Hygrothermal properties of compressed earthen bricks

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Abstract

The present study investigates the relationship between bulk density and hygrothermal behaviour of compressed earthen bricks. The experimental results show that the thermal conductivity linearly increases from 0.5228 W/(m K) to 0.9308 W/(m K) as the bulk density increases, and that the equilibrium moisture content increases with increasing relative humidity. Hysteresis effects are observed. When relative humidity changes, compressed earthen bricks usually reach an equilibrium in four days and it means compressed earthen bricks can be used to regulate indoor relative humidity. The hysteresis values of compressed earthen bricks with different bulk densities are close to each other, especially low relative humidity, as the results of Brunauer-Emmett-Teller (BET) show that samples with different bulk densities have similar porous structure including specific surface area (15.5008 ~ 16.2091 m^2/g), micropore volume (0.000867 ~ 0.001221 cm^3/g) and mesopore volume (0.030785 ~ 0.032239 cm^3/g). Moreover, the hysteresis loops in this study belong to the type H3 hysteresis loops which indicate that there are some slitlike pores inside the matrix.

Keywords

Hygrothermal properties; Thermal conductivity; Hygroscopic behaviour; Porous structure; Compressed

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earthen brick.

Highlights:

- Thermal conductivity of compressed earthen bricks increases with increasing bulk density.
- The response of compressed earthen bricks to a relative humidity change is rather fast.
- Hysteresis values of compressed earthen bricks increased with increasing relative humidity.
- Hygroscopic properties of compressed earthen bricks with different bulk densities are close to each other.

1. Introduction

The increased use of non-renewable resources and increased greenhouse gas emissions result in growing environmental problems. The impact of building performance on the ecological environment gradually attracts more and more public attention. In the western world, buildings account for approximately a third of both all energy use and greenhouse gas emissions [1]. In China, energy consumption in commercial and residential buildings has considerably increased in recent years, the building energy demand will account for 35 % of the total energy consumption in 2020 [2]. The energy requirement for heating, cooling and ventilation accounts for the major part of the building energy consumption. Therefore, several solutions have been implemented to reduce energy consumption, e.g. promotion of increased levels of thermal insulation, use of renewable energy and promotion of energy efficiency [3,4]. Besides, the application of the environmental friendly building materials will be important to reduce embodied emissions (i.e. emission related to building material production and maintenance) [4]. Earth-based materials may be important in this respect [5,6]. They can also have hygrothermal properties that can result in a more stable indoor environment [7].

The hygrothermal properties of building materials can influence the ability of indoor climate stabilization of buildings. For example, a well-insulated building envelope can reduce the heat transfer through the building envelope and weaken the impact of outdoor climate changes on indoor environment. Materials with good moisture storage abilities, on the other hand, are able to absorb water vapour from

50 the air when the relative humidity increases and release water vapour when the relative humidity
51 decreases in order to maintaining a more stable indoor relative humidity [7,8]. In situations where a more
52 stable indoor relative humidity is desirable, the building envelope should have the ability of moisture
53 storage.

54 Soil and/or earthen materials have been used for construction purpose for thousands years; and
55 nowadays, approximately one half of the world's population still live in earthen buildings [7,9].
56 Compared with some common building envelope materials, i.e. fired clay bricks and concrete, earthen
57 materials have a larger moisture capacity [10]. This can be explained from that earthen materials are
58 typical porous materials that have a moisture storage capability caused by single layer adsorption, multi-
59 layer adsorption and capillary condensation [11]. More importantly, as a natural, sustainable and eco-
60 friendly building material, the abundant source of earthen materials can lead to direct site-to-service
61 application, thus reducing the costs caused by acquisition, transportation and production.

62 During the last few years, a growing interest has appeared for the hygrothermal properties of earthen
63 materials. Liuzzi et al. [9] and Cagnon et al. [12], respectively, conducted experimental studies to
64 compare the thermal conductivity values between earthen bricks from different regions; the results
65 showed that the differences of mineral composition and grading level have a huge impact on the thermal
66 conductivity values of earthen bricks. Hall et al. [13], Mansour et al. [14] and Tang et al. [15] studied the
67 effect of bulk density, water content and degree of saturation on the thermal conductivity of compressed
68 earthen bricks. Results demonstrated that there is a linear correlation between the thermal conductivity
69 and density and then the thermal conductivity significantly increases with increasing degree of saturation.
70 Taallah et al. [16], Ashour et al. [17] and Adam et al. [18] respectively added natural fibers and chemical
71 additives into earthen materials to produce stabilized earthen materials, where the results demonstrated

that natural fibers are able to reduce the thermal conductivity as the fibres contain a lot of pores. Conversely, cementitious products formed by hydration reaction increase the thermal conductivity of stabilized earthen materials. For hygroscopic behaviour of earthen bricks, Liuzzi et al. [9] and Cagnon et al. [12] measured earthen bricks from different regions and results indicated that there are obvious differences between them as mineral composition of earthen materials has a serious influence on the adsorption and desorption ability. Ashour et al. [19,20] presented that addition of natural fibers increases the equilibrium moisture content of earthen materials; however, cementitious materials, i.e. cement, lime and gypsum, have the opposite effect on the equilibrium moisture content [11,20]. McGregor et al. [21] indicated that the variation of density influences the pore structure and therefore affects the capillary condensation which leads to the significant increase of the equilibrium moisture content. Raimondo et al. [22] and Randazzo et al. [23] measured the pore size and specific surface area of earthen materials by helium pycnometry and Brunauer-Emmett-Teller (BET) method and obtained the correlation between the porous structure and hygroscopic behaviour.

It is noteworthy that the presented studies have essentially considered the thermal properties or hygroscopic properties of earthen materials, and very few papers have investigated the hygrothermal properties of earthen materials and, in particular, compressed earthen bricks. The objective of this study is to investigate how the porous structures of compressed earthen bricks vary with change of bulk density and then the effect of the porous structure on the hygrothermal properties of compressed earthen bricks. Additionally, such studies presented that bulk density has a significant effect on thermal or hygroscopic properties of earthen materials and that bulk density is a rural macroscopic physical indicator which is easier to control in the production process of earthen bricks. Furthermore, the aim of this study is to try to obtain a relationship between hygrothermal properties and bulk density in order to guide the

preparation of compressed earthen bricks to reach the requirements concerning hygrothermal behavior.

2. Experimental

2.1. Materials

The earthen materials used in this investigation derive from Turpan, located at 42°26' N, 89°5' W in the Xinjiang Uygur Autonomous Region, Northwest of China. The grading curves and the particle sizes of the earthen materials were investigated by grain size analysis, according to GB/T 50123-1999 [24]. The composition of the earthen material is 17 % clay (less than 5 μm), 51 % silt (between 5 and 50 μm) and 32 % sand (between 50 and 2000 μm). The test results are presented in Fig. 1.

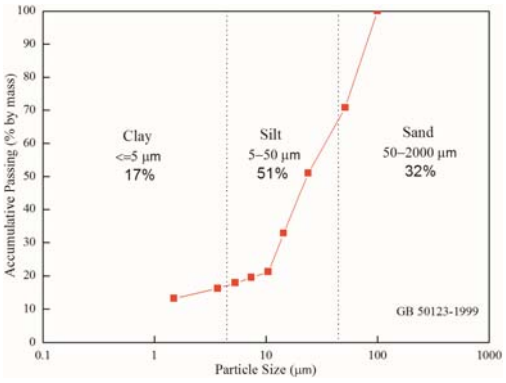

Fig. 1 Grain size distribution of earthen material used.

2.2. Sample preparation

Before the preparation of compressed earthen samples, the earthen material was sieved to remove the oversized gravel (larger than 2 mm diameter) and organic matter. The sieved material was dried in air at a temperature of 105 °C to obtain a constant weight. To produce the compressed earthen samples at different bulk densities, the dried material put into the mould was controlled and the mass of dried material inside the mould was determined by the calculation of target bulk density times volume of mould.

The classification of bulk density includes 1.5, 1.7, 1.9 and 2.1 g/cm^3 and the sample dimensions were 50 mm × 50 mm × 25 mm. Samples were prepared by a hydraulic press under a pressure of from 20 to 70 kN, as shown in Fig. 2. After compaction, the samples were placed in controlled laboratory conditions for 14 days to avoid cracking. The environmental temperature and relative humidity in the laboratory during the drying process were 20 °C and 60 %, respectively.

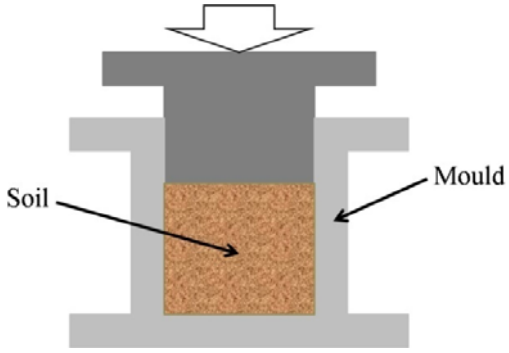

Fig. 2 Process of compaction of samples.

2.3. Characterization

2.3.1. Physical and chemical characterization

The earthen material was characterized by some basic tests including Atterberg limit and chemical composition. The Atterberg limits were determined by the Liquid-plastic Tester according to JTJ051-93 [25] and results are shown in Table 1. The plastic index is 5.5 % which means the earthen material belongs to silty clays and is suitable for production of compressed earthen bricks [26]. The chemical composition of the earthen material was estimated on the basis of x-ray fluorescence and results are given in Table 2. The earthen material in this study was mainly composed of silica, calcium oxide and alumina. Taking into account the above results, it could be concluded that the earthen material was rich in quartz, calcite and aluminum minerals.

127 Table 1 Atterberg limits of the earthen material.

Atterberg limits	
Liquid limit	23.7 %
Plastic limit	18.2 %
Plasticity index	5.5 %

128 Table 2 Chemical composition of soil used (wt%).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	MnO	ZrO_2
47.770	12.210	8.845	19.367	4.261	3.980	1.940	0.454	0.860	0.231	0.082

129 2.3.2. Adsorption-desorption isotherms

130 Adsorption-desorption isotherms are comprised of an adsorption branch which indicates the tested
131 specimen adsorbs water vapour from the surrounding at a series of relative humidity and a desorption
132 branch which presents the tested specimen releases water vapour to surrounding to reach an equilibrium.
133 In this study, method of saturated salt solutions was used to measure adsorption-desorption isotherms of
134 compressed earthen samples in order to describe the hygroscopic behaviour according to GB/T 20312-
135 2006 [27]. All samples were previously oven dried at 105 °C to reach a constant mass. Six relative
136 humidities, i.e. 32.8 ± 0.2, 43.2 ± 0.1, 52.9 ± 0.2, 64.9 ± 0.2, 84.3 ± 0.1 and 97.3 ± 0.3 % RH, were used
137 for tests and different relative humidity were obtained using saturated salt solutions (Table 3). The dried
138 samples were placed on the plastic meshes over airtight containers which were contained with various
139 saturated salt solutions from low to high, respectively (Fig. 3). The airtight containers were placed inside
140 a chamber at 20 °C and 60 % RH and then the samples were weighed periodically until the variation of
141 two consecutive results 24 h apart was less than 0.1 %. When the tested samples reached a moisture
142 equilibrium at 97.3 ± 0.3 % RH condition, the adsorption branch tests were finished and samples were
143 transferred immediately into the 84.3 ± 0.1 % RH condition to measure the desorption branch. The tested
144 samples were placed in the lower relative humidity after reaching an equilibrium at a certain relative

humidity. The desorption branch tests were completed when the tested examples reached an equilibrium at 32.8 ± 0.2 % RH condition.

Table 3 Saturated salt solutions used for obtaining different relative humidity.

Molecular formula	$MgCl_2$	K_2CO_3	$Mg(NO_3)_2$	$CoCl_2$	KCl	K_2SO_4
Relative humidity	32.8±0.2 % RH	43.2±0.1 % RH	52.9±0.2 % RH	64.9±0.2 % RH	84.3±0.1 % RH	97.3±0.3 % RH

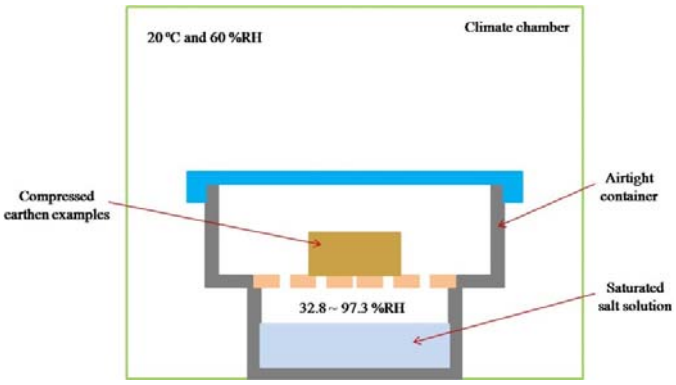

Fig. 3 Operation conditions for adsorption-desorption isotherms.

2.3.3 Thermal conductivity

The thermal conductivity values of compressed earthen samples were measured by using a Hot Disk apparatus (TPS-2500 S) according to ISO/DIS 22007-2:2015 [28]. Prior to testing, the equipment was calibrated with an expanded polystyrene board to ensure the accuracy of the tested results and the flatness of samples was checked to make a perfect contact between the sensor and the surface of sample, as shown in Fig. 4. Additionally, the samples were oven dried at 105 ℃ for 24 hours in order to measure the dry thermal conductivity of the samples .Each measurement was repeated three times and the mean value was reported.

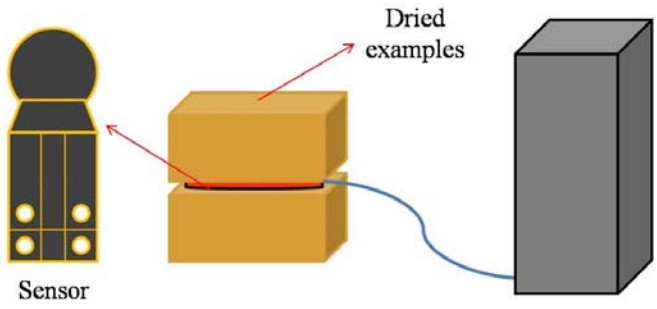

Fig. 4 Experimental setup for thermal conductivity measurements by using Hot Disk apparatus.

2.3.4 Pore structure

TriStar 3000 surface area and porosity analyzer was used to characterize the specific surface area and pore structure of the samples. The specific surface area, total volume of pores and pore size distribution are crucial parameters to describe the pore structure of compressed earthen materials. Specific surface area, pore volume and monolayer adsorption were determined by Brunauer-Emmett-Teller (BET) method. All the samples were degassed at 250 ºC overnight prior the nitrogen adsorption measurements. The nitrogen adsorption measurements were carried out at -196 ºC. The pore size distribution was determined according to the desorption curve of isotherms using the Barrett-Joiner-Halenda (BJH) method [29].

3. Results and discussions

3.1. Microstructure of compressed earthen bricks

The microstructure images of compressed earthen samples for different bulk densities are illustrated by scanning electron microscopy (SEM) in Fig. 5. It can be seen that there is a variation of pore quantity in compressed earthen samples at different bulk densities, the quantity of pore inside the matrix decreases with increasing bulk density. When the bulk density increases from 1.5 g/cm^3 to 2.1 g/cm^3, the matrix of

compressed earthen bricks becomes more and more compact and the pore diameter reduces significantly. When the bulk density is 2.1 g/cm^3, the visible pores barely exist in matrix of compressed earthen sample, which leads to a high compactness and a low porosity inside the matrix.

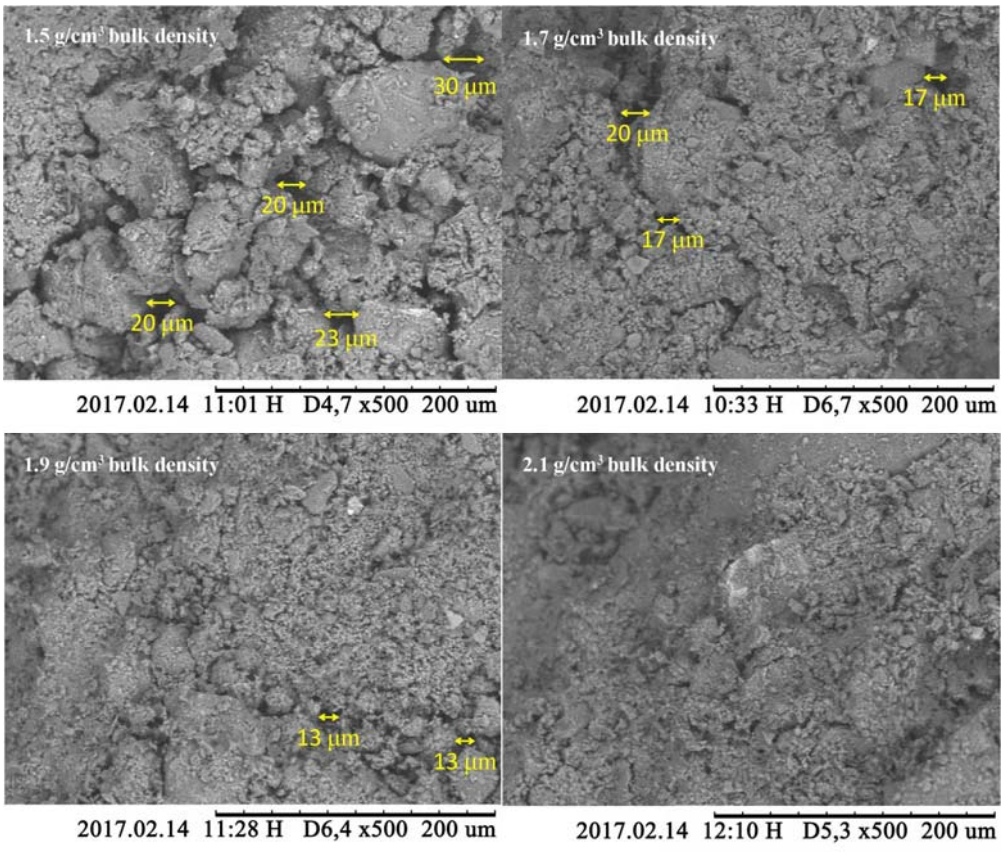

Fig. 5 SEM images of compressed earthen materials at different bulk densities.

3.2. Thermal conductivity

Thermal conductivity is one of the crucial parameters which are used to evaluate the thermal insulation of building materials. The lower thermal conductivity, the lower heat transfer through the building envelope. The effect of bulk density on thermal conductivity of compressed earthen bricks is presented in Fig. 6. In detail, the mean values of the thermal conductivity are reported with the uncertainty calculated as the standard deviation of the mean. As shown in Fig. 6, there is a linear correlation between the thermal conductivity and the bulk density, where the mean values of the thermal conductivity increase

from 0.5228 to 0.9308 W/(m K) with increasing bulk density. This phenomenon can be explained by the difference of thermal conductivity between air (about 0.026 W/(m K)) and compressed earthen bricks (between 0.5228 and 0.9308 W/(m K)), and increasing the bulk density will increase the solid content and decrease the porosity at the same time . In general, the compressed earthen material with low bulk density has more pores which were filled with air at dry state and the thermal conductivity value of air is considerably lower than solid phase. Therefore, the compressed earthen material with lower bulk density has lower thermal conductivity than materials with higher bulk density. The results are similar as the conclusion in the studies of Tang et al. [15] and Taallah et al. [16].

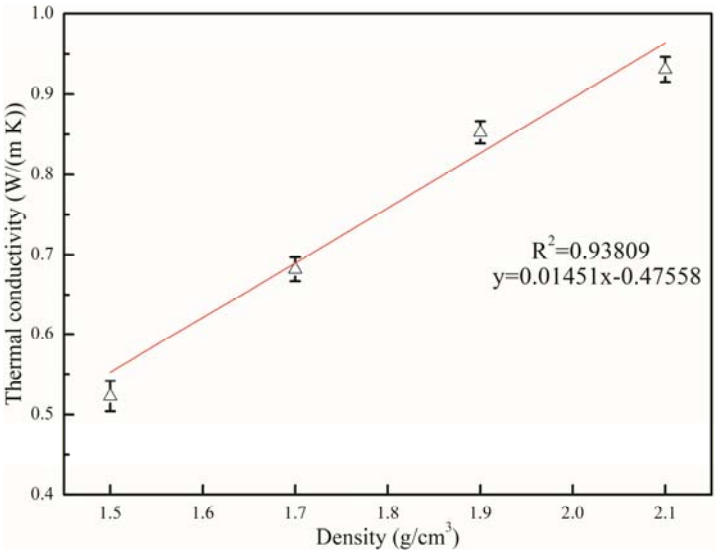

Fig. 6 Thermal conductivity vs. bulk density of compressed earthen material.

3.3. Adsorption-desorption isotherms

Fig. 7 shows the evolution of equilibrium moisture content of compressed earthen bricks with different bulk densities in increasing relative humidity. The evolution with time for compressed earthen bricks subjected to increasing relative humidity demonstrates that the response of samples to a relative humidity change is rather fast and equilibrium moisture content values of examples at different bulk densities usually reach a stabilization in four days. This shows that compressed earthen bricks with different bulk

densities can absorb moisture from surrounding with increasing relative humidity to regulate the indoor relative humidity.

It is possible to interpret the impact of bulk density on equilibrium moisture content presented in Fig. 7 by comparing the results of compressed earthen bricks with different bulk densities. It is interesting to observe that a small variations between the samples existed in the region of high relative humidity. Further, the equilibrium moisture content at high relative humidity reduces firstly for increasing densities (from 1.5 g/cm^3 to 1.9 g/cm^3) and then increases as the bulk density increases from 1.9 g.cm^3 to 2.1 g/cm^3. This result indicates that the variation in bulk density slightly impacts the pore structure inside the matrix as the adsorption at high relative humidity is governed by capillary condensation which is influenced by the pore structure and pore size inside the matrix.

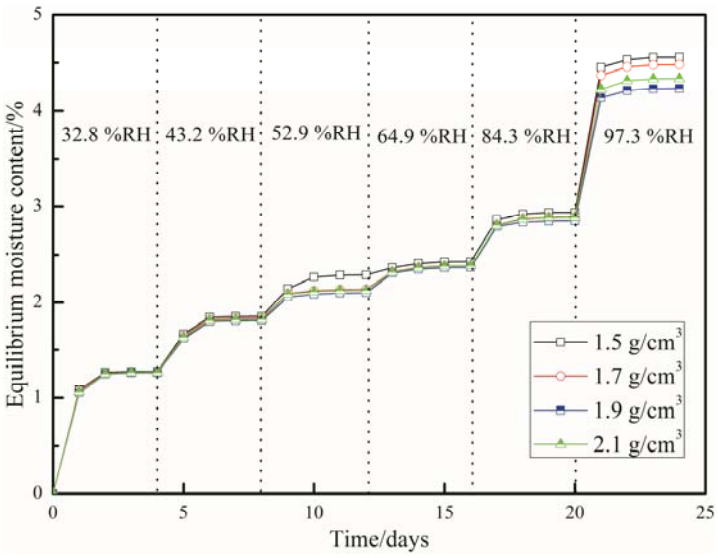

Fig. 7 Evolution of equilibrium moisture content with increasing relative humidity.

Fig. 8 shows the adsorption-desorption isotherms (Fig. 8a) and hysteresis values (Fig. 8b) of compressed earthen samples at different bulk densities. It can be seen from the adsorption curve, equilibrium moisture content increases with increasing relative humidity. This phenomenon indicates that the compressed earthen samples adsorb water vapour from surrounding as relative humidity

219 increases. Two mechanisms are suitable for interpretation of the adsorption [19]: (1) At low relative
220 humidity, water molecules reach to the pore walls and form a monolayer water film by Van der Waals'
221 force. (2) As the relative humidity increases, the water film becomes thicker until the narrow pores are
222 blocked and then the capillary condensation phenomenon occurs. When relative humidity rises and
223 reaches 52.89±0.2 % RH, the first inflection point is appeared on the adsorption curve. It means the
224 single layer surface adsorption occurs at the relative humidity of less than 52.89±0.2 % RH and when
225 relative humidity exceeds 52.89±0.2 % RH, the multilayer surface adsorption occurs. The relative
226 humidity of 84.34±0.1 % RH is the second inflection point which means the capillary condensation
227 occurs in narrow pores as relative humidity exceeds 84.34±0.1 % RH.
228 Fgaier et al. [30] presented that the variation in the adsorption behaviour is mainly related to the type
229 of clay and to the specific surface area of the raw materials. In this study, the adsorption curves of
230 compressed earthen bricks with different bulk densities are very close to each other. On the one hand,
231 the samples used in this study have the similar mineral composition as the rural materials were selected
232 from the same location; on the other hand, it may be explained by the similar specific surface area for
233 compressed earthen bricks with different bulk densities.
234 Additionally, a distinct difference, which is defined as hystersis, can be observed between adsorption
235 and desorption curves as shown in Fig. 8a. McGregor et al. [11] indicated that hysteresis is associated
236 with capillary condensation caused by micropores and mesopores inside the matrix and it can be used to
237 evaluate the moisture buffering as an important parameter. As shown in Fig. 8b, the hysteresis values of
238 compressed earthen bricks increase with increasing relative humidity as the same as Cagnon et al.'s
239 results [12]. This phenomenon can be explained that when the samples, which have completed the
240 adsorption process, start to release water vapour to surrounding, the narrow pores are blocked with

condensed water leads to the adsorbed water molecules can't evaporate outward and then hysteresis occurs. With relative humidity reduces to the degree corresponding to Kelvin's radius, capillary evaporation releases condensed water to surrounding in favour of developing a desorption process and then the hysteresis value reduces with decreasing relative humidity. Fig. 8b also shows that the hysteresis value increases firstly and then decreases with increasing bulk density at a given relative humidity. This result can be interpreted by compactness variation caused by change of bulk density. Fgaier et al. [30] presented a loose soil matrix that had an open texture which facilitated the entry and release of water vapour and impeded increasing of the hysteresis value. When the bulk density increases to 2.1 g/cm^3, increasing of hysteresis values may be explained by increasing amount of mesopores as capillary condensation, which is the reason for hysteresis, usually appears in mesopores [11].

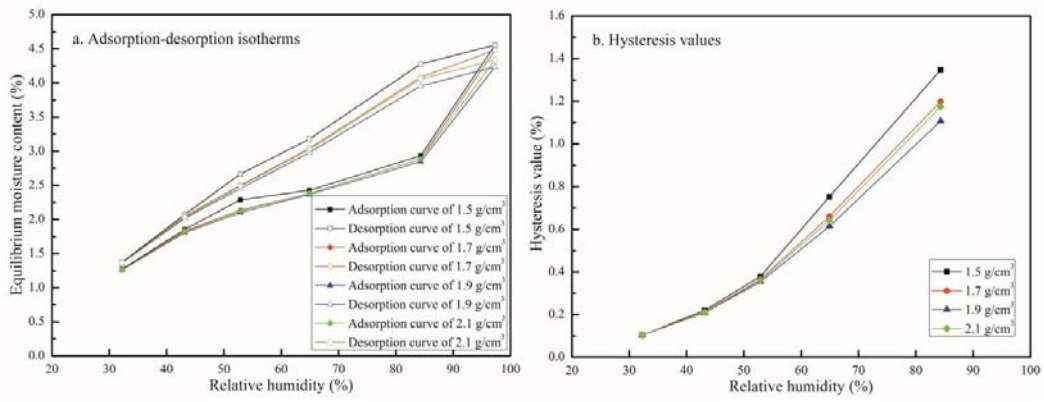

Fig. 3 Adsorption-desorption isotherms and hysteresis values of compressed earthen samples at different bulk densities

3.4. Porosity

Nitrogen adsorption-desorption isotherms of compressed earthen bricks with different bulk densities are examined in order to characterise the effect of porosity on the hygroscopic behaviour. Nitrogen adsorption-desorption isotherms results are presented in Table 3 in terms of specific surface area and pore volume calculated by the BET method, and average pore width obtained with the BJH method.

Table 3 Results of porous structure.

Bulk density (g/cm^3)	Specific surface area (m^2/g)	Micropore volume (cm^3/g)	Mesopore volume (cm^3/g)	Average pore width (nm)
1.50	17.7023	0.001221	0.032239	8.2406
1.70	16.4277	0.001118	0.031268	8.2988
1.90	15.5008	0.000867	0.030785	8.3446
2.10	16.2091	0.000937	0.031019	8.1649

Specific surface area is a key indicator to evaluate the adsorption capacity of porous materials. Fgaier et al. [30] demonstrated a series of experiments for three types of unfired clay bricks and the results showed that the bricks with higher specific surface area have higher adsorption capacity. The experimental values are distributed between 15 and 18 m^2/g in this study, compared with 1 to 4 m^2/g in Raimondo et al.'s study [22] and 4 to 15 m^2/g in Arrigoni et al.'s study [31]. These results interpret that compressed earthen bricks in this study have a larger adsorption capability (between 2.30 % and 2.50 % at 64.92 %RH) than the existing studies (the highest value in Arrigoni et al.'s study is 2.028 % at 58 %RH). Then, a slight difference in the specific surface area between compressed earthen bricks with different bulk densities leads to the tested samples have a similar adsorption behaviour as shown in Fig. 7. In addition, the tiny differences in the equilibrium moisture content between compressed earthen bricks with different bulk densities at high relative humidity are caused by the slight differences in mesopore volume of samples. The micropore volume reveals the hygroscopic ability at low relative humidity and the mesopore volume are responsible for capillary condensation and hysteresis [9]. Furthermore, the relationship between mesopore volume and bulk density results in the hysteresis value firstly reduces and then increases with increasing bulk density.

Nitrogen adsorption-desorption isotherms and BJH desorption pore volume curves of compressed earthen samples at different bulk densities are shown in Fig. 9. Adsorption-desorption isotherms at different bulk densities express the same trend with relative pressure changes and the distinct hysteresis

277 loops existed between adsorption and desorption isotherms. According to a classification of
278 physisorption isotherms raised by the IUPAC in 1984, the hysteresis loops in this study are associated
279 with capillary condensation taking place in mesopores which are between 2 to 50 nm in diameter [29].
280 The IUPAC classified hysteresis loops in 4 types and related them with pore structures [32]. The
281 hysteresis loops in this study belong to the type H3 hysteresis loops which is observed with aggregates
282 of platelike particles. Isotherms with type H3 hysteresis loops indicate that the pores existed in the
283 examples may include micropores or mesopores which interconnect to form a slitlike pore [33]. The
284 compressed earthen bricks in this study contain amounts of clay minerals which demonstrate a platy
285 structure and there are many slitlike pores in the matrix of samples as shown in Fig. 5. In addition, the
286 absorption curves at different bulk densities are biased toward vertical coordinates when the relative
287 pressure of nitrogen is less than 0.1 and absorbed quantity values are lower than 5 cm^3/g, showing that
288 the existing micropore volume values in the samples are very small. Then, the absorption curves rapidly
289 raise in the high relative pressure phase, demonstrating the existing pores in the samples may be
290 composed of irregular pores which are formed by accumulation of earthen materials.

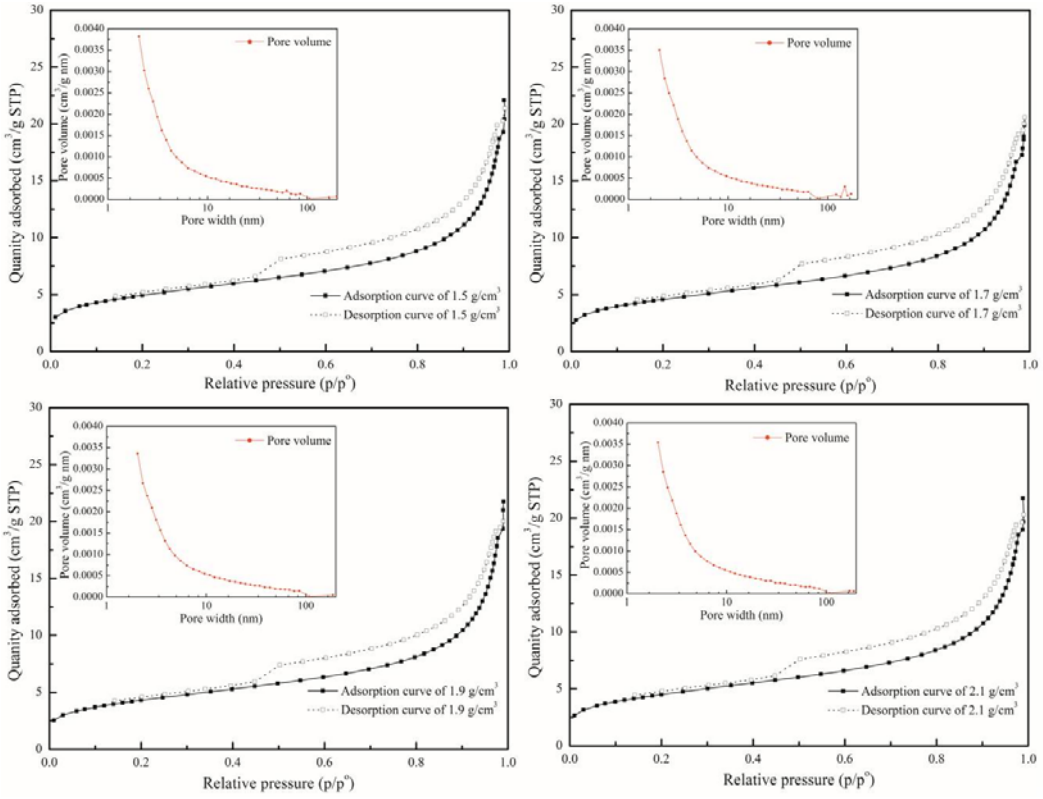

Fig. 4 Nitrogen adsorption-desorption isotherms and BJH desorption pore volume results.

According to the existence of hysteresis between adsorption and desorption isotherms, desorption isotherms curves were used to analyze the distribution of the mesopores' widths. As shown in Fig. 9, there is no obvious peak in the BJH desorption pore volume curves of compressed earthen samples at different bulk densities. It reveals that the pore size of compressed earthen materials distributes in a high content and that no uniform pore structure exist in compressed earthen samples.

4. Conclusions

This study presented a series of experimental analyses for the thermal conductivity and hygroscopic behaviour of compressed earthen bricks at different bulk densities. The effect of bulk density on hygrothermal properties may be summarized as follows:

1. The thermal conductivity of compressed earthen bricks linearly increased with increasing bulk

density. This phenomenon might be explained by both porosity and pore diameter inside the matrix decreased with increasing bulk density.

2. The moisture content in compressed earthen bricks with different bulk densities reached an equilibrium within four days after the relative humidity changed, which indicated that compressed earthen bricks can be used to regulate the indoor relative humidity.

3. Compressed earthen bricks adsorbed water vapour from surrounding as relative humidity increased according to single layer surface adsorption, multilayer surface adsorption and capillary condensation.

4. Compressed earthen bricks in this study have a larger adsorption capability to stabilize indoor relative humidity than in earlier studies.

5. There are many slitlike pores in the matrix of samples and the pore size of compressed earthen materials distributes in a high content.

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