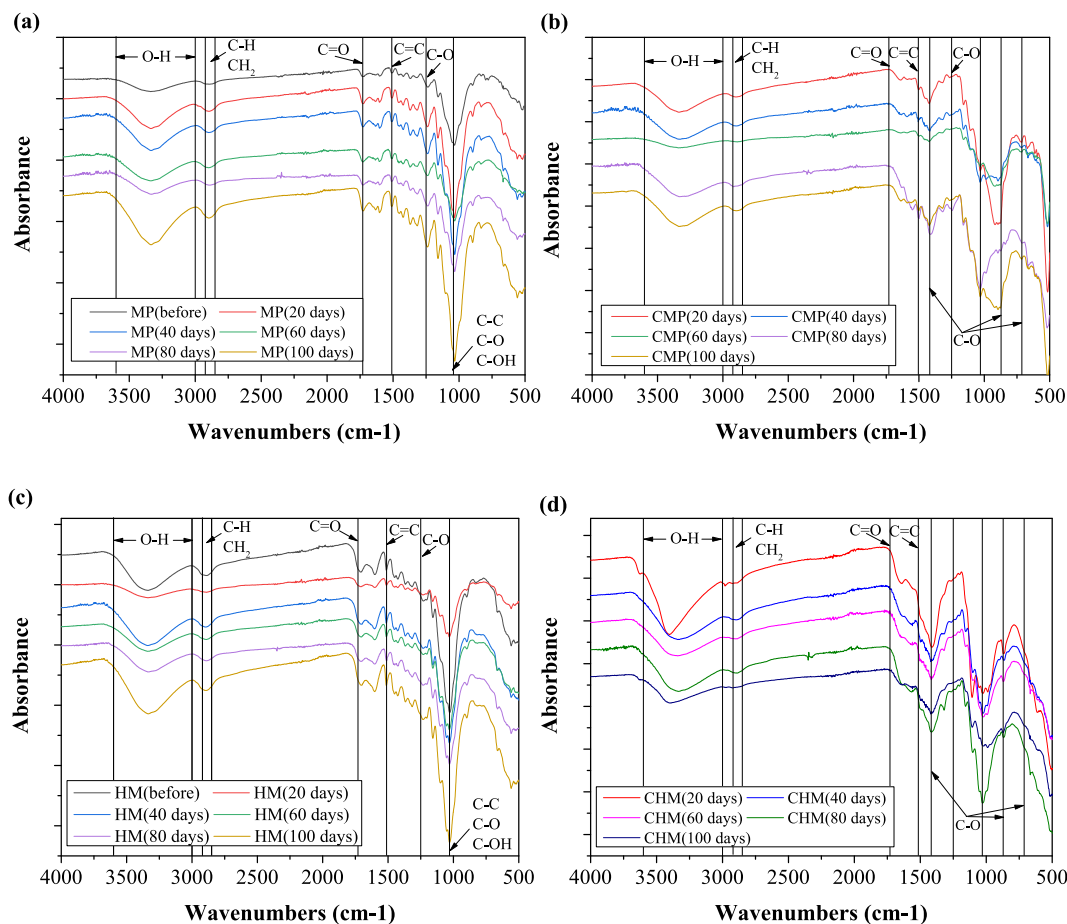


Fig. 7. FTIR data of miscanthus fibre after being submerged in the solution.

Table 5  
pH value of the leachates of the miscanthus fibre.

Sample	Duration of miscanthus fibre submerged in the solution				
	10 days	20 days	30 days	60 days	100 days
MP	5.3	5.1	6.3	5.7	5.2
CMP	11.4	11.6	11.6	11.6	11.6
HM	5.8	5.8	5.7	5.5	5.7
CHM	12.0	12.2	12.4	12.5	12.5

1417  $\text{cm}^{-1}$  for the CMP and CHM. This may be because of the carbonation reaction between portlandite ( $\text{Ca}(\text{OH})_2$ ) from the hydrated product of cement and atmospheric carbon dioxide ( $\text{CO}_2$ ) [41]. An obvious

peak at 1043  $\text{cm}^{-1}$  is attributed to C–C, C–O stretching or C–OH bending of cellulose and hemicellulose [13,14].

(2) XRD analysis

The XRD results of miscanthus fibre before and after water immersion are shown in Fig. 8. Generally, among the cellulose, hemicellulose, lignin, waxes, pectin, etc. only cellulose possesses a crystalline structure [13]. The results show that all miscanthus fibres are amorphous because of the irregular distribution of the cellulose chain molecule in the amorphous area, resulting in a diffuse reflection and no peaks [44]. However, a distinct peak occurs at the diffraction angle ( $2\theta$ ) of 25–30 because of the crystalline cellulose. The HM series have a wider peak

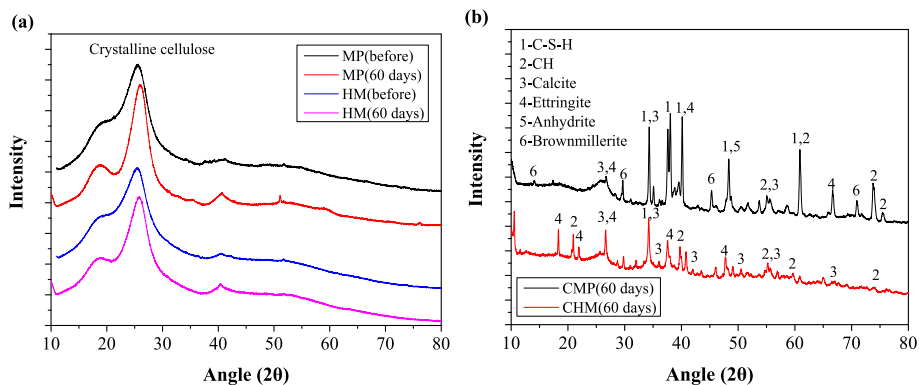


Fig. 8. XRD results of miscanthus fibre before and after water immersion.



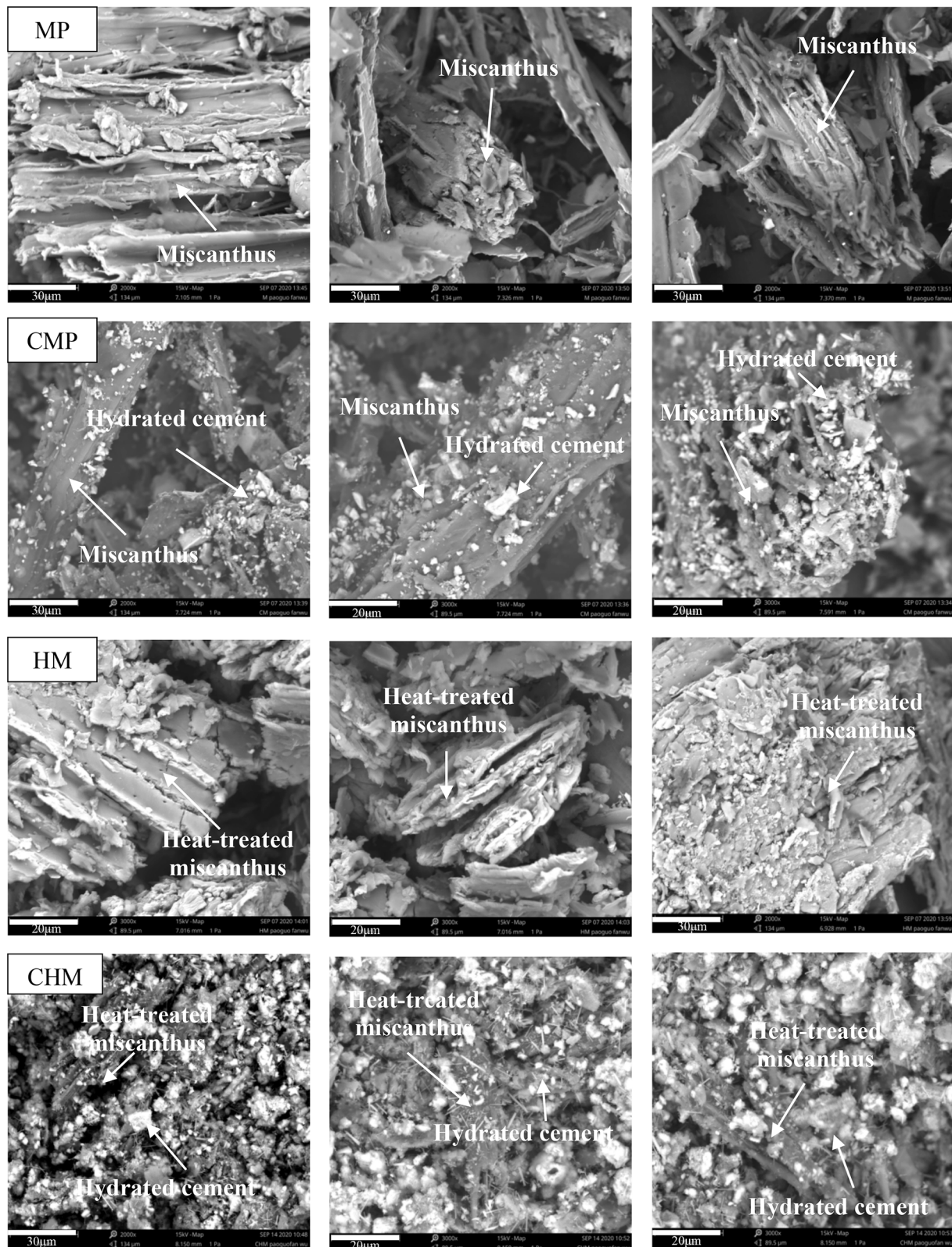


Fig. 9. Microscopic images of the miscanthus fibre after being submerged in the solution for 100 days.

than the RM series owing to the destruction of some cellulose crystals during the pyrolysis process. Similar phenomena are observed in heat-treated bio-based materials [27]. Besides, the RM and the HM after being soaked in water for 60 days show a higher crystallinity because some non-crystalline compounds such as waxes and pectin are removed.

The same phenomenon is observed in the alkali-treated miscanthus [13].

Compared to miscanthus soaked in water (MP and HM), the surface of miscanthus soaked in cement solution (CMP and CHM) is covered with cement paste. The XRD pattern of the CMP and CHM detects some

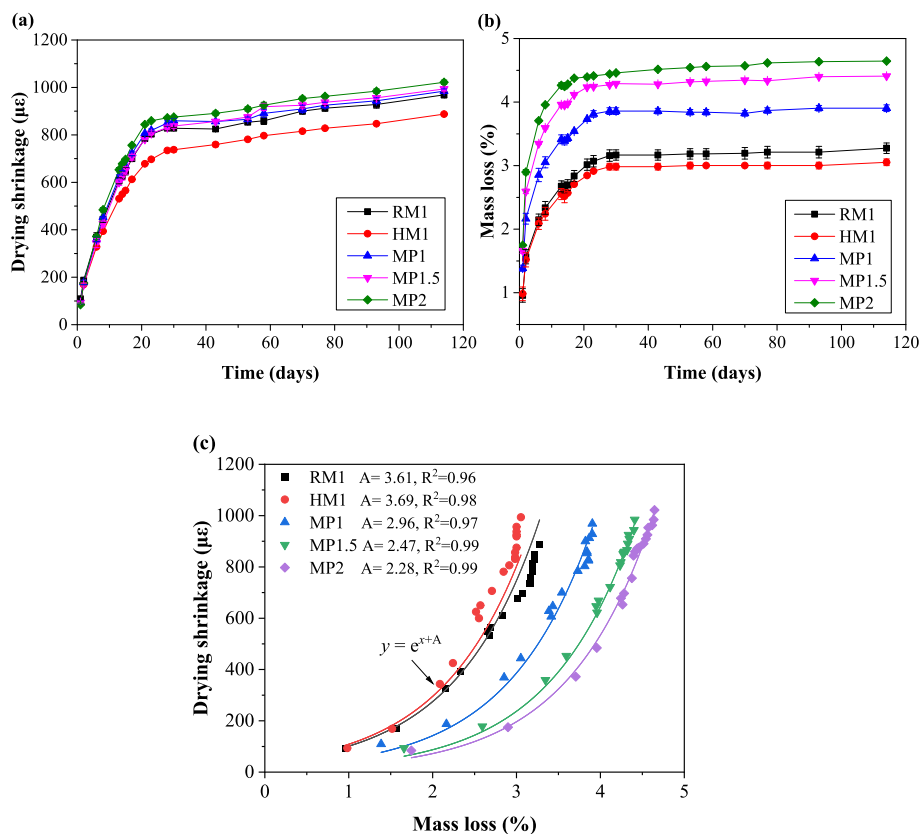


Fig. 10. (a) drying shrinkage, (b) mass loss and (c) relationship between mass loss and drying shrinkage of miscanthus mortar (Eq. (1)).

hydration products such as calcium silicate hydrate and calcium hydroxide, and also calcium carbonate from hydrated products reacts with carbon dioxide in the atmosphere. An obvious C-O stretching band from the calcium carbonate is observed (Fig. 7b, 7d). In this study, for samples CMP and CHM, the hydration products on their surfaces are difficult to fully remove before the FTIR test due to the precipitation effect. Therefore, the effects of hydrated cement products on the crystal pattern are difficult to eliminate, the changes in the crystal structure of the miscanthus soaked in the cement solution should be further determined in future work.

### (3) SEM analysis

The microscopic images of the miscanthus fibre after being submerged in the solution for 100 days are shown in Fig. 9. The small miscanthus fragments appear on the surface of the MP and the HM, and an obvious fibrillated surface is observed. This may be because part of non-cellulosic compounds is extracted by an aqueous solution. Previous studies show that more rough and fibrillated surfaces are observed in alkali-treated miscanthus due to the removal of some lignocellulosic components [13].

For miscanthus soaked in cement solution (CMP and CHM), some hydrated cement products are observed on the miscanthus surface, especially for the CHM. This may be due to heat treatment leading to a better bonding between the CHM and the cement paste. Moreover, some cellulose and xylose leached from miscanthus can be absorbed by cement paste particles [1] to further improve the compatibility of miscanthus fibre and cement paste.

Calcium is detected on the surface of the CMP and the CHM by SEM-EDS analysis. Moreover, the heat-treated miscanthus (HM and CHM) have a higher content of carbon (C) and less oxygen (O) compared to the raw miscanthus (MP and CMP). Silica is also observed on the surface of the CMP and the CHM fibre due to the presence of hydrated cement

products.

### 3.3. Dimensional stability of miscanthus mortar

#### 3.3.1. Drying shrinkage

The bio-based materials are quite sensitive to humidity in the environment, and the shrinkage and swelling of bio-based materials have a negative impact on the performance of bio-based concrete. As shown in Fig. 10a, the drying shrinkage of miscanthus mortar increases rapidly within the first month after demolding, the drying shrinkages of the mortars RM1, HM1 and MP1 at 30 days account for 85.5%, 83.1% and 87.3% of the total observed drying shrinkage value. Besides, the mortar HM1 significantly reduces the drying shrinkage by 8.4% at 114 days, the mortar MP1 has no obvious effect on the drying shrinkage, with an increase of 1.4%, compared to the mortar RM1. However, as the content of miscanthus powder increases from 1% to 2%, the drying shrinkage of the mortars MP1.5 and MP2 increases. Previous studies show that the miscanthus ash obtained by heat treatment exhibits high efficiency for reducing the autogenous shrinkage of cementitious materials [20]. This may be attributed to the good compatibility with the mortar, resulting in a decrease in micropores between the miscanthus fibre and the mortar matrix interface. In addition, the mass loss of miscanthus mortar shows a trend similar to drying shrinkage, which significantly reduces within the first month, as shown in Fig. 10b. The mass loss of the mortars RM1, HM1 and MP1 at 30 days account for 96.7%, 97.7% and 98.8% of the total mass loss value.

After the miscanthus mortar is cast, cement paste and miscanthus fibre are close to the saturated state. For miscanthus mortar, the drying shrinkage is mainly caused by the moisture loss from the C-S-H matrix and the porous miscanthus fibre, which will significantly affect the volumetric stability. The relationship between mass loss and drying shrinkage of miscanthus mortar can be fitted as:

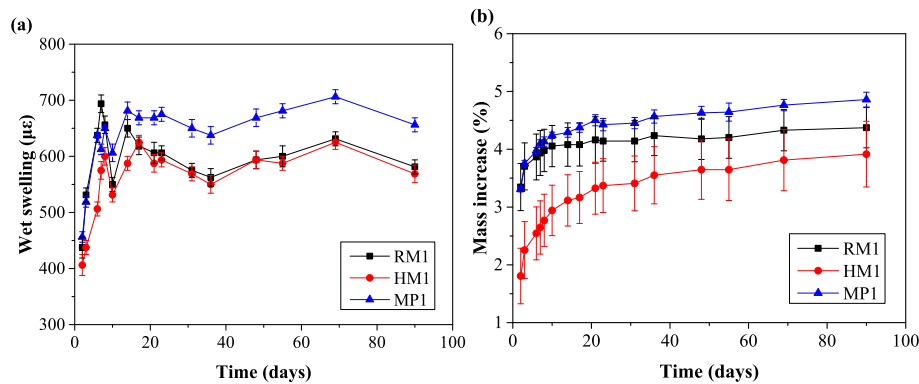


Fig. 11. (a) wet swelling and (b) mass increase of miscanthus mortar.

$$y = e^{x+A} \quad (1)$$

where  $y$  is the drying shrinkage of miscanthus mortar;  $x$  is the mass loss of miscanthus mortar (in %);  $A$  is an empirical constant obtained by experiment, the larger the value of  $A$  in the Eq. (1), indicating that the miscanthus mortar has more mass loss when reaching the same drying shrinkage.

### 3.3.2. Wet swelling

The wet swelling and mass increase of miscanthus mortar are shown in Fig. 11. The wet swelling of the mortars HM1 and RM1 shows the same trend, and the wet swelling of the mortar MP1 is significantly higher than that of the mortars HM1 and RM1 due to the high porosity of the mortar MP1. The wet swelling of miscanthus mortar is mainly the volume change caused by water gradually entering the miscanthus mortar from the microporous channels. The mass increase of the mortar HM1 is significantly lower than that of the mortars RM1 and MP1. The water entering the miscanthus mortar is one of the main reasons for the strength degradation of the miscanthus mortar and the dissolution of some cellulosic components. However, increasing the dimensional stability of miscanthus by heat treatment has a positive effect on reducing the wet swelling characteristics of miscanthus mortar.

## 4. Conclusions

The effects of the raw miscanthus (RM), heat-treated miscanthus (HM) and miscanthus powder (MP) on the long-term performance of bio-based miscanthus mortar are investigated in the present study. The physical and mechanical properties of miscanthus mortar, strength degradation behavior after being submerged in an aqueous solution for 90 days are analyzed. Moreover, the degradation mechanism of miscanthus fibres in water and in a cement solution is evaluated by a multiple analytical approach, including FTIR, XRD and SEM analyses. The dimensional stability of miscanthus mortar including drying shrinkage and wet swelling are discussed. The following conclusions can be drawn based on the acquired results:

- (1) The mortar containing heat-treated miscanthus of 1 vol% (HM1) shows an outstanding compressive strength and flexural strength, with an increase of 82.7% and 26.9%, respectively, compared to the control mortar containing raw miscanthus (RM1), thanks to the lower porosity and enhanced compatibility between fibres and mortar matrix.
- (2) The mechanical strength degradation of bio-based miscanthus mortar after being soaked in an aqueous solution is obvious in the first month. The sugar or other cellulosic components of miscanthus fibre are dissolved in the alkaline environment created by ions that are leached from the miscanthus mortar, which is

beneficial for the strength development, resistance to degradation and dimensional stability of bio-based miscanthus mortar.

- (3) The leachates of the cement solution are highly alkaline, which causes partial dissolution of the cellulosic components of miscanthus fibre. The higher crystallinity of miscanthus fibre after being soaked in water for 60 days is attributed to the removal of some non-crystalline compounds (waxes, pectin, etc.). The degradation of miscanthus fibre such as small miscanthus fragments and the fibrillated surface are observed on the miscanthus fibre surface by microscopic image analysis.
- (4) The morphology and content of miscanthus fibre have an obvious influence on the dimensional stability of miscanthus mortar. The drying shrinkage and wet swelling of miscanthus mortar increase rapidly within the first month because of the moisture loss from the mortar matrix and the porous miscanthus fibre. The heat-treated miscanthus fibre can be applied to significantly improve the dimensional stability of bio-based miscanthus mortar, and is more durable in mortar.

### CRediT authorship contribution statement

**Fan Wu:** Investigation, Methodology, Data curation, Validation, Funding acquisition, Writing – original draft. **Qingliang Yu:** Conceptualization, Supervision, Project administration, Funding acquisition, Writing – review & editing. **H.J.H. Brouwers:** Supervision, Funding acquisition, Writing – review & editing.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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