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Acoustic performance and microstructural analysis of bio-based lightweight concrete containing miscanthus

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HIGHLIGHTS

- Densities and porosities of different forms of miscanthus fibers are measured.
- Influential factors of fibers on mechanical properties of Miscanthus Lightweight Concrete (MLC) are evaluated.
- Acoustic absorption properties of MLC are characterized with impedance tube.
- Reaction kinetics of MLC is investigated through the combination of XRD, TG, hydration heat.
- The ionic behaviour of Ca^{2+}, Na^{+} and K^{+} in the presence of miscanthus fibers is evaluated.

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ABSTRACT

Miscanthus Giganteus (i.e. Elephant Grass) is a cost-effective and extensively available ecological resource in many agricultural regions. This article aims at a fundamental research on a bio-based lightweight concrete using miscanthus as aggregate, i.e. miscanthus lightweight concrete (MLC), with the special focus on the acoustic absorption property and interaction between miscanthus and Portland cement hydration. The effects of the content, particle size and treatment of fibers on the performance of MLC, including the flowability, strength and acoustical properties, are investigated. Furthermore, the effect of miscanthus on cement hydration is analysed by isothermal calorimetry, X-ray Diffraction, thermogravimetry, and scanning electron microscopy. It is demonstrated that the sound absorption of MLC is dramatically improved with the increasing content of miscanthus. The results show that there is a certain amount of closed internal pores in the composites, which contribute to this enhanced acoustic absorption performance.

1. Introduction

The rising agricultural output and the rapid expansion of bio-based manufactures have recently produced great amounts of agricultural wastes. While majority of the bio-wastes cannot be efficiently handled and applied. Bio-based insulating materials made from waste and recyclable plant fibers are interesting alternatives to those obtained from fossil carbon, which are also sustainable and eco-friendly to the environment. For the sake of constructing environmentally-friendly and cost-effective buildings, it is of great interest to design green and low cost building materials to reduce the environmental impact, particularly related to CO_2-emission.

Natural fibers as lightweight aggregate of building materials are developing fast and widely used now, for instance hemp, straw, flax and miscanthus [1]. Among other plant-based natural fibres, the adoption of miscanthus has drawn great attention attributed to its widespread availability and lack of competition with food and animal feed [2]. Miscanthus is a perennial plant, located for several years (up to 20 years), which reduces costs of crop establishment. Compared to wood, miscanthus has a high content of parenchyma, surrounded by a tough fibrous structure. It therefore combines a high rigidity with a low density [3]. The modulus of elasticity of Miscanthus Giganteus and Miscanthus Sinensis varies between 2 and 8 GPa. Miscanthus has other structural component like parenchyma, which provides the thermal insulation, and around the parenchyma there are three rings with relevance to firmness. Moreover, its epidermis contains thick sclerenchyma characteristics and radial allocation of vascular bundles with its own firmness texture. In addition, miscanthus is considerably stronger than straw, besides its chemical composition like silicon,

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it could represent a suitable basic material in construction materials [4].

Besides its general use as structural composite constituent, miscanthus can give additional value due to its expected excellent acoustic performance. Therefore, it is promising to develop miscanthus lightweight concrete, which is expected to have a good performance in acoustical absorption because of its lightweight and porous structure. Miscanthus has a significantly lower bulk density than normal weight aggregates and its porosity has a significant effect on its acoustical property [5]. Constructions built with miscanthus concrete can lower the environmental impact dramatically compared with cellular concrete. Moreover, the lightweight cement-miscanthus composite is a completely recyclable material, which can be ground and later used to produce new lightweight composites [6,7].

Many researchers have studied the acoustic absorption performance of plant-based construction materials. Sound absorption coefficient is the fraction of sound energy absorbed by a material and measured by means of standing waves in impedance tube, ranging from 0 to 1, are often rather low for normal Portland cement concrete, which ranges from 0–0.1 in most cases. Various kinds of other construction materials have been studied for obtaining an enhanced sound absorption property, for instance cellular [8] and autoclaved [9] concretes, and concretes incorporating plant materials like hemp [5]. These construction materials all have porous structures which are beneficial for sound absorption. Because sound waves will convert to heat through the pores inside the matrix. Miscanthus-cement concrete can also be characterized as a high porosity material. The existence of pores of different scales, which consist of inter-particle pores (between the miscanthus fibers) and intra-particle pores (inside the miscanthus fibers), enables the dissipation of sound waves. Profound study by Gle et al. has demonstrated the good sound absorption property of plant fibers-based concrete by experimental [5] and modelling [10] approaches. Cezero et al. [11] studied the influence of binder to fibers proportion, observing a dramatic decrease of acoustic absorption property with an increased Portland cement content. Therefore, it is reasonable to expect that MLC can possess the same acoustic property but further investigation is needed.

Despite all the promising benefits of miscanthus fibers, there exists severe concerns for practical application of natural plant fibers in Portland cement and concrete. Like other natural plant fibers, the miscanthus fibers have a large differentiation of fiber qualities, high water absorption ability, and issues of compatibility with cement paste, which is an alkali environment [12]. The dissolution of lignin and hemicellulose in the middle lamellae of fibers delays the cement hydration process and in return alkali degrades fibers as well [13]. Therefore in order to overcome the aforementioned durability problem, a proper understanding of the interaction mechanism of miscanthus is required [14]. Many researchers explored the methods to enhance the durability of concrete containing natural fibers. Romildo et al. [15] demonstrated that the methods comprise carbonation of the cement paste in a carbon dioxide atmosphere and pre-treatment of fibers with immersion in silica fume slurry before mixing with cement paste. Mármol et al. [16] suggested that the use of low alkalinity cement paste incorporated with supplementary cementitious materials could be an approach to relieve the severe damage on cellulose fibers.

The present research aims at investigation of an environmentally friendly bio-based lightweight concrete with the addition of miscanthus particles, with the special focus on acoustical absorption property and the interaction between Portland cement hydration and miscanthus. Pre-treated miscanthus fibers with different sizes and percentages are adopted to prepare the miscanthus based lightweight composites. Furthermore, the mechanical and acoustical properties are studied. Moreover, the interaction mechanism between miscanthus and cement paste is investigated through advanced test technologies, including X-ray diffractometry, thermogravimetry and isothermal calorimetry. This work will contribute to a deeper understanding of miscanthus usage in construction materials, especially for non-structural walls and ceilings, indoor furniture or some other outdoor structures like noise barriers.

2. Materials and experiments

2.1. Raw materials

2.1.1. Miscanthus

Miscanthus is provided by NNRGY Company (the Netherlands). The miscanthus is harvested in winter and further treated by the company. The morphology of the raw miscanthus fibers is shown in Fig. 1. The size and shape of miscanthus fibers are varied, so sieving is required for producing a regular size of the fibers. 2 mm and 4 mm size sieves were adopted in this study and fibers were sieved in a sieving machine. Fig. 1(a) and (b) show the 0–2 mm and 2–4 mm size fibers in bulk, respectively. Meanwhile miscanthus powder is another type of product from the company, which is shown in Fig. 1(c). The lengths of the fibers are about 2 to 20 mm long while the diameters are classified by the sieving process. Therefore the sieving process is a sorting of diameter rather than of length.

The chemical composition of miscanthus is analysed via acid hydrolysis method. Monomeric sugars after H_2SO_4 hydrolysis measured with HPAEC (High-Performance Anion-Exchange Chromatography) is presented in Table 1. The leachate was prepared by boiling the fibers for 2 h in water with a water to fiber ratio of 5.

2.1.2. Cementitious materials

The cement used in this research is Portland Cement CEM I 52.5R, provided by ENCI, Heidelberg Cement (the Netherlands). The supplementary cementitious material is ground granulated blast furnace slag (GGBS), provided by ENCI as well. The chemical composition of cement and GGBS were analysed by X-ray fluorescence. The results are shown in Table 2. The used slag has a median particle size (d50) of 12.43 μm and a specific density of 2.93 g/cm^3, which are measured by Mastersizer 2000 and Helium pycnometer, respectively. A polycarboxylic ether based superplasticizer (SP) is adopted to modify the flowability of the designed MLC.

2.2. Experimental

2.2.1. Characterization of miscanthus

2.2.1.1. Density and porosity. Bulk density is defined as the mass of particles of one material divided by the total volume they occupy. The total volume includes particle volume, inter-particle void volume and intra-particle pore volume. The bulk density of miscanthus fibers was determined with graduated cup. In brief, fibers were filled in a 1 L graduated iron cup and the mass of the cup before and after filling was measured. No vibration or compaction was adopted in the filling process. Then the bulk density was calculated according to

$$\rho_b = (M - M_0)/V_0 \quad (1)$$

where ρ_b is the bulk density of miscanthus, M_0 and M are the mass of graduated cup before and after filling, V_0 is the volume of the cup, which equals to 1 L.

Particle density is defined as the density of material that particles are composed of, excluding the inter-particle void volume. The particle density of miscanthus fibers was determined according to the Archimedes method. In brief, miscanthus fibers were heated to 80 °C in an oven for 12 h. Afterwards, 20 g of dry fibers was

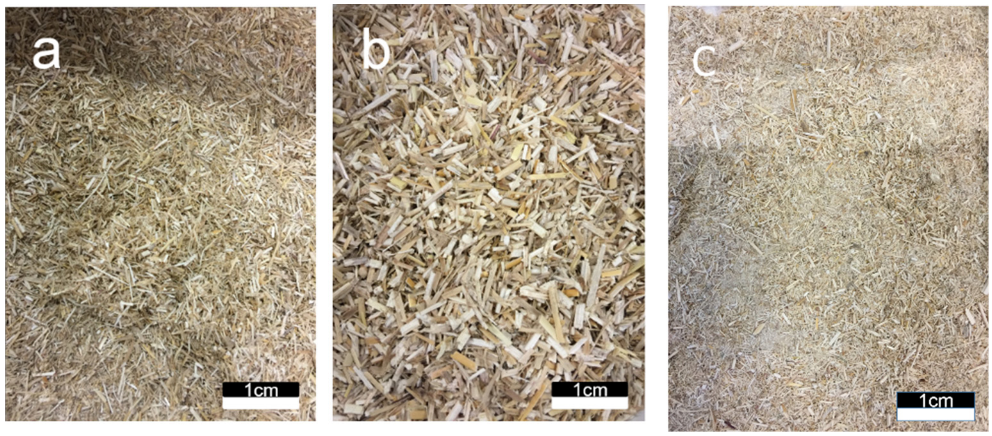


Fig. 1. Pictures of the raw miscanthus.

Table 1
Concentration of sugar leached from miscanthus.

Monomeric sugar (mg/ml)	Arabinose	Galactose	Glucose	Xylose	Mannose
Concentration	0.06	0.09	0.19	0.16	0.05

Table 2
Oxides composition of the used cement and GGBS (wt%).

Oxides	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	K_2O	Na_2O	LOI
Cement	64.60	20.08	4.98	3.24	3.13	1.98	0.53	0.27	0.4
GGBS	4.44	54.62	24.42	7.21	0.46	1.43	1.75	0.73	4.5

immersed in water for 6 h and then the excess water was removed by filtration. Water remaining on the surface of saturated fibers was wiped out by carefully clapping using a filter paper. Then the mass of water saturated fibers was determined. The particle volume was then determined through immersing the saturated fibers in a specific volume of water. Then the increased volume of water was the particle volume V_p. The particle density was calculated according to

$$\rho_p = M_d/V_p \tag{2}$$

where ρ_p is particle density, M_d is the dry mass of fibers, V_p is the particle volume of fibers.

Skeleton density is defined as the density of material that excluding the inter-particle void volume and intra-particle pore volume. The skeleton density of miscanthus was measured by a Helium pycnometer (AccuPyc II 1340 Micromeritics). This density is closely related to the volume of the particles without intra-particle pores and the mass of the skeleton [17].

The total connected porosity of miscanthus fibers is noted as Φ in this research. Miscanthus fiber is a granular aggregate, therefore it can be represented by two kinds of porosities, the intra-particle porosity Φ_{intra} and the inter-particle porosity $\Phi_{inter.}$ The interparticle porosity is defined as the pore volume between particles divided by the total bulk volume of materials possess. Inter-particle porosity, which was determined according to

$$\Phi_{inter} = 1 - (\rho_b/\rho_p) \tag{3}$$

where Φ_{inter} is the inter-particle porosity of miscanthus fibers, ρ_b and ρ_p are bulk density and particle density, respectively.

The intra–particle porosity is defined as the total pore volume inside the particles divided by the particle volume materials possess. Intra-particle porosity was determined according to

$$\Phi_{intra} = (M_s - M_d)/\rho_0/V_p \tag{4}$$

where Φ_{intra} is the intra-particle porosity of miscanthus fibers, M_d and M_s are the mass of dry fibers and water saturated fibers, ρ_0 is the density of water, V_p is the particle volume.

2.2.1.2. Water absorption. Water absorption was measured by immersing the miscanthus fibers in tap water and measuring the soaked miscanthus mass after 5, 10, 30 60, 120, 360, 720 and 2880 min, respectively. The water absorption capability was represented by the ratio of water soaked miscanthus weight and dry miscanthus weight. In brief, 20 g dry miscanthus fibers was immersed in water in a beaker. After specific period of time, the soaked fibers were filtrated so the water was removed while fibers remain on the filter paper. The fibers surface was then carefully clapped with another dry filter paper to remove the surface water. Finally the water-saturated fibers were weighed so the water absorption could be determined.

2.2.2. Pre-treatment of miscanthus

Miscanthus was pre-treated with two approaches, i.e. water saturated and cement impregnated. For the pre-wetting treatment, the dry miscanthus was immersed in water in a container for 6 h and then the excess water was removed using a 200 μm sieve. Then the miscanthus surface was carefully clapped with dry paper to remove the surface water and the miscanthus was ready for use. For the cement impregnation treatment, 200 g of dry miscanthus was placed in a mixing bowl and then mixed with cement slurry with a cement and water usage of 400 g and 400 g, respectively. Then the mixture was mixed for 2 min and then cured in room temperature for 3 days and the cement impregnated miscanthus was ready for use.

2.2.3. Design of miscanthus lightweight concrete

The mix design of miscanthus lightweight concrete is presented in Table 3. The water/binder ratio was designated as 0.45 and GGBS/cement ratio was fixed at 0.25. The GGBS was adopted to decease the alkalinity of cement matrix system and thus reduced the degradation of miscanthus fibers by alkaline environment. Miscanthus fibers replaced the binder volume at fixed percentage, which were 10%, 20%, 30%, respectively. Firstly, the cement and GGBS was blended in the mixer for about 1 min. Afterwards, water was added and mixed with the cement and GGBS for another 2 min. Subsequently the pre-treated miscanthus (both pre-wetted and cement impregnated) was added into the mixer and mixed for an additional 2 min. Later the superplasticizer was slowly added into the mixer until the end of the mixing procedures. The mixing process took about 7 min. The preparation process was under laboratory conditions. The room temperature during the mixing and measuring was constant, around 20 ± 2 °C.

2.2.4. Mechanical properties of miscanthus lightweight concrete

Workability of MLC was measured by the mini spread-flow test, following EN 1015-3 [18]. Fresh MLC was placed into a normal conical ring and followed by a 15 times jolting. The diameter of cement paste were measured 4 times after jolting and the average value was noted as the slump flow.

After mixing, the fresh MLC was cast into 40 mm × 40 mm × 160 mm moulds and vibrated on a jolting table. After 1 day from casting, samples were stripped from the moulds and placed in a climate chamber with a relative humidity of above 95% and temperature of about 20 °C, according to EN 12390-2 [19]. Then after 28 days curing, the density of MLC samples were measured according to EN 12390-7 [20].

The compressive and flexural strength measurements were proceeded following EN 196-1 [21]. The compressive and flexural strength value for each sample were obtained from the average of six and three specimens, respectively.

2.2.5. Acoustical property of miscanthus lightweight concrete

To determine the sound absorption coefficient, the impedance tube (see Fig. 2) at the Acoustical Lab at Eindhoven University of Technology was used. In this research, the impedance tube measures both the normal incidence absorption coefficient and surface impedance under well-defined and controlled conditions. In brief, the measurement principle is through generating a plane wave by a loudspeaker on the one side of the tube that was then spreading through the tube before reflecting by the MLC. The MLC changes the reflected wave and by collecting the generated standing wave, the sound absorption coefficient of the samples can be determined. The wave was measured at six different locations in the tube. This was done to increase the accuracy as described in the measurement protocol for the impedance tube. MLC samples were inserted at the end of the impedance tube and backed by a rigid surface.

2.2.6. Interaction between miscanthus and cement hydration

Qualification and semi-quantification of hydration products of pure cement paste and MLC were assessed by X-ray diffraction (XRD), which were carried out by an X-ray diffractometer D5000 Siemens. The X-ray diffraction (Cu tube, 40 kV, 30 mA, 3–75°, 0.02°/step, 0.2°/min, variable divergence slits V20) was adopted to detect the crystal structure present in MLC.

X-ray diffraction (XRD)/reference intensity ratio (RIR) analysis and DTG can be used to approximately determine the quantity of Ettrigite (AFt) and calcium hydroxide (CH) in the testing samples. XRD/RIR can determine the relative mass relations among different minerals in a sample, which is calculated according to the following equation:

$$\frac{I_i}{RIR_i} : \frac{I_j}{RIR_j} = \frac{m_i}{m_j} \tag{5}$$

where I_i and I_j are the strongest peak of minerals i and j, respectively. RIR_i and RIR_j are the reference intensity ratio of component i and j, respectively. M_i and m_j are the mass of component i and j, respectively.

Thermogravimetric curves of cement pastes with addition of miscanthus were assessed by a thermogravimeter and differential scanning calorimeter (TG/DSC), which were conducted by a NETZSCH STA449-F1 instrument in the range of room temperature to 1000 °C with a heating rate of 5 °C·min^{-1} under nitrogen atmosphere.

The cement hydration process was monitored by an isothermal calorimeter (TAM Air, Thermometric). The cement paste and miscanthus were blended with de-ionized water externally for about 1 min and vibrated with an electrical vibrator, then the mixed paste was injected into a sealed glass ampoule and loaded into the calorimeter. All measurements were conducted for 72 h under a constant temperature of 20 °C. The heat release and heat flow results were normalized by mass of binder, miscanthus and de-ionized water.

The microstructure of miscanthus fibers was observed by scanning electron microscopy (SEM), which was conducted by JOEL JSM-5600 instrument with an accelerating voltage of 15 kv.

The element concentration of cement paste mixtures adopting a constant water to cement mass ratio (W/C = 2) was examined by HPLC (High Performance Liquid Chromatography). In brief, cement paste was incorporated with different fiber contents (3%, 5% and 10% based on mass), which were marked as M3, M5 and M10, respectively. Afterwards, the plastic bottle that contains the paste was placed in a shaker machine to keep the paste fluid for 1 day

Table 3
Mix design of miscanthus cement composite.

Mixture	Cement (kg/m^3)	GGBS (kg/m^3)	Water (kg/m^3)	SP (%)[a]	Pre-treated[b] Miscanthus (kg/m^3)/(L/m^3)		
					Powder	0–2 mm	2–4 mm
Mix A	1020.7	255.2	572.5	0.1%	–	–	–
Mix B	918.6	229.7	515.3	0.1%	–	–	25.0/100
Mix C	816.6	204.2	458.0	0.1%	–	–	50.0/200
Mix D	714.5	178.6	400.8	0.1%	–	–	75.0/300
Mix E	918.6	229.7	515.3	0.1%	–	22.2/100	–
Mix F	816.6	204.2	458.0	0.1%	–	44.4/200	–
Mix G	714.5	178.6	400.8	0.1%	–	66.7/300	–
Mix H	918.6	229.7	515.3	0.1%	17.4/100	–	–
Mix I	816.6	204.2	458.0	0.1%	34.8/200	–	–
Mix J	714.5	178.6	400.8	0.1%	52.2/300	–	–

[a] : SP is used to adjust the flow of the composite the dosage should be adjusted in practical experiment.
[b] : Pre-treatment include water saturated and cement impregnated so every mix includes two kinds of miscanthus.

Fig. 2. Pictures of the used impedance tube.

with a rate of 250 rpm. Then the mixtures were filtrated and the calcium, sodium and potassium ion concentration of extracted aqueous phase were measured by HPLC.

3. Results and discussion

3.1. Characterization of miscanthus

3.1.1. Density and porosity

The bulk density, particle density, skeleton density, inter-particle and intra-particle porosity are shown in Table 4. The particle density is important for the volume calculation of miscanthus used in mix design. The skeleton densities of all types of miscanthus are almost the same, which is due to all miscanthus fibers are made up of cellulose, hemicellulose and lignin and the density of those are 1559 kg/m^3, 1520 kg/m^3 and 1260 kg/m^3, respectively [22]. Inter-particle porosity is a significant factor that influences the acoustical property of biomass. The primary concern is that cement paste may interact with miscanthus fibers. However, the number of affected inter and intra particle pores are difficult to calculate. Cement paste not only has a dramatic impact on the inter-particle pores, but also forms a mineralized coating on the fibers and blocks the intra-particle pores.

3.1.2. Morphology

The scanning electron microscopic images of 2–4 mm miscanthus fibers are shown in Fig. 3. The miscanthus fibers possess very porous structures. From Fig. 3(a) one can see that the surface of the stem has two paralleled linear structures, one is relatively smooth while the other one has regular nodes positioning on the surface of stem. The nodes increases the roughness of the fiber. Fig. 3(b) shows that the internal structure of the stem has a rather rough surface due to shredding process. Lots of cavities and walls can be observed and the size of pores are about 30–50 μm in average, which is smaller than that of hemp [5]. Fig. 3(c) shows the cross section of the fiber. A great amount of pores is shown inside the stem, while the outside of the stem has a denser structure, indicating fibers are more porous from outside to inside. It can be concluded that 2–4 mm miscanthus fiber possesses many cavities and pores inside the stem, which is beneficial when applied as a lightweight aggregate.

However, 0–2 mm miscanthus fibers possess smaller and closed pores, as shown in Fig. 4(a). The internal stem that is exposed outside shows more closed pores. Therefore, this type of fiber is not beneficial for acoustical property of lightweight concrete. The miscanthus powder shown in Fig. 5(b) suggests irregular shape and no pores can be clearly observed by SEM. Although this kind of fine green aggregate can act as filler in cement system, acoustic absorption property are not expected to be significantly improved by adding this type of aggregate.

3.1.3. Water absorption

The water absorptivity of different sizes of miscanthus is shown in Fig. 5. The miscanthus powder and 0–2 mm fibers have larger water adsorption capability compared to 2–4 mm fibers, reaching 524% and 396% at 2880 min, respectively. The finer fibers can absorb water more quickly at early age and reach equilibrium much sooner than larger fibers. For instance the miscanthus powder reached an absorptivity of 440% only 5 min, and increase constantly for another 60 min to 490%.

Meanwhile the absorptivity of 0–2 mm fibers also increase sharply, reaching 330% at 5 min and 380% at 60 min. The finer the miscanthus, the larger amount of water it absorbs; the observations under microscope indicates that the quality of the interpenetration is directly influenced by the porosity of the external wall of the vegetal, mainly by the degree of fragmentation [3]. The water absorption of 2–4 mm fibers reached 180% at 5 min and 290% at 2880 min. Obviously the time to reach equilibrium is longer and the slope of the absorption curve at early age is much lower than those of powder and 0–2 mm fibers. Large fibers contain the intact plant stem and consequently the ability of stem to absorb water becomes a key factor. However, the specific surface area of 0–2 mm fibers and miscanthus pow-

Table 4
Density and porosity of different sizes of miscanthus.

Miscanthus	Bulk density (kg/m^3)	Particle density (kg/m^3)	Skeleton density (kg/m^3)	Inter-particle porosity (%)	Intra-particle porosity (%)
Powder	155.6 ± 3.1	173.9 ± 2.2	1410 ± 10	10.5 ± 0.9	77.2 ± 0.5
0–2 mm	77.6 ± 1.3	222.2 ± 3.6	1406 ± 16	65.1 ± 1.2	58.1 ± 1.5
2–4 mm	119.4 ± 2.6	250.0 ± 1.5	1400 ± 21	52.2 ± 1.6	38.3 ± 2.1

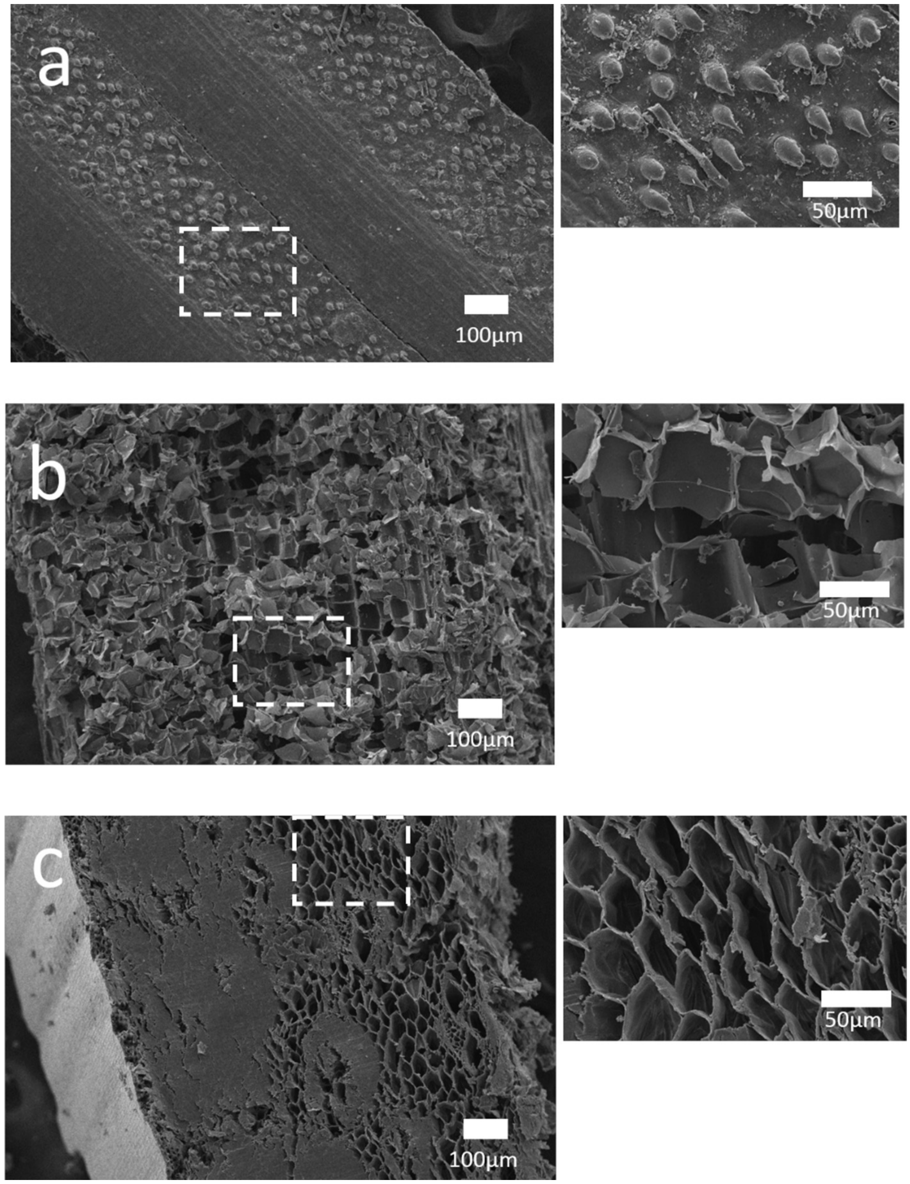


Fig. 3. SEM images of 2–4 mm miscanthus fibers: (a) surface (b) inside (c) cross section.

der are much larger than 2–4 mm fibers, resulting in higher water absorptivities.

3.2. Mechanical properties of MLC

3.2.1. Flowability

The slump flow of the different miscanthus-cement mortars with a w/b ratio of 0.45 is presented in Fig. 6. M0, M10, M20 and M30 represent 0, 10%, 20% and 30% miscanthus volume dosages, respectively. Miscanthus decreases the flowablility of MLC as the content of miscanthus fibres increases. For 2–4 mm and 0–2 mm fibers, the samples show similar slump flow results; while for miscanthus powder, the slump flow is better but also slightly decreases. Due to the pre-treatment of fibers, reduction in flowability is not attributed to the water absorption of miscanthus fibers, but closely related to the fiber aspect ratio and volume fraction in cement paste, which is in agreement with other literatures [23]. Mansur and Aziz concluded the workability of jute fiber cement composite decreased with the increasing length and volume fraction of the fibres [24]. Hence, in order to obtain a better slump flow for a high miscanthus dosage, more superplasticizer should be applied but in an appropriate amount to prevent segregation.

3.2.2. Density and strength

In general, the density and compressive strength of all samples dramatically decrease with the increasing content of miscanthus fibers. This is attributed to more voids and pores in the hardened cement paste introduced by the fibers, and the compressive strength of miscanthus itself is rather weak as well. The incorporation of miscanthus has less negative effect on the flexural strength, attributed to the crack bridging effect of fibers and its good tensile strength (180–260 MPa) [12].

The dry densities of miscanthus concrete with different sizes of pre-wetted and cement impregnated miscanthus is presented in Table 5. The dry density of concrete decreases dramatically from 1540 kg/m^3 to 1160 kg/m^3 with 30 vol% pre-soaked 2–4 mm miscanthus fibers. The obvious reduction in dry density is mainly due to the low density of the used miscanthus fibers. While for

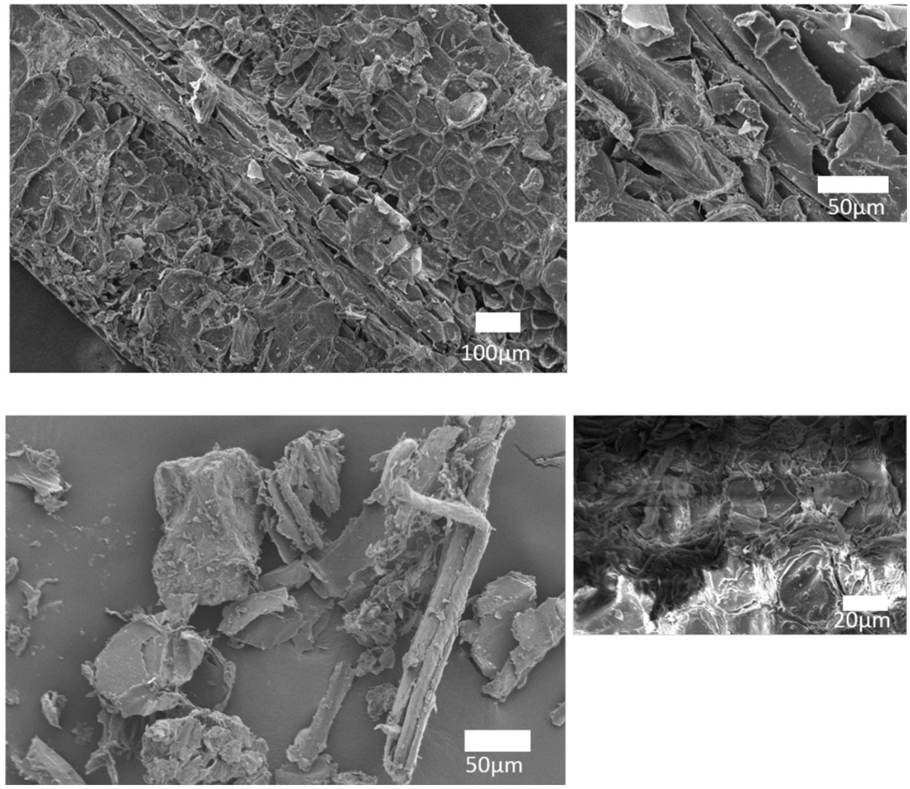


Fig. 4. SEM images of miscanthus: (a) 0–2 mm fibers (b) powder.

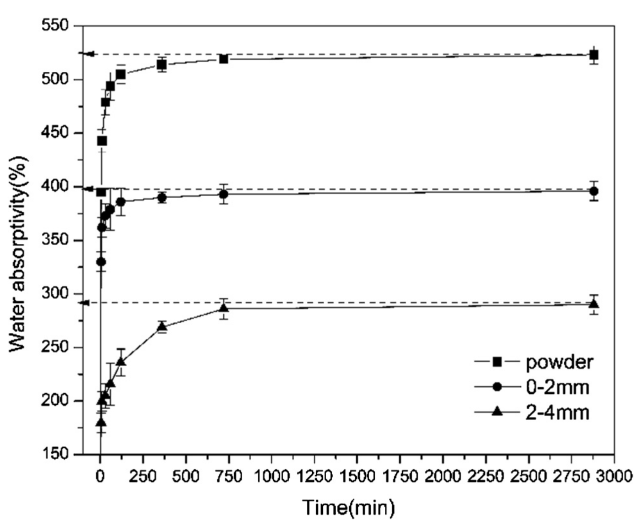


Fig. 5. Water absorptivity of miscanthus with different sizes.

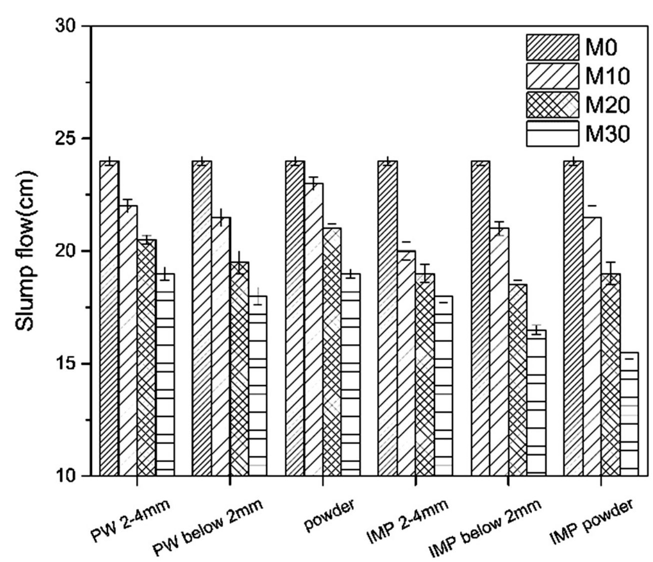


Fig. 6. Slump flow of different cement-miscanthus composite mortars with a w/b ratio of 0.45.

cement impregnated fibers, the dry density of MLC only reduces to 1440 kg/m^3, which is due to the increase of fibers particle density. The trend is also observed for 0–2 mm fibers-cement composite. However, for the powdered miscanthus concrete, the apparent dry density only reduced to 1255 kg/cm^3, which is due to the altered porous structure caused by the grinding process.

The compressive strength of miscanthus concrete with different sizes of pre-wetted and cement impregnated miscanthus at 28 days are presented in Table 6. The compressive strength of composites containing pre-soaked miscanthus decreases by 60–80% in comparison with the pure cement paste. Though several factors might contribute to the loss of mechanical strength, the decrease in composite bulk density and pores and voids introduced by fibers would mainly be responsible for such behaviour [2]. In fact, the fibers can act as voids in hardened cement paste because of their super lightweight property, so more fibers equal to more pores in cement. However, all cement impregnated samples show better performance than pre-soaked samples. Cement impregnated fibers possess a better mechanical strength and a higher density, the surface of fibers is much rougher as well. Moreover, the retarding effect of sugar leached from fibers has disappeared after the pre-treatment. Therefore the only problem is the defect bond between

Table 5
Dry density of miscanthus lightweight concretes (kg/m^3).

Miscanthus	Reference	Pre-soaked			Cement impregnated		
		2–4 mm	0–2 mm	Powder	2–4 mm	0–2 mm	Powder
10%	1540	1504	1479	1488	1520	1510	1520
20%	1540	1406	1333	1356	1436	1475	1487
30%	1540	1160	1129	1255	1340	1415	1460

Table 6
Compressive strength of miscanthus lightweight concretes at 28 days (MPa).

Miscanthus	Reference	Pre-soaked			Cement Impregnated		
		2–4 mm	0–2 mm	Powder	2–4 mm	0–2 mm	Powder
10%	55.4	14.8	13.4	17.58	23.14	23.69	28.21
20%	55.4	11.0	6.8	11.02	15.97	21.17	24.12
30%	55.4	5.21	2.26	3.59	10.78	14.59	16.87

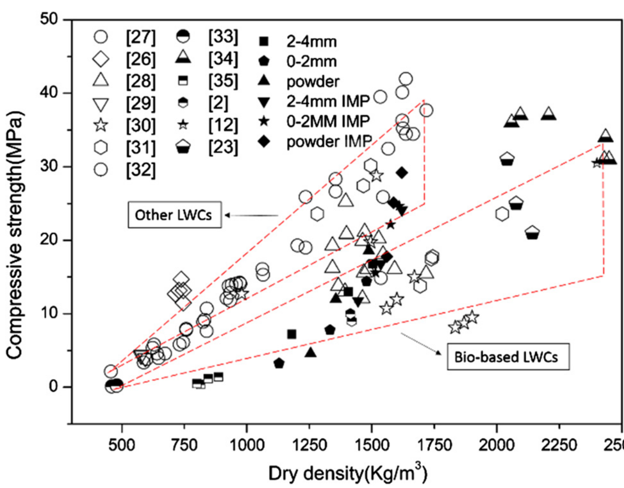


Fig. 7. Relationship between the compressive strength and dry density for different types of lightweight concrete at 28 days.

aggregates and cement paste, which can decreases the compressive strength dramatically [25].

The cross-link comparison between oven dry density and compressive strength of various types of lightweight concretes (LWC) is presented in Fig. 7. An obvious increasing tendency of the compressive strength can be noticed when the oven dry density of lightweight concretes increases from around 300 kg/m^3 to about 2100 kg/m^3 [2,12,23,26–35]. Furthermore, it can be noticed that the compressive strength of miscanthus lightweight concrete is lower than those of other LWCs at the same density range, especially at low densities, but slightly higher than those of other bio-based LWCs, which implies that the developed miscanthus lightweight concrete obtain moderate mechanical performance between these two kinds of LWCs.

The flexural strength of miscanthus concrete at 28 days is presented in Table 7. The flexural strengths of all composites manufactured with miscanthus do not dramatically change compared to the pure cement paste. For 2–4 mm fibers, the flexural strength slightly decreases with the increasing amount of fibers and cement impregnated samples have a better performance. For 0–2 mm fibers, the flexural strength dramatically decreases, especially for 30% pre-soaked miscanthus sample. However when the fiber is cement impregnated, the flexural strength of 30% content sample is much higher, even exceeding the reference sample at 28 days. For miscanthus powder, the flexural strength is comparable with the reference at low miscanthus dosages, but becomes very low at 30%. When powder is cement impregnated, the strength becomes higher than pure cement paste, indicating cement impregnated miscanthus powder can reinforce the flexural strength of cement mortar. Overall, the smooth flat surface of the pre-soaked fibers leads to a poor bonding with the cement paste and a fiber pull-out failure without any stress transfer [12]. Hence the adoption of pre-wetted fibers as reinforcement in concrete is limited. However, cement impregnated fibers can have a positive effect on flexural strength of cement composite.

3.3. Acoustic absorption property

In this research, acoustical characterization was carried out on MLC incorporated with 2–4 mm miscanthus fibers, due to its different pore structure compared with 0–2 mm fibers and powder. Concretes prepared with 2–4 mm miscanthus fibers pre-treated with pre-wetting treatment, were tested with 40 mm diameter impedance tube.

The acoustic absorption of MLC incorporated with 2–4 mm fibers for various contents of miscanthus is presented in Fig. 8. Fig. 8(a) shows that the pure cement paste has a rather weak sound absorption capability, which is due to the dense structure and less porosity of cement paste. However, when miscanthus fibers are incorporated, the absorption coefficient increases significantly with increasing the content of miscanthus, from 0.28 to 0.63. For 10% replacement of miscanthus, the frequency of absorption peak remains the same at about 700 Hz as that of pure cement. While for 20% and 30% replacement the sound absorption frequency is

Table 7
Flexural strength of miscanthus lightweight concretes at 28 days (MPa).

Miscanthus	Reference	Pre-soaked			Cement impregnated		
		2–4 mm	0–2 mm	Powder	2–4 mm	0–2 mm	Powder
10%	4.53	3.7	2.96	3.36	2.88	3.17	5.89
20%	4.53	2.14	1.98	3.2	2.9	3.56	5.98
30%	4.53	1.93	1.12	1.29	3.21	4.57	5.78

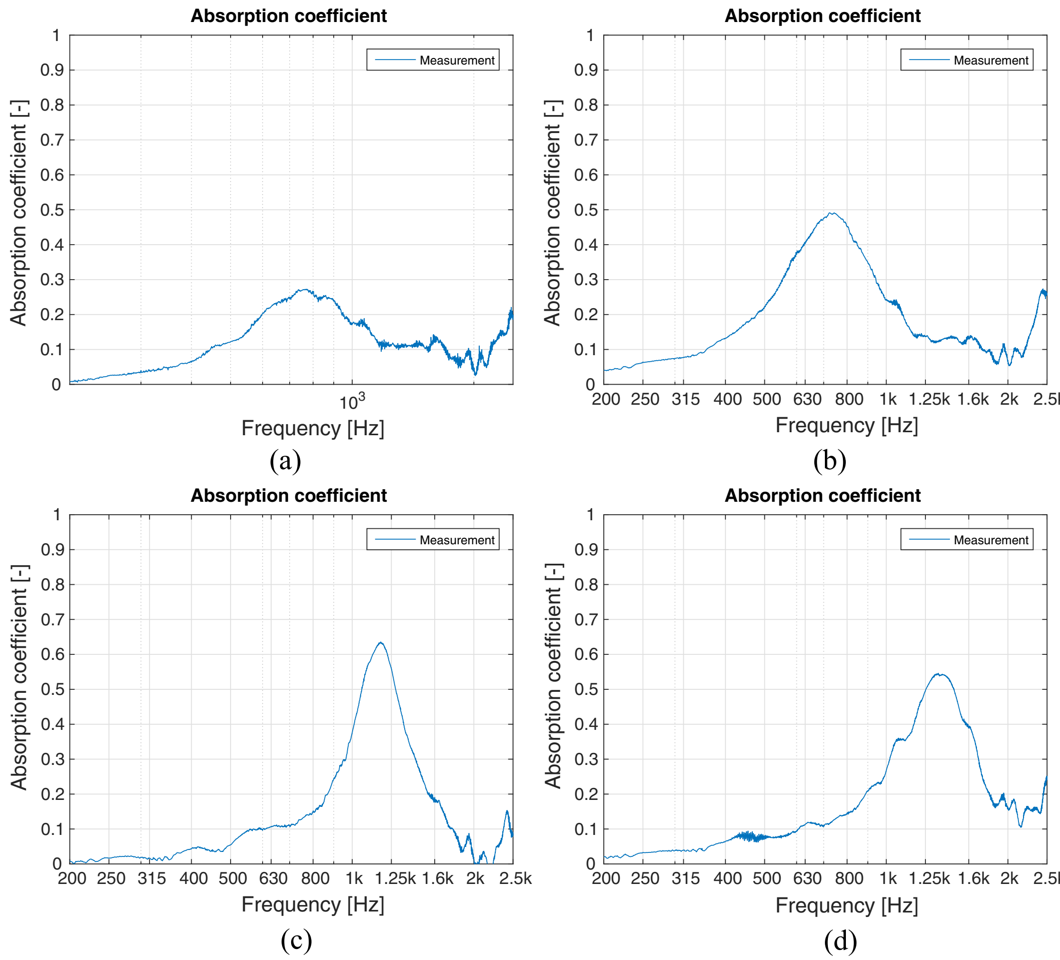


Fig. 8. Sound absorption of miscanthus cement composites for various content of 2–4 mm fibers: (a) Ref (b) M10 (c) M20 (d) M30.

shifted to higher value at about 1200 Hz and 1300 Hz, respectively. It is due to the increased content of miscanthus fibers which means more interconnected pores with different scales inside the cement paste [5]. Porous materials have a better sound absorption among high frequency range. The results imply that the sound absorption frequency shifted to high values with the increasing content of miscanthus fibers. The reduced density of MLC makes the frequency shift more to high values. Miscanthus fiber is a porous material with a great amount of open pores and cement paste porosity can increase with the incorporation of miscanthus. All these factors lead to a better performance in acoustic absorption.

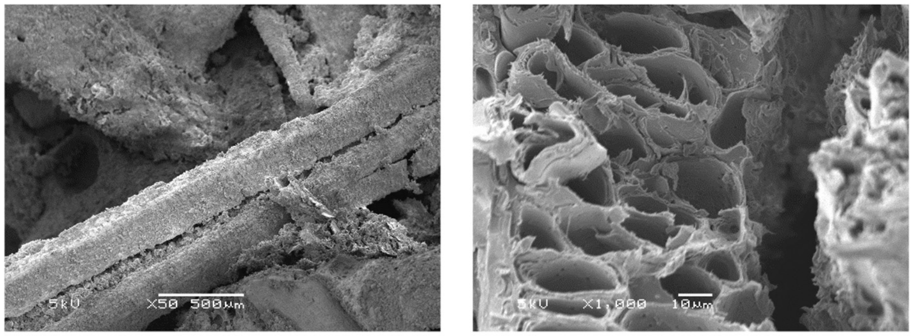
As can be seen in Fig. 7, the compressive strength of MLC reduces with the decreasing density of MLC. Moreover, there exists a reverse relation between bulk density and sound absorption coefficient. It can be concluded that a better sound absorption coefficient generally sacrifices the strength of miscanthus lightweight concrete. Nevertheless, proper processing method and raw materials selection can help to improve the mechanical properties. Specifically in this study, pre-wetting treatment and 2–4 mm fibers could obtain better sound absorption coefficient and compressive strength than that of other forms of miscanthus.

The SEM images of MLC matrix showing the microstructures is presented in Fig. 9. As can be observed in Fig. 9(a), inter-particle pores are obviously presented between the miscanthus fibers, which is an advantageous feature for sound absorption property. The 2–4 mm fibers can lower the workability of cement paste and consequently the porosity of matrix increases. Furthermore the microstructure shown in Fig. 9(b) exhibits a clear structure of fibers intra-particle pores. The dense distribution of pores in the straw of miscanthus can also have a positive influence on the acoustic property of MLC.

There exists also other types of green lightweight concrete used as acoustical absorber, especially for hemp shiv, which is similar as miscanthus concrete. Starch-hemp composite designed by A.T. Le et al. [36] can reach a sound absorption coefficient of 0.7 at frequency of 1250 Hz. Hemp concrete [5] prepared with lime binder can reach a sound absorption coefficient of 0.6 but at low frequency of about 300 Hz, which may be due to the larger impedance tube adopted (a length of 5.5 m and a square section of 60×60 cm^2, samples are 10 cm in thickness). Other types of green aggregate such as sunflower stalks and corn stalks were also investigated [37,38], but the results are not that good compared with the hemp and miscanthus fibers, which may be explained by the fabrication method and the nature of vegetables. Gle et al. [5] demonstrated acoustical properties are influenced by the pores of plant-based aggregate concrete, which is mainly combined with micro grade pores of cement paste and granularity of plant particle, and larger pores among the plant aggregates. Therefore, the acoustic absorption of MLC falls in between the perfect acoustic absorption of vegetable fibers, and the variation of the sound absorption of cement paste.

3.4. Interaction between miscanthus and cement paste

The influence of miscanthus fibers on cement hydration was analysed by advanced testing technologies. The X-ray diffraction patterns of pure cement paste and cement paste with 10% miscanthus fibers cured for 3 days are presented in Fig. 10. It is evident



(a) (b)

Fig. 9. SEM images of fracture surface of MLC.

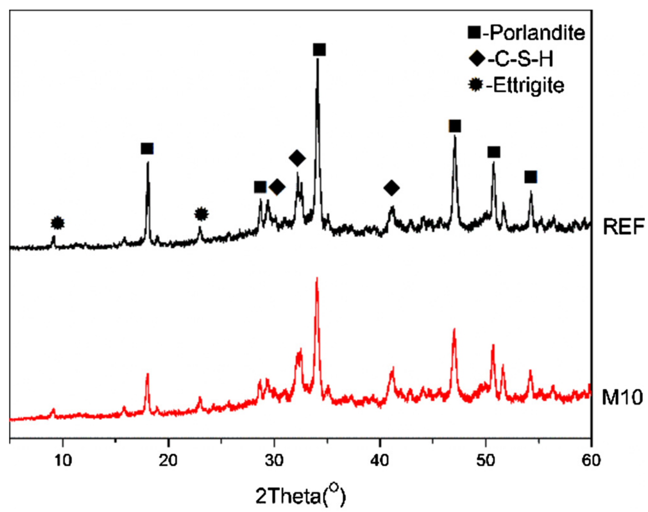


Fig. 10. XRD patterns of pure cement paste and cement paste with 10% miscanthus fibers cured for 3 d.

that the hydration products are primarily C-S-H gel, portlandite and ettringite. The characteristic peaks of portlandite of pure cement paste are much higher than those of M10, indicating a higher hydration degree of cement. According to the XRD/RIR analysis, the mass percentage of Portlandite in REF sample is 14.43% at the age of 3 days, while for M10 sample this value is only 6.78%. Furthermore, it is calculated that the percentage of Ettrigite in hardened cement paste of REF and M10 are 2.66% and 1.25%, respectively. Meanwhile the amount of C-S-H gel generated is also much larger than that of M10, which may due to the retarding effect of sugars dissolved from miscanthus. The hypothesis of organic retarding effect on cement hydration needs further confirmation. The C-S-H gel is the primary phase for strength of hardened cement paste, which explains when 10% miscanthus fibers is incorporated, a much lower strength is obtained. Similar XRD results are obtained by other researchers studying the interaction of plant fibers and cement. Fan et al. [39] demonstrated that the intensity of porlandite peaks decreased dramatically for the plant-cement composites. It is attributed to poisonous organic extracted from wood that leads to a reduction of portlandite formed.

Thermogravimetric curves of pure cement paste and cement paste with 3%, 5% and 10% mass percent miscanthus fibers cured for 3, 7 and 28 days are shown in Fig. 11. All test specimens present a significant mass reduction before about 100 °C due to the evaporation of water, which is physically bound in the cement paste. With the increasing temperature, the gradual and continuous mass loss ranging from around 180 °C to 420 °C is attributed to the breakdown of calcium silicate hydrate gels. It can be noticed that with the rising percentage of miscanthus fibers the C-S-H gels formation is decreased significantly for all the ages, indicating a slower hydration process.

The significant reduction of thermal gravity curve between 420 °C and 480 °C is attributed to the decomposition of calcium hydroxide, which is a direct indicator of cement hydration degree. It can be monitored that 3.0% mass loss is detected for pure cement paste at 3 days while for 10% miscanthus fibers only 1.3% mass loss is detected. Hence the cement hydration process is delayed since the very initial age.

Because the used cement is a rapid cement, the hydration is very quick during the first 7 days while for miscanthus cement the hydration degree is much slower, which can be evidenced by Fig. 11(b). However when it approaches 28 days, the hydration degree of 3% miscanthus cement is largely increased, indicating the retarding effect is gradually disappeared while for 5% and 10% miscanthus cement the increase of cement hydration degree is negligible. This may be due to the retarding effect of high concentration of leached sugars from miscanthus fibers, which poisoned the cement severely.

The normalized heat flows of pure cement pastes and pastes with the addition of different types of miscanthus fibers are presented in Fig. 12 (left). The characteristic peak of the acceleration period is situated at about 12 h after the beginning of the test, indicating the formation of reaction products. It can be noticed that as the miscanthus fibers are added, the peak of the acceleration period significantly transfers to later times and exhibits lower intensities, suggesting the reaction process of cement hydration is dramatically delayed with the addition of miscanthus. The results are in accordance to previous studies on the influence of miscanthus or other plant fibers on cement hydration [40]. Miscanthus dissolute organics in cement matrix, which poison cement and prevent the generation and growth of cement hydration products [41]. However the most severely poisoned sample is the one incorporated with 0–2 mm fibers, the intensity of the heat flow is the lowest while the time delayed is the longest as well. For 2–4 mm and the powder samples, they show similar trend and powder sample is slightly delayed longer while with more heat released.

Fig. 12 (right) shows the cumulative heat evolution of the pure cement paste and paste with the addition of different types of miscanthus fibers. For a fixed miscanthus content, test specimens with 0–2 mm fibers show the lowest total heat release, suggesting the existence of 0–2 mm fibers strongly delayed the cement hydration during the test period; however for specimens with 2–4 mm and powder miscanthus, they show dramatically larger total heat release, which indicates a less harmful impact on cement hydra-

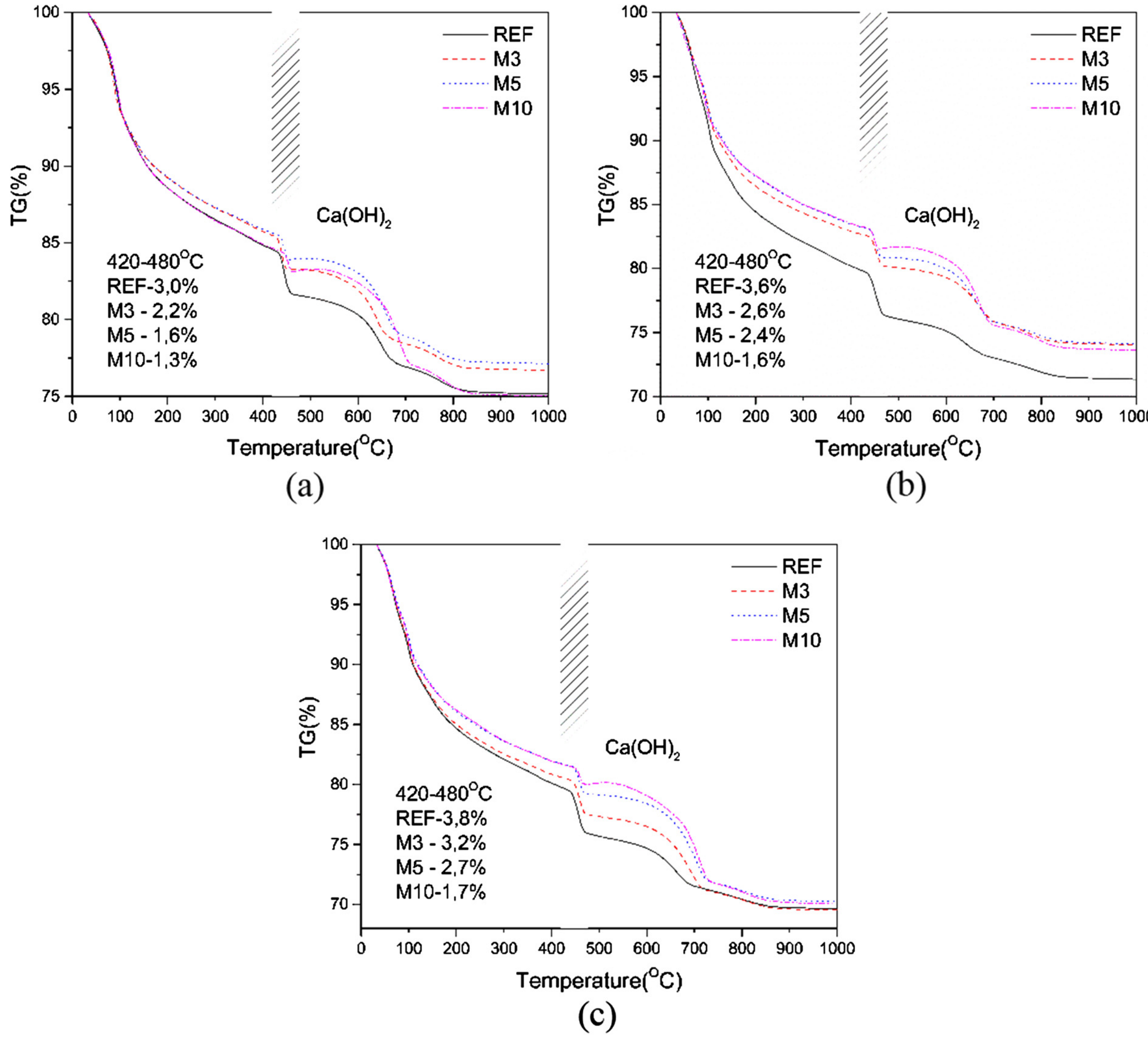


Fig. 11. Thermogravimetric curves of pure cement paste and cement paste with 3%, 5% and 10% miscanthus fibers cured for (a) 3 d, (b) 7 d and (c) 28 days.

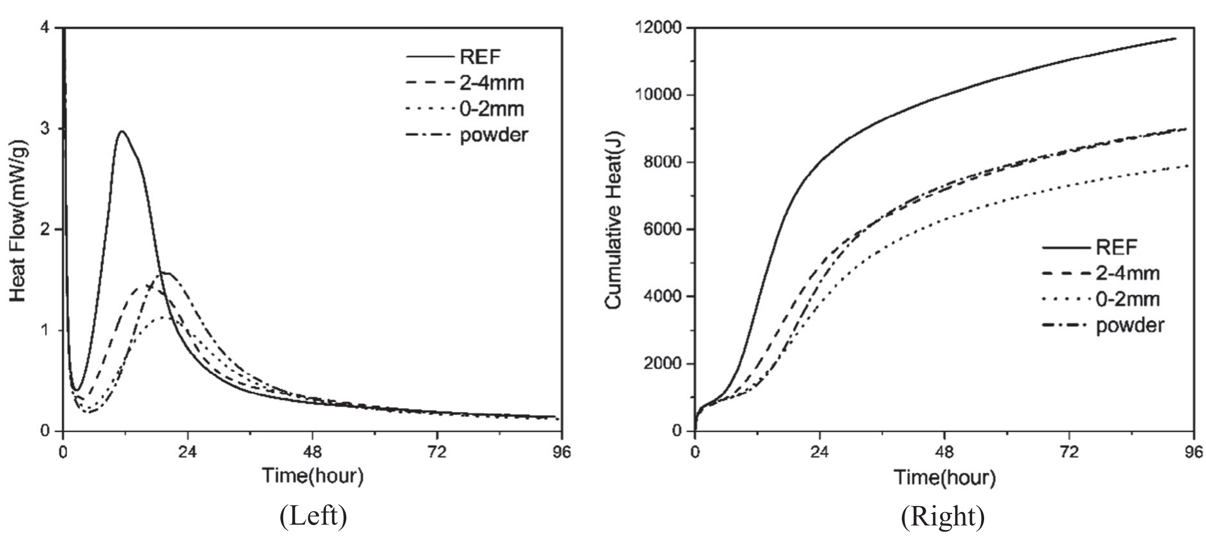


Fig. 12. (Left) Normalized heat flow, (Right) Cumulative heat release of pure cement pastes and with addition of different types of 5% miscanthus.

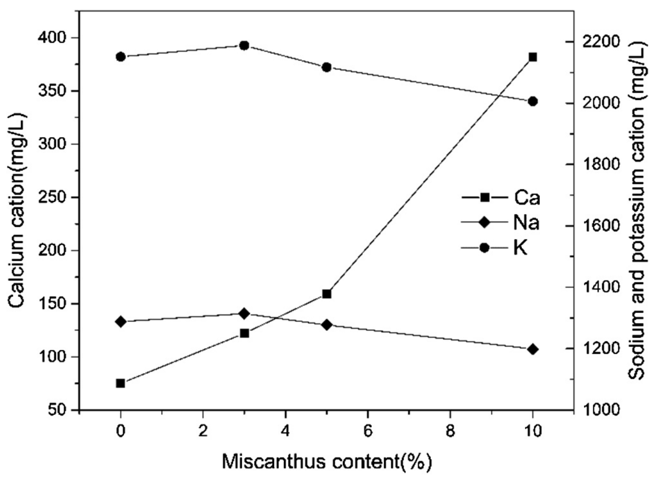


Fig. 13. Element concentration of cement pore solution with different miscanthus dosages.

tion. It is noteworthy that 0–2 mm fibers show darker extracted solution when immersed in water, indicating more organic materials dissolved in water, which is detrimental to cement hydration.

The element concentration in the pore solution with different miscanthus dosages is shown in Fig. 13. First of all, it supports the previous conclusion that cement hydration is delayed significantly with the increasing content of miscanthus. The calcium concentration in the cement filtrated solution increased from 75 to 382 mg/L when the miscanthus content raised from 0 to 10%, indicating that calcium is dissolved in cement solution, but it can only form a limited quantity of hydration product like C-S-H gel and calcium hydroxide. In fact, Biblis et al. [42] demonstrated that organic extractives like glucose and cellulose severely interrupt the cement reaction. The negative effect of organic materials is attributed to the ability of sugar to be bound onto the hydration product containing Ca^{2+}, hence interrupting the C_3S hydration and the formation of C-S-H gel. However no calcium absorption is observed, which is in disagreement with Sedan et al. [43], who discovered and described a reduction in calcium ion concentration of cement pore solution. The sodium and potassium concentration showed minor fluctuations, indicating the dissolution of cement in water is not influenced by the incorporation of miscanthus.

4. Conclusions

This study investigates the properties of a bio-based lightweight concrete containing miscanthus, including workability, strength, acoustical absorption, reaction kinetics and microstructure. Furthermore, the role of miscanthus in blended cement systems is discussed. Based on the current results, the following conclusions can be drawn:

- 2–4 mm miscanthus fibers obtain intact and large pore structure and perform better than those of 0–2 mm fibers and powder miscanthus with regard to acoustical properties.
- The high absorptivity of miscanthus fibers can be the obstacle for concrete mix design. Therefore, pre-treatment such as pre-wetting or cement impregnation is necessary to prepare miscanthus lightweight concrete.
- Workability of miscanthus lightweight concrete is closely related to aspect ratio of fibre and volume percentage in cement paste.
- The compressive strength decreases significantly with the addition of miscanthus, e.g. from 56 MPa to 15 MPa for 2–4 mm fibers (20% volume replacement) at 28 days. Nevertheless, flexural strength remains stable and for samples with cement impregnated fibers the strength is slightly enhanced, from 2.88 MPa to 3.21 MPa for 2–4 mm miscanthus fibers at 28 days. Cement mortar with pre-wetted miscanthus particles gains a lower strength than those of cement impregnated, but the density is lower as well. This is mainly caused by the denser aggregate resulted from cement impregnation.
- Acoustical absorption properties are significantly enhanced by incorporation of miscanthus fibers. More miscanthus represents more open pores and voids inside the cement paste and consequently more energy can be absorbed by the miscanthus cement composite.
- The interaction between miscanthus and cement starts at the initial age. All fibers show retarding effect on cement hydration, but 0–2 mm fibers has the worst effect on the cement hydration. The XRD evidence indicates the low strength of cement composite is also attributed to a less C-S-H gel formation. A high dosage of miscanthus can poison the cement paste, resulting in a lower hydration degree.

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