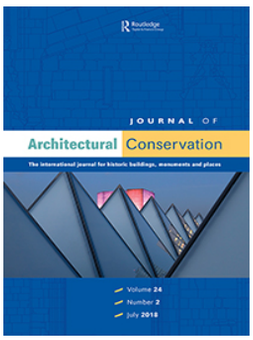

Journal of Architectural Conservation

ISSN: 1355-6207 (Print) 2326-6384 (Online) Journal homepage: http://www.tandfonline.com/loi/raco20

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To cite this article: Shibing Dai (2018) Preliminary study on wind slaked lime used before Qing Dynasty in China, Journal of Architectural Conservation, 24:2, 91-104, DOI: 10.1080/13556207.2018.1491136

To link to this article: https://doi.org/10.1080/13556207.2018.1491136

Published online: 20 Jul 2018.

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JOURNAL OF ARCHITECTURAL CONSERVATION
2018, VOL. 24, NO. 2, 91–104
https://doi.org/10.1080/13556207.2018.1491136

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Preliminary study on wind slaked lime used before Qing Dynasty in China

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ABSTRACT

Historic lime mortars found in the masonry or architectural ruins before Qing Dynasty (1644–1912) show thicker bedding, higher strength, which may be related to the lime slaked by wind described by literature published before Qing Dynasty. The literature recorded that there existed two ways to slake lime: wind slaked and water slaked, which would have caused different properties. In order to assess the building lime properties slaked by wind, the quicklime produced by a lime manufacturer using traditional kiln techniques was collected, and then slaked by wind, mist spray and wet. The mineralogical studies show the calcium silicates like belite have been found both in quicklime and limes slaked by wind and mist. But no calcium silicates have been identified in the wet slaked lime. The lime slaked by wind has got higher strength, its compressive strength can reach 1.5 Mpa in 28 days stored in the indoor air climate, while it needs much shorter setting time. The preliminary results show that the performance of lime slaked by wind is similar to NHL2 classified by EN-459, if the quick lime is kilned from limestone with impurities. The wind slaked lime with higher strength and shorter setting time might have contributed to the magnificent constructions before Qing Dynasty in China. Both the literature and built heritage before the Qing Dynasty should be studied further. More comprehensive researches on wind slaking are needed in China in order to revitalise this traditional lime use combined with ancient wisdom for the economic conservation of built heritage in China.

KEYWORDS

Wind slaking; natural hydraulic lime (NHL); built heritage conservation; *Heavenly Creations*

Introduction

Thanks to modern scientific analysis and testing technologies we know that there exist two types of building lime: air lime and lime with hydraulic properties.[1] Lime with hydraulic properties can be produced either naturally or artificially, there are three types of lime with hydraulic properties classified in the new European standard EN 459–1,[2] i.e. hydraulic lime, formulated lime and natural hydraulic lime (NHL).[3] Hydraulic lime and formulated lime are mostly applied in civil engineering or new architectural decoration, while the NHL is becoming more important, especially in the field of built heritage restoration due to its favourable mechanical and physical performance with low water soluble

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salts.[4] The good mechanical compatibility between NHL and historic masonry from natural stones or bricks in terms of structural strengthening[5] has encouraged both scientists and administrators to rethink this traditional material.[6]

NHL has hydraulic properties produced by burning more or less argillaceous or siliceous limestones (including chalk), grinding it into powder and then slaking with or without grinding.[7] It has the property of setting and hardening both by hydration of the calcium silicates when mixed with water and by carbonation of calcium oxide. The hydraulic property of the natural building lime is not only related to the raw material composition (called cement index, CI) and kilning temperature, but also to slaking methods.[8] Dry slaking and grinding techniques had been applied in Europe and this led to the scientific definition of 'hydraulic lime' in the early eighteenth century, and eventually the invention of Portland cement in the middle of the eighteenth century.[9]

In China, however it was always believed that wet slaking was dominantly used as a standard slaking technique in the past,[10] i.e. lime was slaked into lime putty and there was only air lime, or wet lime used for ancient Chinese building and construction. Even the latest Chinese building lime standard is only a classification of air lime,[11] which reflects the current situation in China.

Therefore, most research efforts in the past two decades in the field of cultural heritage conservation in China were concentrated on the modification of air lime to improve its durability and workability with the help of natural organic materials and fibres, like sticky rice, blood or Tungoil[12] and other additives. The studies of hydraulic properties of historic hardened lime mortars or slaking techniques were ignored.

The latest research on lime mortars from archaeological sites, such as the city walls, the castle and the emperor's palace from the Ming Dynasty (Figure 1) in Fengyang, Anhui

Figure 1. Massive masonry lime in Dong Hua Men Ruins (left) of Central Emperor Palace in Fengyang, Anhui Province, built between 1369 and 1375, at the beginning of the Ming Dynasty.

Province, shows that those lime mortars have higher strength and the chemically hydraulic components according to Wisser and Knöfel's test methods[13] can reach up to16–20 wt% in the binder with brick fragments, whereas the lime mortar, without any aggregates or brick fragments underneath the brick wall, consists of app. 7wt% hydraulic components. These are macroscopic high strength coincidences with a high content of hydraulic components. All those mortars are characteristically much thicker bedded (Figure 2) with fat lime inclusions than the mortars from later construction periods in the Qing Dynasty.

For a long time the hydraulic properties of lime were not known, neither by the construction industry or cultural conservation in China. After a few important research projects were completed, NHL was recognised as a compatible inorganic material for built heritage conservation, and thus practically implemented and further evaluated.[14] Revised evaluation on historic research reports, most of which were not published, has revealed the long history of application of building lime with more or less hydraulic properties.[15] The magnificent construction like the Great Wall was built with 'stronger' lime (Figure 3), which might belong to a kind of lime which is not known today. More comprehensive linguistic analysis of traditional Chinese literature shows that 'wind' slaking was a conventional slaking technique in the past,[16] certainly from the beginning of the North Song (960) to the end of the Ming Dynasty (around 1644).

For more than a decade – under the historic conservation programme with technical support from the Architectural Conservation Laboratory of Tongji University – comprehensive research on historic lime mortars and approaches to using traditional building lime for conservation has been completed. Recently a re-evaluation of ancient Chinese literature about lime for construction and architecture has begun and aims to outline the true picture of building lime in the history of China. Thus a developing a kind of lime that is historically and technically compatible with that used originally in some structures,

Figure 2. Thick bedded historic lime mortars (light beige-ocker coloured) with inclusion built between 1369 and 1375. Modified air lime (light grey coloured) was used for the restoration of the brick wall in 2017 using, which is not compatible neither in colour, texture, nor in the physi-chemical properties.

Figure 3. Historic mortar remains in the Great wall of Lao Long Tou in Shanhaiguang City, Eastern Beginning of Great Wall built around 1381, Ming Dynasty.

has been started being adaptable to modern conservation practice, which began in Europe almost 20 years ago.[17]

This article summarizes the most important preliminary results both from literature research and scientific performance comparison of wind slaked lime, and tries to discover the 'secret' of the traditional slaking technique. The proposal for further approaches pertaining to the resurrection of this slaking technique is also discussed.

1. A review of ancient Chinese literature on lime slaking and application.

As early as 635BC, oyster shell lime was widely applied for both construction and decoration.[18] However, it is not clear, whether the oyster shells were kilned and slaked near the coast regions where they came from or *in situ* where they were used. But it is a tradition in southern China that oyster shell lime was slaked under wind before being used, this lime has feeble hydraulic properties (Figure 4).

In the medicine book *Illustrated Handbook for Materia Medica* (本草图经, c. 1061) by Su Song(1020–1101), two kinds of lime were recorded 'lime slaked by wind'(风化) and 'lime slaked by water'(水化). He described clearly:

> … lime slaked by wind means: the limestone is kilned to quick lime, the quick lime is to be left under wind and disintegrates automatically. The lime slake by wind is STRONG. The lime slaked by water means is to pour water onto the quick lime and the quick lime disintegrates by steam. The lime slaked by water is WEAK.[19] (Original quotation[20])

The 'strong' and 'weak' may indicate either the mechanical strength when slaked lime was used for building or medical effectiveness (faster/shorter) when it was used as a medical additive.

Figure 4. Physical documentation of oyster shell lime for masonry and plastering in East Gate Ruin of former ciry wall of Taizhou, Zhejiang province, bulit in 1387.

The most valuable documentation is the *Heavenly Creations* written by Song Ying-Xing (1587–1666 or 1661). He described the production method of quick lime, its slaking processes and various application possibilities in construction and industry. His recordings[21] (Original quotation[22]) provide us the following important information:

(1) At that time, the quality of limestone for lime production was divided mainly according to the color, the best was cyan-colored or dark gray, yellow and white-coloured were of less quality. The surface deteriorated limestone was not used for lime production.
(2) 90% of fuel for kilning lime was coal and the rest of them was firewood or charcoal.
(3) The quick lime was divided into two categories, 'block quick lime' was of good quality, the 'kiln ash or ash dregs' was of bad quality. The term 'block quick lime' can be traced back to *Ying Zao Fa Shi* (Rules of architecture construction in Song Dynasty, published around 1103).
(4) The quick lime was placed in the air and slowly blown into powder, this was called 'wind slaking'. Only when the project was 'urgent', then the quick lime was slaked with 'water'. Therefore, it can be concluded that the standard method of slaking

lime in the Ming Dynasty was 'wind slaking', 'water slaking' was only used in the case of 'urgency'.

(5) For masonry, 'wind slaked lime' was firstly sieved, then mixed with water. No addition of other materials was indicated.
(6) The lime produced by this method was very durable and could be used even against seawater erosion.
(7) In case of construction of 'tomb' or 'water tank,' lime was added with 'glutinous rice', 'fruit cane juice' etc.

From the above description, it is clear that at least from the Song Dynasty (from 960) to the end of the Ming Dynasty (seventeenth century), when the book *Heavenly Creations* was published, the quick lime was slaked predominately by wind, and in the case of urgency it was also slaked by water to putty, which is termed 'wet slaking' today. The difference between the two slaking techniques was that wind slaked lime was STRONG, water slaked lime was WEAK.

2. Simulation and preliminary performance test of 'wind slaking'

In order to understand 'wind slaking' and compare the performance of 'wind slaked lime' and lime slaked by water, quick lime from a lime manufacturer in Guangde, Anhui Province, was slaked by wind and by water between December 2016 and February 2017.

This lime manufacturer produces different purity of Ca $(OH)_2$ – lime for steel or environmental industries. As for raw materials, there are three kinds of parent lime stones (Figure 5), massive limestone (the fresh surface is cyan-dark grey), banded limestone and layered limestone (both are grey). All three kinds of lime stone are composed

Figure 5. Limestone mine in Guangde, Anhui Province, for calcium hydroxide production and typical banded limestone(right), the raw materials of quicklime for the wind slaking tests (right corner).

of a certain amount of argillaceous and siliceous impurities. The hydraulic index CI ranges between 0.21 and 0.54 based on chemical analysis. The quick lime was produced in a traditional shaft kiln (Figure 6) using coal as fuel, as described in the *Heavenly Creations* in the seventeenth century.

Three kinds of slaking methods were applied to the quick lime from the same patch: wind, water and mist spray.

Wind slaking: leave kilned quick lime in an outdoor open space under a simple shed to prevent rain shower (Figure 7). Rel. humidity ranged from 50–95% during wind slaking (wind speed was not measured).

Water slaking: according to the conventional method, leave quick lime in a bucket, add excess water, sieve the lime milk and store it for 15 days so it becomes putty.

Mist spray: 1.3 times theoretically calculated water is sprayed in one day and stored for 15 days.

In order to obtain the comparison data, mineral composition of quick lime and slaked lime from the same lime manufacturer was analyzed with help of X-ray fluorescence spectrometer. Soundness, setting time, compressive strength according to GB/T1346–2011[23] or European standard EN459–2[24] have been tested to all three kinds of slaked lime, compared with NHL2 from Hessler-Kalkwerk Germany and industrial air lime CL90 from a local Chinese supplier. The mechanical strength was tested on specimen lime with standard sand, which were stored under indoor free air climate (Figure 8) to simulate the actual construction situation.

The preliminary results show that the quick lime from this lime manufacturer consists of mostly calcium oxide (app. 85wt%), but also a small amount of bi-calcium silicates

Figure 6. Shaft kiln for lime production, where the quick lime was collected for testing, coal as fuel as described in the *Heavenly Creations* in the seventeenth century (photo taken in 2016 before environmental upgrading).

Figure 7. Slaking by wind of quick lime under simple plastic shelter (left), photo right shows the lime app. 7 days after exposure to free air.

Figure 8. Storage of lime mortars under indoor climate.

(belite), a small quantity of calcium carbonate, quartz, tri-calcium silicates (alite) (Table 1). The existence of alite shows the kiln temperature has reached more than 1250°C.

The lime slaked by wind consists of not only of calcium hydroxide, but also calcium silicate (suspected belite or larnite), as did the lime slaked by mist spray.

In contrast, in the lime slaked by water, no calcium silicates have been identified, the main minerals are calcium hydroxide (portlandite) and a small amount of calcium carbonate. This could be because during wet slaking, excess water was added to quicklime, calcium silicates begin to hydrate with surplus water and form amorphous calcium silicate hydrates, which are not identifiable by XRD.

Table 1. Characteristics of the lime from various resources.

Quick lime from Guangde /Anhui Province	CaO, $Ca(OH)_2$, C2S(Belite), SiO_2, C3S(Alite), $CaCO_3$			NHL2 (Hessler/ Germany)	CL90
Slaking method	Wind	Water	Mist spray		
Slaked lime	$Ca(OH)_2$, C2S, SiO_2, $CaCO_3$	$Ca(OH)_2$, $CaCO_3$	$Ca(OH)_2$, C2S, SiO_2, $CaCO_3$	$Ca(OH)_2$, C2S, SiO_2, $CaCO_3$*	No analysis
Final Setting time, (h)	3–8	168	27	28	No analysis
Compressive strength**, Mpa (7d)	0.95	0.76	0.67	0.63	0.55
Compressive strength**, Mpa (28d)	1.55	1.17	0.97	1.6	0.83

Notes: *based on manufacturer's data and G. Strübel et al. 1998.
**stored in a free air climate.

The performance of lime slaked by wind has got a shorter setting time, after 21 days, the initial setting time was 1 h, the final setting time was 3 h. From the same quick lime, initial setting of lime slaked by water exceeded 70 h. In terms of strength, the compressive strength of the wind slaked was high, even under the free air curing condition, after 7d reached 1Mpa, 28d reached 1.55Mpa, close to NHL2.

3. Weather conditions for wind slaking

Slaking of quick lime needs water. The water can come from liquid form, gas form or moisture from wet aggregate like sand. The latest research, with the help of atmosphere scanning electronic microscopy, shows that once calcium oxide contacts moisture, its crystal turned into calcium hydroxide with volume increase.[25] The calcium oxide reacts so fast that the hydraulic components remain unchanged when the moisture for slaking is limited.

It can be assumed that the high relative humidity will favour the slaking. The measurement of temperature and rel. humidity of free air from the roof of Wenyuan Building (Campus of Tongji University, No. 1239 Siping Road, Shanghai) provides evidence of high rel. humidity in Shanghai (Figure 9), app. 120 km from Huzhou, where the wind slaking was tested. In the comparable period, the rel. humidity remained very high, between 40% and 100% (Figure 10).

The high humidity favours the carbonation of slaked calcium hydrate as well. Oates[26] stated the air lime slaked in air has less reactivity because the quick lime reacts with vapour and atmospheric carbon dioxide to produce a mixture of CaO, $Ca(OH)_2$ and $CaCO_3$. It is

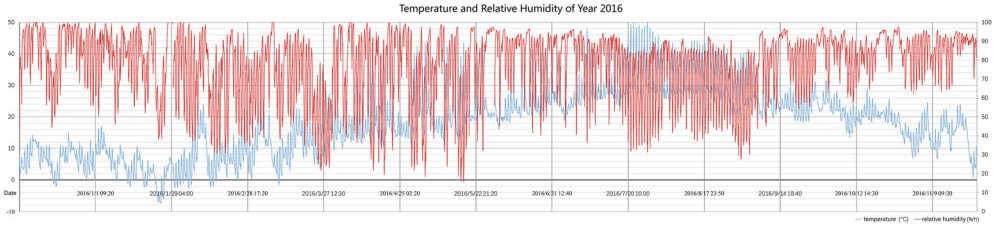

Figure 9. Temperature and rel. humidity of free air from the roof of Wenyuan Building (summarised by Zhong Yan).

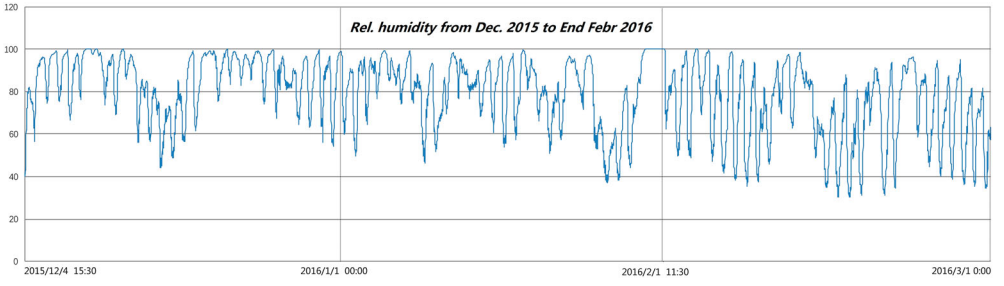

Figure 10. The relative humidity of free air from December 2015 to February 2016 from the roof of Wenyuan Building (summarised by Zhong Yan).

proven by mineralogical research that the calcium carbonate has been identified in all hydrated lime. The earlier setting and higher strength in the hardening of wind slaked lime can only be explained by the contribution of hydraulic hardening, which remains reactive during slaking with limited moisture. The wind slaked lime may be less reactive for industries like steel or environmental protection, but for building $CaCO_3$ or limestone powder might accelerate the carbonation of lime mortars,[27] which is positive for practical application.

4. Conclusion and discussion

The lime slaked from quick lime by wind with low silicious and argillaceous contents shows feeble hydraulic properties, but with much shorter setting time. Its performance in terms of compressive strength is close to the NHL2 defined by European standard EN-459–1 (2010/2015). Those properties shall come from the contribution of hydraulic setting.

Further studies also show such wind-slaked lime has higher expansion.[28] The reasons are not clear, it may be related to the CaO, which has been identified in the wind slaked lime after 14 days, but no more identifiable after 21 days, but it always exists in the NHL[29] or hydrated lime slaked with limited moisture.

Wind slaking disappeared in the literature from the Qing Dynasty (c. 1644–1911) in contemporary construction manuals.[30] It remains unknown which was and is slaked by wind in the southeastern coastal region, except of the practice of using oyster shell lime. Before our project, almost no researchers in China acknowledged this lime slaking technique. The traditional wind slaking technique, which was popular for over 800 years, has been lost and for this there has been much speculation. Song Ying-Xing's answer is the negatives of wind slaking, because wind slaking needs more time than water slaking, 'if the construction project is urgent, quick lime is slaked in the fertile water'.[31] One of the other reasons is that the quality of wind slaked lime is not easy to control. The soundness test shows that it needed at least 21 days (open air) or 40 days (indoor air) for wind slaked lime to be used as binder for masonry without expansion cracks. In ancient times, there was no such test method to control the lime quality. But a more likely reason is that the book *Heavenly Creations* was banned by the Qing Dynasty. Because some works of Song Ying-Xing's brother, Song Ying-Sheng and his friends were politically minded against Qing Emperor, none of their publications were allowed to be published for almost 100 years.

5. Outlook and further studies

At least from the Northern Song to the end of the Ming Dynasty, quick lime was dominantly slaked by wind. Such lime was known as 'strong or powerful' or medicinally as 'quick acting'.

Preliminary research has proven that the hydrated lime slaked by wind from quick lime with certain impurities has got higher strength and a shorter setting time. This might has contributed to the construction of giant architectural pieces, such as the emperor's palace, the city walls or the Great Wall. This conventional wisdom was lost for almost 400 years in China.

Future scientific research to resurrect this 'conventional wisdom' is of extreme importance, because in China lime with shorter setting time and higher strength is needed. Cement was widely used for restoration, one of the reasons is cement sets faster and shortens the restoration time. But today almost everybody knows that cement can cause more damage than conservation (Figure 11).

According to new conservation principles,[32] cement is generally forbidden for restoration of ancient architecture or ruins. In practice, however, small portions of cement are still being mixed to improve the strength and meet today's administration requirements. Air lime with modification of some chemicals is also being promoted, but in most cases such lime formulation is incompatible neither in colour, texture, nor the physic-chemical properties with historic fabrics (Figure 2).

For some restoration projects in China NHL, imported from Europe, is used. NHL from Europe is very expensive due to transport costs, import taxes and logistics. Producing NHL locally in China has been reviewed under the financial support of at least four national foundations,[33] but local production is still almost commercially impossible because of limited demand and extremely strict environmental protection movement. The economic and ecological alternative is to replicate this traditional slaking technique using local quick lime from existing lime manufacturers. With the help of modern testing and quality control

Figure 11. Brick work of the Great Wall from Ming Dynasty was restored many times with cement mortars, which have caused more deterioration than conservation (photo by Hu Zhan-Yong).

technology, a special lime could be produced with favourable mechanical and chemical properties to meet today's administration and conservation principles.

More research is needed. Firstly, the research on historic lime mortars, both on physical objects and literature shall be consolidated. Mineralogical and chemical compositions, texture and mechanical strength of historic mortars, especially from the North Song to the Ming Dynasty, should be documented and provided as basic knowledge for conservation and restoration.

Secondly, comprehensive studies should be carried out to understand wind slaking, soundness relationship between physical performance and slaking time, relative humidity, wind speed, etc. The unwanted expansion of lime may cause damage, which could be avoided when wind slaked lime is used. All slaking parameters shall be strictly defined.

The third aspect is the practical implementation of wind slaked lime mortars to conservation and restoration of historic masonry, earthen architecture, roofs as well as ruins. The limited expansion of wind slaked lime might be particularly suitable for rehabilitation or structural grouting. The long-term durability and interaction of such lime with other historic and modern conservation materials shall also be monitored and assessed.

Wind slaked lime may become a new, but also traditional, material to economically conserve cultural heritage with authenticity, efficiency and traditional wisdom in China.

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20. 风化者, 以锻了石, 置风中自解, 此为有力; 水化者以水沃之, 则热蒸而解, 力差劣。
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22. **凡石灰**经火焚炼为用。成质之后, 入水永劫不坏。亿万舟楫, 亿万垣墙, 窒隙防淫, 是必由之。百里内外, 土中必生可燔石, 石以青色为上, 黄白次之。石必掩土内二三尺, 掘取受燔, 土面见风者不用。燔灰火料, 煤炭居十九, 薪炭居十一。先取煤炭、泥和做成饼, 每煤饼一层, 垒石一层, 铺薪其底, 灼火燔之。最佳者曰矿灰, 最恶者曰窑滓灰。火力到后, 烧酥石性, 置于风中, 久自吹化成粉。急用者以水沃之, 亦自解散。凡灰用以固舟缝, 则桐油、鱼油调, 厚绢、细罗和油杵千下塞舱。用以砌墙、石, 则筛去石块, 水调黏合。甃墁则仍用油、灰。用以垩墙壁, 则澄过, 入纸**筋涂墁**。**用以襄墓及**贮水池, 则灰一分, 入河沙、黄土三分, 用糯粳米、杨桃藤汁和匀, 轻筑坚固, 永不隳坏, 名曰三和土。其余造靛造纸。功用难以枚述。凡温、台、闽、广海滨, 石不堪灰者, 则天生蛎蚝以代之。
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Acknowledgement

Chemical composition of limestone, XRD analysis of lime were carried out with help of South China University of Science and Technology. Mr Shi Deng-Ke, Ms Zhou Yue-e, Mr Hu Zhan-Yong, Mr Zhang De-Bing from Zhejiang Desaibao Materials Technology Co Ltd., Ms Zhong Yan PhD student and technical assistant at the Architecture Conservation Laboratory of Tongji University, have taken part in part of the research works. The research works are carried out under the of the financial and administration supports of NSFC-Grant No. 51378351 and No. 51738008, the Architecture Conservation Laboratory of the Key Laboratory of Ecology and Energy-saving Study of Dense Habitat, Ministry of Education, China. The author thanks all for their contributions and assistance.

Disclosure Statement

No potential conflict of interest was reported by the author.

Funding

The research works are carried out under the of the financial and administration supports of National Natural Science Foundation of China Grant No. 51378351 and No. 51738008, the Ministry of Education, China.

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